

ÇUKUROVA UNIVERSITY
INSTITUTE OF NATURAL AND APPLIED SCIENCES

PhD THESIS

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**PRODUCTION AND ANALYSIS OF PHYSIO-MECHANICAL
PROPERTIES OF JUTE AND HEMP FIBER REINFORCED
UNSATURATED POLYESTER MATRIX HYBRID
COMPOSITES**

DEPARTMENT OF TEXTILE ENGINEERING

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ABSTRACT

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Today, the environmental problem is a major concern around the world. Although synthetic fiber reinforced composites have wide applications in different fields due to their higher mechanical strength and stiffness, their processing and production is still a major source of environmental hazards. The main disadvantages of natural fiber reinforced composites (NFRC) are their low mechanical strength, less adhesion between fiber and resin, which can be compensated by hybridization of fibers and surface treatment. In this study, three different fiber-reinforced polyester resin hybrid composites with hemp and jute fibers, 30%, 50% and 70% by volume, were produced and the analysis of the mechanical properties of the composites was investigated. Before the reinforcement process, the fibers were subjected to alkali treatment with 3% and 5% NaOH concentration in order to eliminate the adhesion problem between the fiber-matrix and improve the mechanical properties. The composites were produced by hand lay-up method in hybrid form with varying layer arrangement of jute (J) and hemp (H) fibers (J/H/H/J and H/J/J/H). The composites were subjected to mechanical tests for tensile, bending and impact strength. Among all composites, 3% alkali treated composites were found to have the best tensile strength, tensile modulus, flexural strength, flexural modulus and impact strength compared to the others. 30% fiber reinforced composites showed the best tensile strength, tensile modulus and 70% fiber reinforced composites showed the best flexural and impact strength. In terms of fiber layer, J/H/H/J hybrid fiber layer composites exhibited better mechanical properties than H/J/J/H layer composites, except for impact strength.

Keywords: Natural fiber reinforced composite, Hemp fiber, Jute fiber, Sodium hydroxide, Tensile strength, Flexural strength, Impact strength.

ÖZ

DOKTORA TEZİ

**JÜT VE KENEVİR ELYAF TAKVİYELİ DOYMAŞMIŞ POLYESTER
MATRİSLİ HİBRİT KOMPOZİTLERİN ÜRETİMİ VE FİZYO-MEKANİK
ÖZELLİKLERİNİN ANALİZİ**

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Günümüzde çevre sorunu, dünya çapında büyük bir endişe kaynağıdır. Sentetik lif takviyeli kompozitler, daha yüksek mekanik mukavemetleri ve sertlikleri nedeniyle farklı alanlarda geniş uygulama alanına sahip olsa da, bunların işlenmesi ve üretimi hala büyük bir çevresel tehlike kaynağıdır. Doğal lif takviyeli kompozit(NFRC)'lerin ana dezavantajları, düşük mekanik mukavemetleri, lif ile reçine arasında, liflerin hibridizasyonu ve yüzey işlemi ile telafi edilebilecek daha az yapışma olmasıdır. Bu çalışmada kenevir ve jüt lifleri, hacimce %30, %50 ve %70 olmak üzere üç farklı lif takviyeli, polyester reçineli hibrit kompozitlerin üretimi yapılmış ve kompozitlerin mekanik özelliklerinin analizi araştırılmıştır. Takviye işleminden önce, lif-matris arasındaki yapışma problemini gidermek ve mekanik özellikleri iyileştirmek için lifler %3 ve %5 NaOH konsantrasyonu ile alkali işleme tabi tutulmuştur. Kompozitler, jüt (J) ve kenevir (H) liflerin değişen katman dizilimi ile (J/H/H/J ve H/J/J/H) hibrit formda, el yatırma metodu ile üretilmiştir. Kompozitler çekme, eğilme ve darbe dayanımı mekanik testlerine tabi tutulmuştur. Tüm kompozitler arasında, %3 alkali işlem görmüş kompozitler, diğerlerine göre en iyi çekme mukavemeti, gerilme modülü, eğilme mukavemeti, eğilme modülü ve darbe mukavemetine sahip olarak bulunmuştur. %30 lif takviyeli kompozitler en iyi çekme mukavemeti, gerilme modülü ve %70 lif takviyeli kompozitler de en iyi eğilme ve darbe mukavemeti göstermiştir. Lif katmanları açısından, J/H/H/J hibrit lif katmanlı kompozitler, H/J/J/H katmanlı kompozitlerden, darbe dayanımı hariç, daha iyi mekanik özellikler sergilemiştir.

Anahtar Kelimeler: Doğal elyaf takviyeli kompozit, Kenevir elyafı, Jüt elyafı, Sodyum hidroksit, Çekme mukavemeti, Eğilme mukavemeti, Darbe mukavemeti.

EXTENDED SUMMARY

Petroleum based fossil fuels are the original source of production and processing of synthetic fiber. Synthetic plastics and composites reinforced with synthetic fibers are nor biodegradable nor decomposable; instead, they accumulate in the landfill and the natural environment. According to literature, only about 9% of all created plastics are recyclable, with the remaining 79% ending up in landfills and the environment. Plastics made from petroleum and its byproducts are incredibly harmful to the environment and to wildlife. Because of this, there is an increasing demand for high-performance bio-based plastics that are environmentally benign and can replace the use of petroleum resources.

With the increasing awareness of ecological concern as well as new rules and legislation on sustainable and biodegradable product, researchers are looking for the environmentally friendly material thus replace the synthetic fiber to lessen the dependence of petroleum-based products. Natural fibers are obtained from renewable resources, biodegradable, abundant available, lower cost which is responsible for the perfect substitution of synthetic fibers. Natural fiber reinforced composites are also playing as better alternative to synthetic fiber reinforced composites due to their lower specific strength and stiffness, easy and environmentally friendly processing and lower production cost. NFRCs are widely using now from household item to automobile, construction and aerospace for their lightweight characteristics compared to synthetic counterpart.

The mechanical properties of NFRCs depends on several factors such as interfacial bonding between matrix and reinforcing element, fiber orientation, fiber loading, surface condition etc.

One of the major limitations of NFRCs are their lower mechanical properties, which happens for their weak interfacial bonding with matrix. The compatibility of the matrix phase with the reinforcing phase, as well as the surface conditions of the reinforcing phase, are the most important factors in interfacial

bonding. Because NFs and matrixes are polar and non-polar in nature, incompatibility develops during composite fabrication, and the smoothness of NFs surfaces produces poor interlocking between matrixes.

The outer surface of fibers and hydroxyl groups must be modified using different chemical treatments, couplers, and reactive fillers in order to optimize the interface interaction between fibers and matrix. Chemical treatments expose more reactive groups across the fiber surface, resulting in a more effective matrix coupling.

In this work, this problem was overcome by treatment of jute and hemp fiber with two different percentage of sodium hydroxide.

Hybridization is another way to improve the mechanical performance of NFRCs. In compared to separate reinforced polymer composites, a hybrid composite combines two or more types of fibers in a single polymeric matrix to create increased stiffness and strength. Typically, one type of fiber in a hybrid composite has a low modulus and/or lower cost, such as glass or Kevlar, while the other type, such as boron or carbon fibers, has a greater modulus and/or higher cost. Low modulus and low cost fibers make hybrid composites more damage resistant and lower total costs, whereas high modulus fibers provide load bearing capabilities and composite rigidity. As a result, hybrid composites can provide high stiffness and strength, improve impact and fatigue resistance, increase fracture toughness, and reduce weight and/or total cost all at the same time.

Hybrid composites' performance is a weighted sum of the individual constituents, with a more favorable balance between intrinsic benefits and disadvantages. The advantages of one type of fiber may be able to compensate for features that are lacking in other types of composite materials. As a result, effective material design could help establish a cost-performance balance.

Natural fiber -natural fiber hybrid composite may not effective alternative for synthetic fiber reinforced hybrid composites as considered to performance characteristics, but it is valuable in terms of biodegradability and sustainability.

The ultimate performance of the hybrid composite is determined by several factors, such as fiber content, hybrid ratio, fiber orientation, stacking sequences, and innate fiber features.

When fibers are positioned parallel to the direction of applied load, the composites have the best mechanical properties. Natural fibers are harder to reinforce in composites than continuous synthetic fibers. Natural fibers that are short are orientated in different directions. Parallelizing and orientation of the fibers along the fiber axis is the most critical operation for achieving optimal mechanical characteristics.

In this thesis, short jute and hemp fibers are used. Carding was performed to parallelize the fibers and increase the fiber volume per unit weight to verify the mechanical performance in the unidirectional loading condition. One of the most important requirements for natural fiber composites is to be lighter than synthetic fiber composites. The higher volume/weight ratio of natural fiber composites makes it an attractive alternative to synthetic fiber composites. Especially when it comes to different applications in the automobile and aviation industry, lighter weight saves fuel and saves costs.

Appropriate matrix selection is important for the mechanical properties of NFRCs. The matrix plays a very important role in fiber reinforced composites. While the matrix distributes the load to the fibers, it also protects the surfaces of the fibers from mechanical wear and environmental influences. The shape, surface appearance, environmental tolerance and overall longevity of the hybrid composite product are determined by the resin. In the production of natural fiber reinforced composites, the matrix must have a high fiber wetting performance, especially in applications such as the hand lay-up method. In this study, unsaturated polyester resin was used as the matrix material. Unsaturated polyester resin is an important thermoset material used for lamination and coating in the composite industry. It is an adhesive with low cost and good mechanical properties. Unsaturated polyester resin is a polymer that initially cures with heat or catalyst to form liquid monomer or oligomer or insoluble

material. Natural fiber reinforced polyester composites are widely used in many different application areas.

The mechanical performance of composites is affected by the manufacturing technique. Various production techniques are used for the production of composite materials. The selection of the appropriate manufacturing method is influenced by the end-use properties of the composites. In this study, hand lay-up method was preferred for the production of hybrid composite materials due to the ease of application and the fact that it does not require heat. Hand lay-up is the cheapest and simplest technique commonly used for composite material production. In this technique, an open mold produced in accordance with the thesis study was used. Before fiber layering, some form of mold release agent is used for easy removal of the composite. The fibers are in the form of comb strips, and the resin containing accelerator and hardener at specified ratios is poured on the fiber and applied to the mold. A doctor roller was used to properly mix the matrix component resin and to remove the air trapped in the fibers. Layered hybrid composites were obtained by layering to achieve the desired thickness.

Fiber dispersion has been identified as an important factor affecting the mechanical properties of short fiber composites and poses a particular problem for natural fiber composites (NFC) with hydrophilic fibers and hydrophobic matrices. Using longer fibers can create a tendency to agglomeration. By ensuring that the fibers are fully wetted by the matrix, effective fiber dispersion promotes good interfacial bonding and reduces voids, so that the improvement in fiber matrix bonding positively affects mechanical properties and service life. In this thesis, the mechanical properties of jute and hemp fibers with 3% and 5% NaOH concentrations were compared with fibrous composites treated with alkali and without alkali treatment.

The mechanical properties of a composite are affected by its fiber content. Optimization of fiber content is linked to improvement of mechanical properties. Up to a certain point, the mechanical properties improve as the fiber content increases.

Subsequently, the mechanical properties deteriorate due to insufficient matrix wetting of the fibers. When the fibers are absorbed into the matrix, the fiber volume ratio (V_f) is the percentage of the fiber volume in the total volume of the NFC material. The physical and mechanical properties of composites are largely determined by V_f . In this study, the effect of the reinforcement ratio of natural fibers on the mechanical properties of the volume ratio was investigated in three different ways: 30%, 50% and 70% by volume.

When determining toughness (impact strength), fiber order in layers is more important than composition, and different placements maximize different toughness properties such as total energy, initiation energy, and diffusion energy. In this study, jute (J) and hemp (H) fibers were selected in two different layered structure levels as J/H/H/J and H/J/J/H in order to see the effect of layer placement on mechanical properties in comb strips. The aim of this research is to improve the mechanical properties of the composite by changing the order of hybridization, surface treatment and also layering. Although many studies on the improvement of mechanical properties with hybrid composites have been published to date, none of them have revealed the mechanical properties of hybrid composites produced layer-by-layer from carded short jute and hemp fibers, according to the authors' literature knowledge. Three factors that are thought to have an effect on the mechanical properties of composite materials were determined and production was carried out accordingly. These factors are; The % fiber content (3 levels), the %NaOH alkaline concentration (3 levels) and the order of natural fibers jute and hemp in layers (2 levels). Considering these factors, 18 different composite plates were produced. The effects of these three factors on the mechanical properties of composite materials were investigated, supported by statistical methods.



GENİŞLETİLMİŞ ÖZET

Petrol bazlı fosil yakıtlar, sentetik elyafın orijinal üretim ve işleme kaynağıdır. Sentetik liflerle güçlendirilmiş sentetik plastikler ve kompozitler ne biyolojik olarak parçalanabilir ne de bozunabilir; bunun yerine atık yerlerde ve doğal çevrede birikir. Üretilen tüm plastiklerin kabaca %9'u geri dönüştürülebilirken, kalan %79'u çöplüklere ve atmosfere karışıyor. Petrol bazlı plastikler ve yan ürünleri toprak, su ve vahşi yaşam üzerinde yıkıcı bir etkiye sahiptir. Bu nedenle dünyada petrol kaynaklarının tükenmesini telafi edebilen ve çevre dostu olabilen yüksek performanslı biyo bazlı plastiklerin kullanımına yönelik artan bir talep başlamıştır.

Sürdürülebilir ve biyolojik olarak parçalanabilen ürünlere ilişkin yeni kurallar ve mevzuatların yanı sıra ekolojik kaygı konusundaki artan farkındalıkla birlikte, araştırmacılar çevre dostu malzeme arıyorlar, böylece petrol bazlı ürünlerin bağımlılığını azaltmak için sentetik elyafın yerini alıyorlar. Doğal lifler yenilenebilir kaynaklardan elde edilir, biyolojik olarak parçalanabilir, bol miktarda bulunur, sentetik liflerin mükemmel ikamesinden sorumlu olan daha düşük maliyetlidir. Doğal elyaf takviyeli kompozitler (NFRC), daha düşük özgül mukavemet ve sertlikleri, kolay ve çevre dostu işlemleri ve daha düşük üretim maliyetleri nedeniyle sentetik elyaf takviyeli kompozitlere daha iyi bir alternatif olarak oynuyorlar. NFRC'ler, sentetik muadillerine kıyasla hafif özellikleri nedeniyle artık ev eşyasından otomobil, inşaat ve havacılık sektörüne kadar yaygın olarak kullanılmaktadır.

NFRC'lerin mekanik özellikleri, matris ve takviye elemanı arasındaki arayüzeyel bağ, fiber oryantasyonu, fiber yükleme, yüzey durumu vb. gibi çeşitli faktörlere bağlıdır.

NFRC'lerin en büyük sınırlamalarından biri, matris ile zayıf arayüzey bağları nedeniyle meydana gelen düşük mekanik özellikleridir. Matriks fazının takviye fazı ile uyumluluğu ve ayrıca takviye fazının yüzey koşulları, ara yüzey bağlanmasında en önemli faktörlerdir. NF'ler ve matrisler doğaları gereği polar ve apolar

olduklarından, kompozit üretimi sırasında uyumsuzluk gelişir ve NFs yüzeylerinin düzgünlüğü matrisler arasında zayıf kilitlenme üretir.

Lifler ve matris arasındaki arayüz etkileşimini optimize etmek için liflerin ve hidroksil gruplarının dış yüzeyi farklı kimyasal işlemler, bağlayıcılar ve reaktif dolgular kullanılarak modifiye edilmelidir. Kimyasal işlemler, elyaf yüzeyi boyunca daha reaktif grupları açığa çıkararak daha etkili bir matris eşleşmesi sağlar.

Bu çalışmada, jüt ve kenevir lifinin iki farklı yüzdede sodyum hidroksit ile muamele edilmesiyle bu problemin üstesinden gelinmiştir.

Hibridizasyon, NFRC'lerin mekanik performansını iyileştirmenin başka bir yoludur. Hibrit kompozit, artırılmış sertlik ve mukavemet oluşturmak için iki veya daha fazla lif lif-tipini tek bir polimerik matriste birleştirilerek elde edilir. Tipik olarak, bir hibrit kompozitteki bir lif tipi, cam veya Kevlar gibi düşük modüle ve/veya daha düşük maliyete sahipken, bor veya karbon lifler gibi diğer tip, daha yüksek modüle ve/veya daha yüksek maliyete sahiptir. Düşük modüllü ve düşük maliyetli lifler, hibrit kompozitleri hasara karşı daha dayanıklı ve toplam maliyetleri düşürürken, yüksek modüllü lifler yük taşıma yetenekleri ve kompozit sertliği sağlar. Sonuç olarak, hibrit kompozitler aynı anda yüksek sertlik ve mukavemet sağlayabilir, darbe ve yorulma direncini iyileştirebilir, kırılma tokluğunu artırabilir ve ağırlığı ve/veya toplam maliyeti azaltabilir.

Hibrit kompozitlerde bir lif türünün avantajları, diğer hibrit kompozit malzeme türlerinde eksik olan özellikleri telafi edebilir. Sonuç olarak, etkili malzeme tasarımı, maliyet-performans dengesinin kurulmasına yardımcı olabilir.

Doğal lif - doğal lif hibrit kompozit, performans özellikleri dikkate alındığında sentetik lif takviyeli hibrit kompozitler için etkili bir alternatif olmayabilir, ancak biyolojik olarak parçalanabilirlik ve sürdürülebilirlik açısından daha değerlidir.

Hibrit kompozitin nihai performansı, lif içeriği, hibrit oranı, lif oryantasyonu, lif yerleşim katmanları ve doğuştan gelen lif özellikleri gibi çeşitli faktörler tarafından belirlenir.

Kompozitlerin doğal liflerle güçlendirilmesi, sürekli sentetik liflerle güçlendirmekten daha zordur. Kısa olan doğal lifler farklı yönlerle yönlendirilir. Lif eksenine boyunca liflerin paralelleştirilmesi ve oryantasyonu, optimum mekanik özelliklerin elde edilmesi için en kritik işlemdir.

Bugün dünya sera gazı salınımından ciddi şekilde etkileniyor. Doğal lifler yenilenebilir kaynaklardan elde edilir, daha düşük yoğunluğa, daha yüksek özgül mukavemete ve sertliğe sahiptir, bu da sentetik liflerin kullanımını azaltır, daha fazla CO₂ emisyonunu koruyarak çevrenin korunmasını sağlar. Jüt ve kenevir liflerinin üretimi ve işlenmesi sırasında daha az CO₂ salınımı çevresel olumsuz etkinin azaltılması sayesinde dünyayı kurtarır, insan ve diğer hayvanların hayatını kurtarır.

Bu tezde kısa jüt ve kenevir lifleri kullanılmıştır. Tek yönlü yükleme durumunda mekanik performansı doğrulamak için lifleri birbirine paralel hale getirmek ve birim ağırlık başına lif hacmini artırmak için taraklama işlemi yapılmıştır. Doğal lifli kompozitlerin en önemli gereksinimlerinden biri, sentetik lifli kompozitlerden daha hafif olmasıdır. Doğal lifli kompozitlerin daha yüksek hacim/ağırlık oranı, onu sentetik lifli kompozitlere göre çekici bir alternatif haline getirir. Özellikle otomobil ve havacılık sektöründe farklı uygulamalar söz konusu olduğunda, daha hafif ağırlık yakıt tasarrufu sağlar, maliyet tasarrufu sağlar.

NFRC'lerin mekanik özellikleri için uygun matris seçimi önemlidir. Lif takviyeli kompozitlerde matris çok önemli bir rol oynar. Matris, yükü liflere dağıtırken, liflerin yüzeylerini de mekanik aşınmadan ve çevresel etkilerden korur. Hibrit kompozit ürünün şekli, yüzey görünümü, çevresel toleransı ve genel uzun ömürlülüğü reçine tarafından belirlenir. Doğal lif takviyeli kompozitlerin üretiminde matrisin, özellikle el yatırma metodu gibi uygulamalarda, lifleri ıslatma performansının yüksek olması gerekmektedir. Bu çalışmada matris malzemesi olarak doymamış polyester reçinesi kullanılmıştır. Doymamış polyester reçine, kompozit endüstrisinde laminasyon, kaplama yapmak için kullanılan önemli bir termoset malzemedir. Düşük maliyetli ve iyi mekanik özelliklere sahip yapıştırıcıdır. Doymamış polyester reçinesi, başlangıçta sıvı monomer veya oligomer veya

çözünmeyen malzeme oluşturmak üzere ısı veya katalizör ile kürlenene bir polimerdir. Doğal elyaf takviyeli polyester kompozitler, birçok farklı uygulama alanında yaygın olarak kullanılmaktadır.

Kompozitlerin mekanik performansı üretim tekniğinden etkilenir. Kompozit malzeme üretimi için çeşitli üretim teknikleri kullanılmaktadır. Uygun üretim yönteminin seçimi, kompozitlerin son kullanım özelliklerinden etkilenir. Bu çalışmada, hibrit kompozit malzeme üretmek için, uygulama kolaylığı, ısı gerektirmemesi gibi nedenlerden ötürü, el yatırma metodu tercih edilmiştir. El yatırması, kompozit malzeme üretimi için yaygın olarak kullanılan, en ucuz ve en basit tekniktir. Bu teknikte, tez çalışmasın uygun olarak üretilmiş, açık kalıp kullanılmıştır. Lif katmanlamadan önce, kompozitin kolayca çıkarılması için bir tür kalıp ayırıcı ajan kullanılır. Lifler tarak şeritleri halinde olup, kalıba yerleştirilmiş ve belirlenmiş oranlarda hızlandırıcı ve sertleştirici içeren reçine lif üzerine dökülerek kalıba uygulanmıştır. Matris bileşeni reçinenin uygun şekilde karıştırılması ve liflerde sıkışan havanın çıkarılması için rakle rulo kullanılmıştır. Katmanlı hibrit kompozitler, istenen kalınlığa ulaşmak için katman katman tutularak elde edilmiştir.

Lif dispersiyonu, kısa fiber kompozitlerin mekanik özelliklerini etkileyen önemli bir unsur olarak tanımlanmıştır ve hidrofilik liflere ve hidrofobik matrislere sahip doğal lifli kompozitler (NFC)'ler için özel bir sorun teşkil etmektedir. Daha uzun lif kullanımı, aglomerasyon eğilimini yaratabilir. Liflerin matris tarafından tamamen ıslatılması sağlanarak, etkili lif dispersiyonu, iyi ara yüzey bağlanmasını destekler ve boşlukları azaltır, böylece lif matris turunmasındaki iyileşme mekanik özellikleri ve kullanım ömrünü olumlu yönde etkiler. Bu tez çalışmasında jüt ve kenevir lifleri %3 ve %5 NaOH konsantrasyonu ile alkali işleme tabi tutulmuş ve alkali işlem uygulanmamış lifli kompozitlerle mekanik özellikleri karşılaştırılmıştır.

Bir kompozitin mekanik özellikleri, lif içeriğinden etkilenir. Lif içeriğinin optimizasyonu, mekanik özelliklerin iyileştirilmesiyle bağlantılıdır. Belirli bir noktaya kadar, lif içeriği arttıkça mekanik özellikler iyileşir. Daha sonra, liflerin yetersiz matris ıslatması nedeniyle mekanik özellikler bozulur. Lifler matrisin içine

emdirildiğinde, lif hacim oranı (V_f), NFC malzemesinin toplam hacmindeki lif hacminin yüzdesidir. Kompozitlerin fiziksel ve mekanik özellikleri büyük ölçüde V_f ile belirlenir. Bu çalışmada, doğal liflerin takviye oranı, hacimce %30, %50 ve %70 olmak üzere üç farklı şekilde seçilerek hacim oranının mekanik özellikleri etkisi araştırılmıştır.

Tokluğu (darbe dayanımı) belirlerken, katmanlardaki lif sırası bileşimden daha önemlidir ve farklı yerleşimler toplam enerji, başlama enerjisi ve yayılma enerjisi gibi farklı tokluk özelliklerini en üst düzeye çıkarır. Bu çalışmada jüt (J) ve kenevir (H) lifleri, tarak şeritleri halinde, katman yerleşiminin mekanik özelliklere etkisi görmek amacıyla, J/H/H/J ve H/J/J/H şeklinde iki farklı katmanlı yapı düzeyinde seçilmiştir.

Bu araştırmanın amacı, hibridizasyon, yüzey işleme ve ayrıca katmanlama sırasını değiştirerek kompozitin mekanik özelliklerini iyileştirmektir. Bugüne kadar hibrit kompozitlerle mekanik özelliklerin iyileştirilmesi üzerine birçok çalışma yayınlanmış olmasına rağmen, yazarların literatür bilgisine göre bunların hiçbiri, karde kısa jüt ve kenevir liflerinden katman katman üretilen hibrit kompozitlerin mekanik özelliklerini ortaya koymamıştır. Kompozit malzemelerin mekanik özelliklerine etkisi olduğu düşünülen üç faktör belirlenmiş ve buna göre üretim gerçekleştirilmiştir. Bu faktörler; % Lif içeriği (3 seviye), %NaOH alkali konsantrasyonu (3 seviye) ve doğal lifler jüt ve kenevirin katmanlardaki sırası (2 seviye) şeklindedir. Bu faktörler göz önünde bulundurularak birbirinden farklı 18 kompozit plaka üretilmiştir. Bu üç faktörün kompozit malzemelerin mekanik özellikleri üzerindeki etkileri, istatistiksel metotlarla da desteklenerek, araştırılmıştır.



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1. INTRODUCTION

1.1. Composites Structure

In today's world, the high fast technology and the unlimited expectation of human beings are the reason for creating the demand for innovative materials (Raghavendra et al., 2017). In this perspective, When compared to traditional materials such as metal and wood, composite materials appear to be one of the most favoured alternatives as innovative materials (Agarwal et al., 2014; Gopinath et al., 2014).

According to literature, the earliest composite material was created by the ancient Egyptians 3000 years ago. It was a straw-reinforced composite material made of clay that was utilized in building construction (Sapuan & Maleque, 2005). Since that time, composite materials have been employed in a variety of industries, including the automotive, aerospace, marine, sporting goods, electrical, and defense industries (Jawaid et al., 2013; D. G. Lee & Suh, 2006; Sapuan & Maleque, 2005).

When two materials with differing physical and chemical properties and distinct interfaces between them combined to form a single material termed as composites. The continuous phase is matrix while discontinuous phase is called reinforcing element. The reinforcement is load bearing member whereas matrix binds reinforcing fiber and distributes load among them. Composites are generally possess superior chemical and mechanical properties than the parent materials (Shekar & Ramachandra, 2018).

1.1.1. Matrix

Matrix provides the composite structure and shape, as well as protecting the fibers from corrosion and chemical effects (Potluri, 2019). The matrix material might be made of metal, polymer, or ceramic.

Metal matrix Composites have a number of advantages over monolithic metals, including higher specific modulus, specific strength, improved high-

temperature properties, and a lower coefficient of thermal expansion. Combustion chamber nozzles (in rockets and space shuttles), housings, tubes, cables, heat exchangers, structural components, and other uses are all being considered for metal matrix composites (Chandramohan and Marimuthu, 2011).

Ceramics are chosen as matrix material because they tolerate in very high application temperature ($>2000\text{ }^{\circ}\text{C}$), low densities and they have very high elastic modulus value. The drawbacks of ceramic matrix composites are brittleness and lack in uniform properties (Akovali and Uyanik, 2001).

Polymers are one of the most utilized materials in composite materials as the matrix phase. When polymer is used as matrix, the composite is called polymer matrix composite. Firstly, for many structural applications, the mechanical characteristics of polymer matrix are typically insufficient. Their strength and stiffness are significantly lower than those of metals and ceramics. By employing polymers to reinforce other materials, these problems can be resolved. Secondly, neither high pressure nor high temperatures are required for the production of polymer matrix composites. Additionally, polymer matrix composites can be made with simpler machinery. The two categories of synthetic polymers are thermoplastic and thermoset polymers (Boopalan et al., 2013; Chandramohan and Marimuthu, 2011; Gupta and Srivastava, 2016; Potluri, 2019; Syduzzaman et al., 2020). To binds the natural fibers, both thermoplastic and thermoset polymers have been used.

1.1.1.1. Thermoplastic Resin

The molecules of thermoplastics do not crosslink and can be melted and solidified by heating and cooling, allowing for repetitive reshaping and reformation. They're ductile and tougher than thermosetting resins in general, and they're frequently used for nonstructural applications that don't require reinforcements or fillers. The mechanical features of thermoplastic resins include outstanding tensile strength and stiffness, good compression and fatigue strength, excellent dimensional stability, and exceptional durability and damage tolerance. They are also wear-

resistant and flame-retardant, making them suitable for a wide range of applications (Nguyen et al., 2017). The viscosity of thermoplastic resins in the melted state is roughly 500–1000 times that of uncured thermoset resins (Gholampour & Ozbakkaloglu, 2020). Polypropylene, polycarbonate, polyimides, and polyethylene terephthalate have been widely used as thermoplastic polymer.

1.1.1.2. Thermoset Resins

Thermosetting polymers are the most utilized matrix materials in the fabrication of polymer-based composites, owing to their simplicity of processing. At first, they are telechelic reactive oligomers with a low molecular weight. They usually have two (telechelic oligomer and curing agent) or more components, including a catalyst and/or a hardener. When the components are combined, solidification begins (at either ambient or elevated cure temperatures). A rigid, strongly crosslinked network or a vitrified system is formed because of the subsequent reaction (Akovali and Uyanik, 2001). They are insoluble and infusible compounds that cure with heat or a catalyst. Thermosets aren't like thermoplastics in that they can't be melted and reformed by heating. This type of resin has a stronger modulus, improved creep resistance, higher thermal stability, and higher chemical resistance than thermoplastic resins due to three-dimensional covalent bond between the polymer chains. At room temperature, they are brittle and have a low fracture toughness (Gholampour and Ozbakkaloglu, 2020). Unsaturated polyester, epoxy and phenol have been largely used as thermosets resin.

Table 1.1. Properties of some thermosets resin (Campbell, 2010).

Resin Type	Characteristics
Polyester	Higher flexible in processing. Application could be both for continuous and discontinuous composites, cheap
Epoxy	More expensive and more temperature resistance than polyester and vinyl ester resins.
Polyamide	Difficult to process, High temperature resistance
Phenolic	High fire resistance, difficult to process
Vinyl Ester	Lesser inexpensive Tougher than polyester resin.

Unsaturated Polyester Resin: The most versatile class of thermosetting polymers is unsaturated polyesters. They're macromolecules made up of an unsaturated component (maleic anhydride or its trans isomer, fumaric acid; which supplies reaction sites) and a saturated dibasic acid or anhydride including dihydric alcohols or oxides (typically phthalic anhydride, which can be replaced by an aliphatic acid, like adipic acid, for improved flexibility). It's a viscous liquid that can be cured with a free radical curing technique to produce solid plastics that are insoluble and infusible (Akovali and Uyanik, 2001; Osman, 2012).

The UP resin is a mixture of unsaturated polyester and styrene monomer. The unsaturation in the backbone provides sites for peroxide initiators to react with the double bonds in the styrene monomer, resulting in the development of a three-dimensional network. The UP resin capable of forming strong bonds with a variety of materials. In addition, it has good toughness, excellent resistance to chemical, high design flexibility, good processing capabilities, resistance to corrosion and fire retardant properties. It has a limitation in using for poor resistance to crack

propagation, low impact strength and small elongation to break (Bakar & Djaider, 2007; Naguib et al., 2015).

The UP resin can easily be processed in hand lay-up method, resin transfer moulding, filament winding process in liquid form. They possess excellent mechanical properties, weather resistance. They can also be handled in the process of sheet molding compound, bulk moulding compound, insulation coating, fiber reinforced plastics etc. (Waigaonkar et al., 2011).

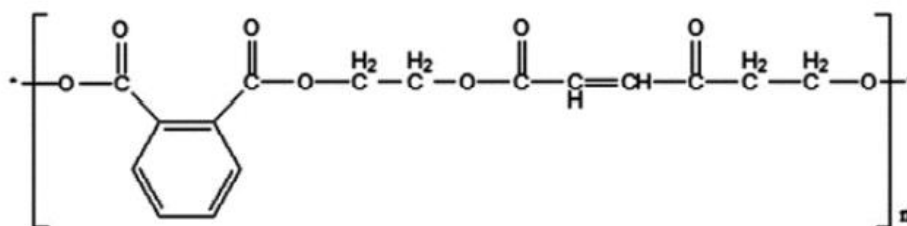


Figure 1.1. Chemical structure of unsaturated polyester resin (Kargarzadeh et al., 2015).

Polyester resins cure quickly, but they have higher value in curing shrinkage than that of epoxy (Seyhan et al., 2007). High resistance to chemical and environmental attack is a property of polyester resin. In comparison to epoxy resin, they are very inexpensive and also have greater dimensional stability and low viscosity (Manfredi et al., 2006; Pıhtılı and Tosun, 2002).

Polyester resin is mostly used with cellulosic and glass fibers as reinforcement (Aziz and Ansell, 2004; Pıhtılı and Tosun, 2002). A catalyst must be added to the polyester to start the polymerization process, and as well as an accelerator to speed up the reaction (Waigaonkar et al., 2011).

Table 1.2. Properties of polyester resin (Ku et al., 2011).

Property	Polyester Resin
Density (g/cm ³)	1.2 - 1.5
Elastic modulus (GPa)	2 - 4.5
Tensile strength (MPa)	40 – 90
Compressive strength (MPa)	90 – 250
Elongation (%)	2
Cure shrinkage (%)	4 – 8
Water absorption (24h @20°C)	0.1 – 0.3
Izod impact strength (J/m)	0.15 – 3.12

Catalyst and Accelerator: Catalyst is the substance which initiate free radical polymerization to cure unsaturated polyester matrix. The decomposition of initiator accomplished by heat or irradiation to generate free radical that initiate or propagate polymerization. Peroxide catalysts such as MEKP, benzoyl peroxide and cumene hydroperoxide are generally employed for crosslinking unsaturated polyester.

Accelerators are added to polyester resin to accelerate the decomposition of the catalysts to free radicals. The best-known accelerators are metal compounds (primarily those of cobalt), amines and sulphur compounds. At present, cobalt compounds and amines are the most widely used accelerators. Cobalt octoate is the most used accelerator for polyester resins catalyzed with MEKP. The reactivity of the polyester to be cured, size and shape of the product affect the choice of curing system. The concentration of catalyst and accelerator could be able to control the length of cross-link and consequently the mechanical properties of polyester resin (Nasr & Abdel-Azim, 1992).

1.1.2. Reinforcing

Reinforcing material present in the matrix in the form of sheets, fragments, particles, fibers, or whiskers made of natural or synthetic materials. Continuous fiber

reinforcement and discontinuous fiber reinforcement composites are made up with long and short fiber respectively. In continuous fiber composites, fibers can be inserted unidirectionally or bidirectionally in the matrix structure (Rajak et al., 2019).

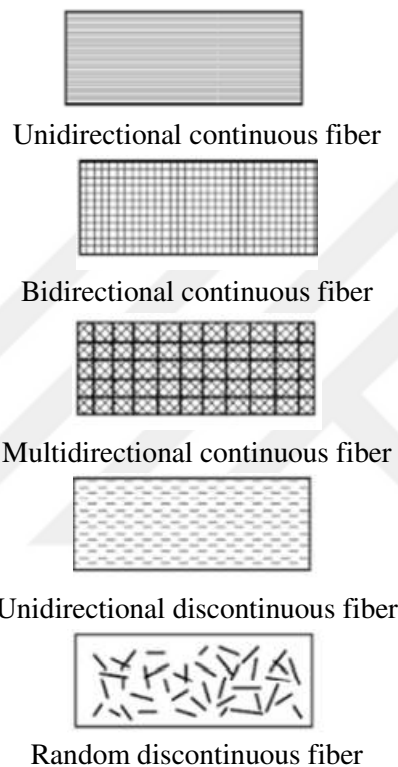


Figure 1.2. Different forms of textile fibers (Mallick, 2007).

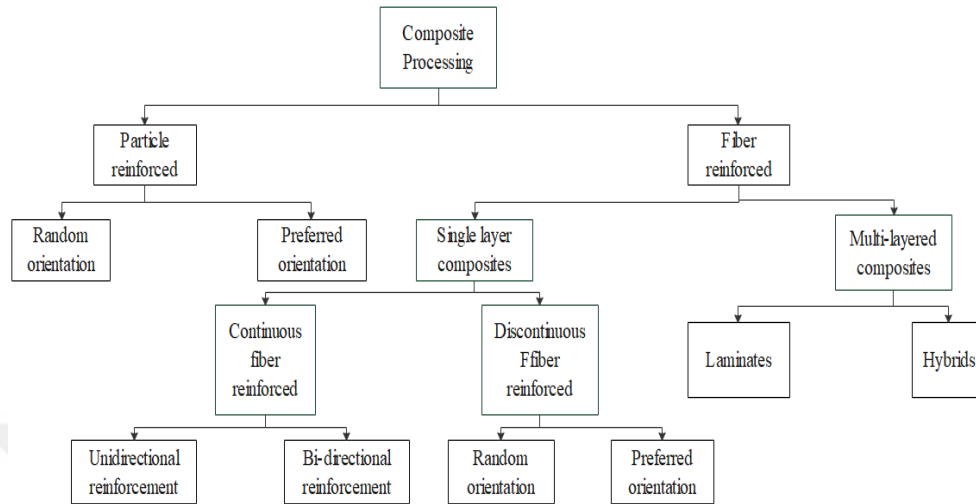


Figure 1.3. Classification of composites on the basis of reinforcement element (Gupta & Srivastava, 2016).

1.1.2.1. Textile Fiber

Fibers are a type of hair-like material that comes in either continuous filaments or discrete elongated peaces, much like thread. It's possible to spin them into filaments, thread, or rope. They can beused in composite materials as a component (Verma and Senal, 2019). Txetile fibers are classified into two types, Natural fiber and man made fiber.

1.1.2.1.(1). Natural Fibers For Composites Material

Natural fibers are classified according to their source, such as whether they come from plants, animals, or minerals. Plant fibers are made of cellulose, whereas animal fibers are made of protein. Mineral-based natural fibers, which are found in the asbestos group of minerals and were once widely employed in composites, are now avoided due to health concerns (carcinogenic when inhaled or ingested) and are banned in many countries. Silk, wool, and feathers are examples of animal-derived natural fibers. Higher performance plant fibers can typically achieve substantially higher strengths and stiffnesses than widely accessible animal fibers. Silk is an

exception, as it has exceptional strength but is also more expensive and stiffer. Plant fibers are the most common natural fibers used in fiber reinforced composites as reinforcement. Plant fibers are categorized into bast fibers, leaf fibers, fruit/seed fibers and grass/reed fibers. Jute, hemp, flax, kenaf, and ramie are some of the mostly used bast fibers. Leaf fibers include sisal, pineapple, abaca, and banana fibers. Coir, kapok, coconut, and cotton are examples of fruit/seed fibers (Akil et al., 2011; Benin et al., 2020; Chandramohan and Marimuthu, 2011; Jariwala and Jain, 2019; Nguyen et al., 2017; Njuguna et al., n.d.; Rajak et al., 2019; Ramamoorthy et al., 2015; Shekar and Ramachandra, 2018; Yahaya et al., 2015) Figure 1.4 shows classification of natural fibers

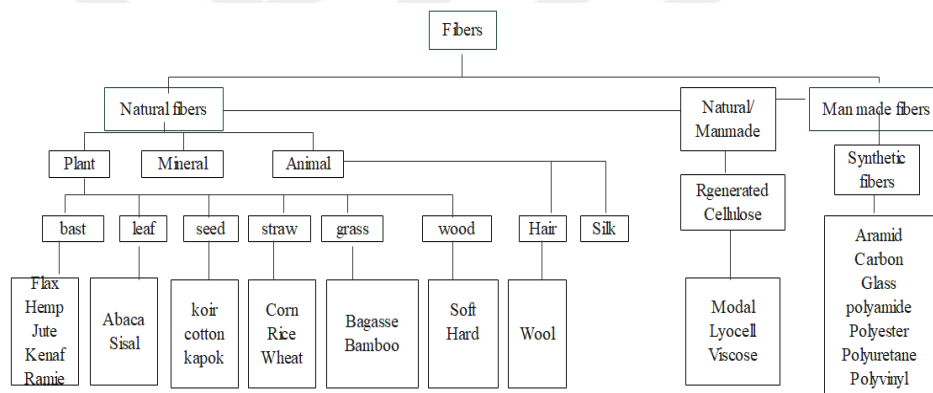


Figure 1.4. Classification of natural fibers (Ramamoorthy et al., 2015).

Table 1.3. Annual natural fiber production of most commercially important fibers (Lotfi et al., 2019).

Fiber type	Origin	World production (10 ³ tons)
Coir	Fruit	100
Kenaf	Stem	970
Flax	Stem	830
Bamboo	Stem	30000
Abaca	Leaf	70
Jute	Stem	2500
Sisal	Leaf	378
Ramie	Stem	100
Cotton	Seed	25000
Banana	Leaf	200
Agave	Leaf	56
Kapok	Fruit	316
Silk	Animal	202
Wool	Animal	2000
Hemp	Stem	215
Pineapple	Leaf	74
Bagasse	Stem	75000

Cellulose: Cellulose is a natural homopolymer, also known as polysaccharide, that occurs naturally in plant fibers and has a relatively high modulus. A D-glucopyranose ring and glycosidic linkage connect this homopolymer to each molecule. Plant fibers are made up of elements including carbon, hydrogen, and oxygen. Cellulose is a linear polymer made up of multiple glucose molecules ($C_6H_{10}O_6$) that act as a basic monomer and a repeat unit called a cello-bios dimer. Furthermore, the molecular formula for cellulose is $C_6H_{10}O_6)_n$, where n is the number of glucose molecules. Depending on the source of cellulose, this amount

varies from plant to plant. Cellulose provides inherent strength and stability of the fiber (Jamir et al., 2018; Njuguna et al., n.d.; Ouarhim et al., 2018).

Hemicellulose: Unlike cellulose, hemicellulose contains a large number of chain ramifications. It has a noncrystalline nature, and it is different from one plant to another. It is very hydrophilic, soluble in alkali solution, and easily hydrolyzed in acids. Hemicellulose is a polymer attached to several kinds of sugar units such as xylose ($C_5H_{10}O_5$), mannose ($C_6H_{12}O_6$), arabinose ($C_5H_{10}O_5$), glucose ($C_6H_{12}O_6$), glucuronic acid ($C_6H_{10}O_7$). Hemicellulose acts as a compatibilizer to support microfibrils, cellulose, and lignin. Hemicellulose contributes structure of the fiber (Njuguna et al., n.d.; Ouarhim et al., 2018; Zwawi, 2021).

Lignin: Lignin is a three-dimensional phenolic polymer that is the second most prevalent biopolymer. This component protects plants from harmful organisms while also contributing to cell wall structural rigidity. It is completely amorphous and has a hydrophobic character. It gives natural fibers a coarse and rigid texture, and its mechanical capabilities are less extensive than those of cellulose. Acids do not hydrolyze lignin, but it is soluble in alkali and condensable with phenol. Lignin keeps water into fiber, protects it from biological attack and as a stiffening stem to provide its resistance against gravity forces and wind (Njuguna et al., n.d.; Ouarhim et al., 2018).

Table 1.4. Chemical Composition of several plant based natural fibers (Kapatel, 2021; Punawala et al., 2019).

Fiber	Cellulose	Lignin	Hemicellulose
Flax	71	2.2	20.6
Hemp	68	10	15
Jute	61-71	12	14
Kenaf	72	9	20.3
Abaca	56-63	7-9	20-25
Sisal	65	9.9	12
Banana	60-65	5-10	6-8
Coir	43	45	<1
Pineapple leaf	80	12	-
Sisal	60-67	10-15	8-12
Wood	45-50	27	23
Ramie	80-85	0.5	3-4

Table 1.5. Mechanical properties of several plant based natural fibers (M. M. Kabir et al., 2012; Nguyen et al., 2017).

Fiber	Density (g/cm ³)	Elongation at break%	Tensile strength (MPa)	Young modulus (GPa)
Flax	1.5	2.7-3.2	345-1100	27.6
Hemp	1.14	1.6	690	30-60
Jute	1.3-1.4	1.16-1.5	393-773	13-26.5
Kenaf	1.2	2.7-6.9	295	-
Ramie	1.5	2-3.8	220-938	44-128
Abaca	1.5	3-10	400	12
Banana	1.35	5.3	355	33.8
Curaua	1.4	3.7-4.3	500-1150	11.8
Heneguen	1.4	3-4.7	430-580	-
PALF	1.5	1-3	170-1627	82
Sisal	1.33-1.5	2-3	400-700	9-38
Cotton	1.5-1.6	3-10	287-597	5.5-12.6
Bagasse	1.2	1.1	20-290	19.7-27.1
Bamboo	0.6-1.1	-	140-230	11-17

Jute: After cotton, jute is the most common vegetable fiber. It belongs to the Corchorus genus in the Tiliaceae family. Commercially, only two species of Corchorus, *C. capsularis* L. and *C. olitorius* L., are grown. White jute comes from *Corchorus capsularis*, while tossa jute comes from *Corchorus olitorius*. In terms of chemical composition, they are very similar. Jute fibers can reach a height of 2.5 to

3.5 meters. Jute fiber produces in Bangladesh, India, Thailand, China. Bangladesh exports approximately 90% of the world's raw jute and associated fibers (Khan and Khan, 2015).

Jute is a multicellular fiber. Jute fiber contains 61-71% cellulose, 14% hemicellulose, 12% lignin, pectin 0.2%, wax 0.5%, water 10%. The cellulose based helical crystalline, helical angle ranging from 20–30° from the cell. This microfibrils are connected to each other by lignin and hemicellulose to form a complete layer. This multiple layers of such cellulose lignin/hemicellulose in one primary cell wall and three secondary cell walls stick together to form multiple layers of composites. Each cell wall differs from other cell wall in terms of composition (ratio between cellulose/ lignin hemicellulose) (Chand and Fahim, 2008). X-sectional view of jute fiber was shown in Figure 1.6

The distinctive qualities of jute fibers are mostly connected to those of cellulose. The distinctive qualities of jute fibers are mostly connected to those of cellulose (Gassan and Bledzki, 1999). Jute has high tensile strength and higher brittleness and low breaking elongation (Acha et al., 2005).

Due to its high modulus, low cost, good thermal and electrical insulating qualities, moderate moisture regain, biodegradability, silky sheen, high specific strength, and commercial availability, jute is one of the most popular natural fiber reinforcements (Acha et al., 2005; K. S. Ahmed and Vijayarangan, 2007; Hasan et al., 2016; Raghavendra et al., 2017; Shahinur et al., 2015; Shanmugam and Thiruchitrambalam, 2013; Tripathy et al., 2000). The drawbacks of jute fiber include its low extensibility, low abrasion resistance, high degree of non-uniformity, low moisture resistance, coarseness, inadequate resistance to microbiological attacks, and poor wettability (Acha et al., 2005; Bhowmick, 2015; Gowda et al., 1999; Saha et al., 1999).

Jute fiber has hydrophilic characteristics because it includes hydroxyl and certain other polar groups. The mismatch between the hydrophilic fiber and the hydrophobic matrix material is primarily responsible for the low wettability of jute

fiber (Acha et al., 2005; Mohanty et al., 2000). Furthermore, it is widely known that the characteristics of jute fiber vary depending on its developing environment, place of origin, and processing methods, just like those of all plant fibers (Gassan and Bledzki, 1999; Gowda et al., 1999).

Jute fiber is mostly used, in combining with thermoset resins including phenolic, vinyl ester, epoxy, and unsaturated polyester for the development of composite material. Jute's employment with thermoplastic resins is limited because of the weak bonding between hydrophilic jute and hydrophobic thermoplastics (Bhowmick M, 2015; Gopinath et al., 2014; Roe and Ansell, 1985).

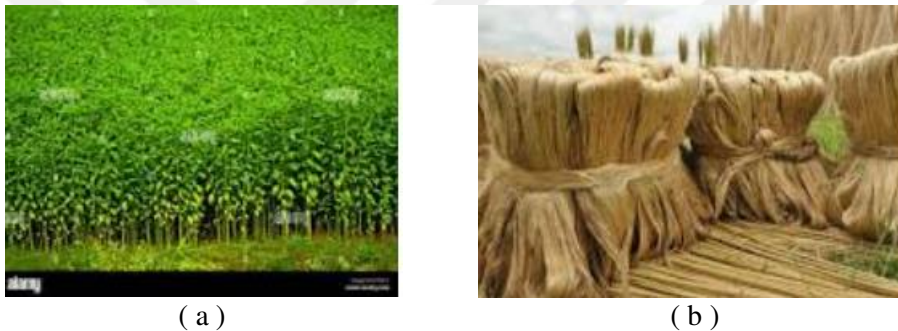


Figure 1.5. (a) Jute plant (b)Jute fibers (*Jute Plant - Google Search, 2022*).

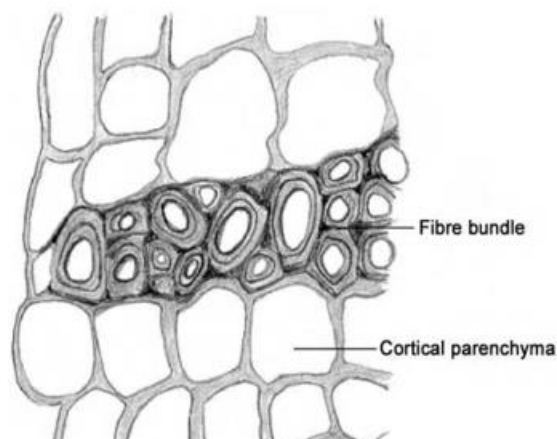


Figure 1.6. Crosssection through jute fiber (Sponner et al., 2015).

Table 1.6. Mechanical properties of jute fibers (Bledzki and Gassan, 1999).

Density (g/cm ³)	Elongation %	Tensile strength (MPa)	Young's modulus (GPa)
1.3	1.5-1.8	393-773	26.5

Hemp Fibers: History of uses hemp fiber dated back to 5000-4000 B.C. (Karche & Singh, 2019). It is now grown mostly in the EU, Central Asia, Philippines, and China (Shahzad, 2012). Hemp fibers are increasingly being used in composite materials as reinforcements, typically substituting glass fibers. These fibers, which are found in the bast of the hemp plant, exhibit strength and rigidity that are comparable to glass fibers (Shahzad, 2014). Hemp fiber contains 68% cellulose, 15% hemicellulose, 10% lignin and 0.8% oil and waxes (Ramamoorthy et al., 2015). Within the hemp stalks, there are multiple layers of fiber bundles. A bundle is made up of many fibers, and unit cells join bundles together. In contrast to the outer layers, the bundles in the inner layers are often shorter and finer. Unit cells might be triangular or heptagonal in shape, with rounded corners and a big pith. Hemp processing is based on loosening and dissolving the lignified pectin link that holds unit cells together. The unit cells' diameter ranges from 15 to 50 microns. The cells' length can range from 5 to 100 mm, with an average length of 35 to 40 mm. The length of fiber bundle is about 1500-2500 mm (Sponner et al., 2015). Cross-sectional view of hemp fiber was shown in the Figure 1.8



A



(b)

Figure 1.7. (a) Hemp plant (b) Hemp fiber (P. K. Kumar and Naidu, 2019).

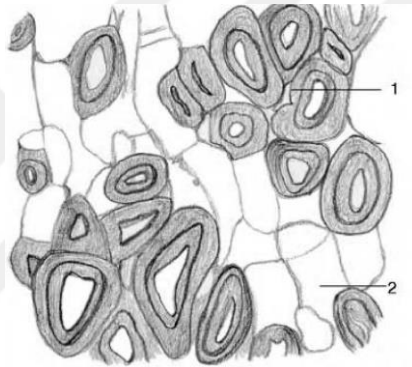


Figure 1.8. Crossection through hemp fibers (1=fiber bundle; 2=cortical parenchyma) (Sponner, 2015).

Table 1.7. Typical physical and mechanical properties of hemp fiber (Shahzad, 2012).

Properties	Values
Length (ultimate) (mm)	8.3–14
Diameter (ultimate) (mm)	17–23
Aspect ratio (length / diameter)	549
Specific apparent density (gravity)	1500
Microfibril angel (θ)	6.2
Moisture content (%)	12
Cellulose content (%)	90
Tensile strength (MPa)	310–750
Specific tensile strength (MPa)	210–510
Young's modulus (GPa)	30–60
Specific Young's modulus (GPa)	20–41
Failure strain (%)	2–4

Table 1.8. Advantages and disadvantages of natural fibers (Jawaid and Khalil, 2011).

Advantages	Disadvantages
Low specific weight, higher specific strength and stiffness	Low strength (specially impact strength)
Renewable resources, low CO ₂ emission	Variable quality
Production with low investment at low cost	Poor moisture resistant
No wear of tool and no skin irritation	Limited maximum processing temperature
High electrical resistant	Lower durability
Good thermal and acoustic insulating properties	Relatively poor fiber matrix adhesion
Biodegradability	Poor fire resistant

1.1.2.1.(2). Synthetic Fibers for Composite Industry

Synthetic fibers are primarily using for the development of fiber reinforced composite due to better qualities than natural fibers (Begum and Islam, 2013) (Begum and Islam, 2013). Synthetic fibers used in the composite material have better strength than plant fibers (Acha et al., 2005). They are less variable than natural fibers. Additionally, they are less dense than metal materials and have higher strength, stiffness, and thermal stability (Begum, K. & Islam, 2013; Unterweger et al., 2014). Long fatigue life, high corrosion and wear resistance, and environmental stability can all be achieved in composite constructions by employing synthetic fibers (Begum and Islam, 2013). However, they have much higher densities and higher costs than natural fibers (Acha et al., 2005; Begum and Islam, 2013). Moreover, poor recyclability and non-biodegradability are the disadvantages of synthetic fibers (Begum and Islam, 2013).

Advantages of Natural Fiber Composites: NFRPCs are environmentally friendly, lightweight, inexpensive, biodegradable, plentiful, and renewable. They are more sustainable alternative to metal-lic and synthetic fibers. Their low cost and high performance complement the economic side of the business.

- When compared to steel fibers or synthetic fibers like glass, aramid, and carbon, NFRPCs have a low density.
- Due to the abrasive character of fiber, recycling and technical processes for composite materials are simple.
- In nonstructural applications such as automobile components such as doors, bonnets, and so on, glass fiber-reinforced composites are being replaced by NFPRCs.
- In comparison to synthetic fibre reinforced polymer composites, NFRPCs are easy for disposal.
- Natural fiber-reinforced polymers have excellent surface quality of molded part composites and have good relative mechanical properties such as tensile modulus and flexural modulus. NFPRCs are also corrosion and fatigue resistant (Verma and Senal, 2019).

Disadvantages of Natural Fiber Composites: Natural fibers are typically hydrophilic, because they absorb moisture. The plant fiber cell wall has a large number of hydrogen bonding (hydroxyl groups OH). When moisture is absorbed in the fiber, these hydroxyl groups form new hydrogen bonds with water molecules. This is owing to the composites' low mechanical characteristics, inadequate fiber-matrix bonding, matrix cracking, dimensional instability, and poor mechanical properties (Jamir et al., 2018).

Hybrid Composites: Natural fibers have several advantages, but they also have some drawbacks, such as lower impact resistance and increased water absorption. These disadvantages can be mitigated by incorporating additional fibers to a single fiber in a polymer matrix, resulting in the development of hybrid composites (Gupta and Srivastava, 2016).

The rule of mixing component for the preparation of hybrid fiber reinforced composite and to calculate volume fraction for the composite following equation has been used

$$V_{c1} + V_{c2} = 1 \quad (1)$$

$$V_{c1} = \frac{V_{f1}}{V_f} \quad (2)$$

$$V_{c2} = \frac{V_{f2}}{V_f} \quad (3)$$

$$V_f = V_{f1} + V_{f2} \quad (4)$$

$$W_f = W_{f1} + W_{f2} \quad (5)$$

$$V_{f1} = \rho_c \frac{W_{f1}}{\rho_{f1}} \quad (6)$$

where, V_f is the total reinforcement volume fraction, V_{c1} and V_{c2} are the relative volume fraction of first and second reinforcement. V_{f1} and V_{f2} are the volume fraction of first and second fiber. ρ_f and ρ_c are the density of the fiber and composites and W_f is weight of the fiber (Sathishkumar et al., 2014).

The mechanical properties of hybrid composites are determined by fiber properties, aspect ratio of fiber content, fiber orientation, individual fiber length, fiber to matrix interface bonding, and fiber dispersion. Randomly oriented short fiber composites have a strength that is halfway between longitudinally and transversely oriented fiber composites. Polymer materials that suitably moisten the fiber surface increase interfacial bonding. Strong interfacial bonding is achieved by include the

appropriate fiber content in the matrix material, which allows for adequate wetting and mixing (Das, 2017; P. V. Joseph et al., 2003; Singha and Rana, 2012).

1.2. Mechanical Performance of Composite Structure

The mechanical properties are the principal criteria that affects the end use area of composite material (Wang et al., 2011b) Three major categories can be made out of the variables that influence the mechanical characteristics of composite constructions. These are i. reinforcement and matrix material parameters, ii. interface characteristics and iii. voids (Wang et al., 2011b; Shah, 2013).

1.2.1. Reinforcement and Matrix Parameters

The characteristics of the reinforcement material primarily influence the mechanical properties of composite material (Wang et al., 2011b). The performance of composites is influenced by the fiber length, orientation, shape, and material type.(Campbell, 2010; Huang and Talreja, 2005; J. Zhang et al., 2012). The primary factor that affects the mechanical characteristics of composite materials is the fiber volume fraction (Elanchezhian et al., 2014; Lee and Suh, 2006; Shah, 2013). Because the reinforcing material gives the composite material its strength and stiffness, the mechanical properties of the structure can be improved by reducing the amount of matrix material (Wang et al., 2011b).

1.2.2. Interface Characteristics

The region between the matrix and the reinforcement material known as the composite interface is what gives a material its durability (Peterson et al., 2011; Wang et al., 2011c). The schematic view of a composite interface is displayed in Figure no. 1.9. The middle phase of the composite material is defined as the interface, which is a layer with a specific thickness (Wang et al., 2011c).

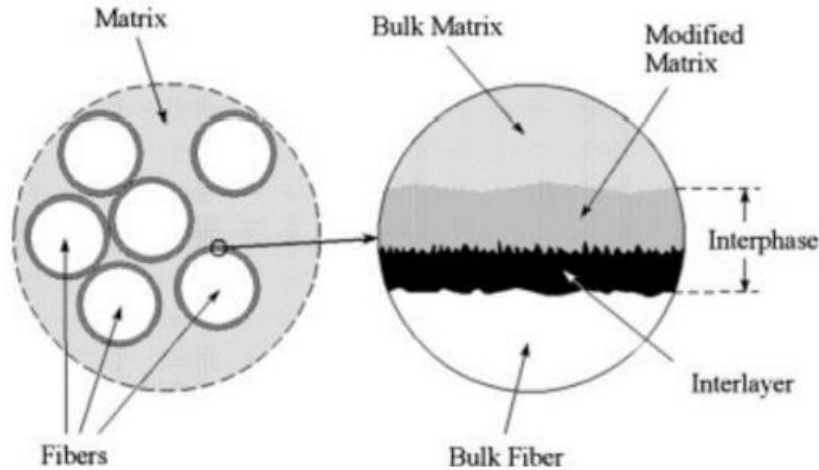


Figure 1.9. Schematic display of the composite interface (Cech et al., 2013).

The liquid matrix material flows over and covers the reinforcement material during the process of "wetting." To achieve the wetted reinforcement, the matrix material's viscosity shouldn't be too high (Lee and Suh, 2006). There is a direct relationship between the interface bonding strength of material and wettability. The wetting property of a material can be altered by adjusting the surface tension of the reinforcement (Wang et al., 2011c).

The interfacial region is potentially a site for crack development, interfacial debonding, and stress concentration due to the differing mechanical characteristics of the matrix and the reinforcing material (Peterson et al., 2011; Thomas et al., 2012). Additionally, mechanical or thermally induced fatigue causes cracks to grow which leads to failure in mechanical properties (Peterson et al., 2011).

The primary mechanical properties of composite materials that are affected by interfacial bonding strength are impact and tensile strength. The failure mode of the composite, which is directly related to the material's impact properties, is greatly influenced by the bonding strength (Acha et al., 2005; Wang et al., 2011a). The interfacial bonding strength of a composite material affects its fracture resistance as

well. A fragile structure is produced by a strong contact. This indicates that low fracture resistance results from a strong contact (Lee and Suh, 2006; Ornaghi et al., 2010; Wang et al., 2011c).

Polymer matrices are hydrophobic, but natural fibers are hydrophilic. Natural fibers and polymer matrices' interfacial bonding is poor as a result of these mismatched features (Portella et al., 2016). Additionally, natural fibers' dangling hydroxyl and polar groups contribute to the poor adherence of the fiber to the matrix (Ornaghi et al., 2010; Tripathy et al., 2000).

1.2.3. Voids

The void is the additional factor that influences the mechanical characteristics of composite structures. Most of the time, the words void and porosity are used interchangeably. Porosity denotes multiple small pores, whereas void denotes a single large pore. In composite research, the word "void" predominates over "porosity" (Campbell, 2010).

In composite structures, voids are areas where air or moisture has been trapped. Although they occasionally have a favorable impact on density (reduction of density), they have a particularly negative impact on the mechanical properties of composite materials (tensile, flexural, shear strengths, etc.) (Ashworth et al., 2016; Bodaghi et al., 2016; Chambers et al., 2006; Hagstrand et al., 2005; Huang & Talreja, 2005; Jawaaid et al., 2011; Lee and Suh, 2006). Voids can be both located in plies or in ply interfaces (Campbell, 2010).

Although they are typically expressed as void content, the quantity of voids in composites are occasionally referred to as void volume fraction (Huang and Talreja, 2005). Modelling of voids in a composite structure is given in Figure no 1.10

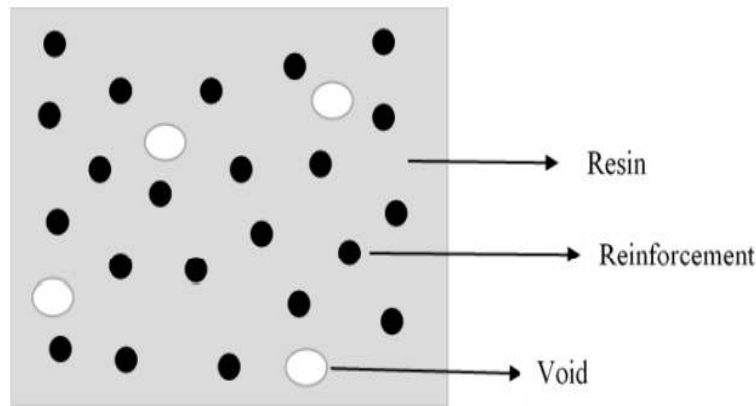


Figure 1.10. Modelling of voids in a composite structure.

The air gaps that developed during the lay-up process, the viscosity of the resin, the permeability of the reinforcement material, and the insufficient resin pressure that retains the residual moisture in resin until resin solidification begins are the main causes of voids in composite materials (Campbell, 2010; Jawaid et al., 2011; Protz et al., 2015). Low resin passage caused by insufficient pressure will result in void formation (Chambers et al., 2006; Huang and Talreja, 2005).

The incidence of the void content is strongly influenced by the process parameter used in composite production procedures. Faulty process parameters may cause the material to develop air gaps, which can lead to voids (Ashworth et al., 2016).

Void content usually increases with the increment of fiber content (Sreekala et al., 2002). However, several research claimed that the relationship between void content and fiber volume percentage is not clearly correlate (Roe and Ansell, 1985; Shah et al., 2012; L. Zhang and Miao, 2010). Additionally, it is noted in the literature that a composite material's void content considerably rises when the fiber volume ratio exceeds 40 (Protz et al., 2015).

1.3. Enhancement of Mechanical Properties of Composite Structures

There are several numbers of methods to enhance the mechanical properties of composite materials. These methods will be studied under two headings. These are: hybridization and changing the stacking sequence of fibers and surface treatment.

Due to the fact that interfacial strength is the key parameter influencing the mechanical properties, interfacial treatments are mostly used to improve the properties of composites (Sapuan and Maleque, 2005). The two main interfacial treatment techniques are the incorporation of fillers and fiber surface treatments (Tugrul Seyhan et al., 2008). Changes in the stacking order of the fiber layers as well as hybridization of low and high strength fibers have a significant impact on mechanical performance.

1.3.1. Hybridization and Changing the Stacking Sequence of Fiber Layers

In comparison to conventional composites, higher mechanical properties and lower material costs can be obtained by employing two or more types of reinforcement materials (Agarwal et al., 2014; Jawaid and Abdul Khalil, 2011; Pandya et al., 2011; Portella et al., 2016; Sezgin and Berkalp, 2017). The hybrid composite include the advantageous properties and eliminate the undesirable quality of each component (Hamouda et al., 2017; Hasan et al., 2016). Although it is stated in the definition of hybrid composite materials that hybrid structures may use two or more reinforcement materials, it is declared in the literature that the best qualities are obtained when only two types of reinforcing materials are used (Banerjee and Sankar, 2014).

A high strength fiber is combined with a natural fiber to provide a good balance between performance, cost, and environmental affects (Ashworth et al., 2016). The design of a laminated composite material depends on choosing the best arrangement of the materials (Ghiasi et al., 2010). The mechanical properties of

composites are greatly affected by changing the place of fiber layers (Ramesh et al., 2013; Wang et al., 2011b).

1.3.2. Fiber Surface Treatment

The rich hydroxyl group present in the natural fiber has strong attraction towards moisture and make it hydrophilic in nature. The polymers using as matrix material like unsaturated polyester, polypropylene, polyethylene poly vinyl chloride, polylactic acid are water repellent. The hydrophilicity of natural fiber and hydrophobicity of polymer matrix are responsible to make weak interfacial bonding between them (Sood and Dwivedi, 2018).

Poor wettability of natural fiber also causes insufficient adhesion, leading to debonding during use of composite material. For this reason, different surface modification is adopted to increase interfacial bonding of fiber with matrix, results in achieving better mechanical properties of composite material. Surface modification by chemical treatment is one of them which purposely activate hydroxyl group or to implement unique moieties that can interlock fibers excellently with the matrix. A range of chemical treatment applying to natural fiber are reported, from which very well-known are alkali, silane, combination of alkali and silane, monomer grafting under UV radiation, maleic anhydride grafted polypropylene (T.-T.-L. Doan et al., 2005; Kapatel, 2021).

1.4. Applications

Due to its eco-friendliness, light weight, good mechanical performance, sound attenuation, and vibration dampening, natural fiber composites have been used in the automotive, construction, aircraft components, packaging, sporting equipment, electrical parts, and biomedical industries (Gholampour and Ozbakkaloglu, 2020).

Natural fiber polymer composites can be used to replace glass in a variety of applications where load bearing capability is important. In structural applications, fiber reinforced composites outperform polymer resins due to their superior

mechanical properties and lower cost (Karthi et al., 2019). The uses of hemp composites remarkably increased in the German and Austria automotive sector in compared to other natural fiber composites (Ahmed et al., 2022).

1.4.1. Automotive Sector

Natural fibers are replacing petroleum-based synthetics in automotive applications due to the growing interest in sustainable raw resources and product recycling or biodegradability. Natural fiber composites have been used in the automotive industry since the 1940s, when Henry Ford developed the first composite components in a car using hemp fiber (Peças et al., 2018).

In the automotive industry, NFRCs are widely used. It is estimated that 75% of fuel consumption is directly related to vehicle weight, with every 10% reduction in vehicle weight resulting in a 6–8% increase in fuel economy and every 100 kg reduction in vehicle weight resulting in a CO₂ emission reduction of approximately 20 g/km for commonly used powertrains (Li et al., 2020). In the car industry, hemp fiber reinforced composites are mostly used for door panels, passenger rear decks, pillars, and boot linings (Dhakal and Zhang, 2015).

1.4.2. Others than Automotive Sector

Despite most prominent applications of natural fiber composites are in automotive sector there are additional fields of applications of natural fiber composites mainly: textiles, medical, healthcare and pharmaceuticals, home and personal care, food and feed additives, construction and furniture, packaging, pulp and paper, bioenergy and biofuels (Peças et al., 2018). Research has been focused on the hemp composite to manufacture sporting goods, musical instrument. Hemp composites also used to develop electronics racks for helicopter which was 55.6% lighter than existing steel electronic racks (Ahmed et al., 2022)

Jute has traditionally been used for packaging, rope, and other household items. Door panels, roofing, and sanitary products have all been made with jute fiber

reinforced. Jute reinforced thermoplastic laminates and composites are a possible replacement for thermoplastic and synthetic fiber reinforced composites due to their good physical qualities at low weight. Footwear, building/construction, home/garden furniture, and toy industries are all potential future applications of jute composites (Khan and Khan, 2015).

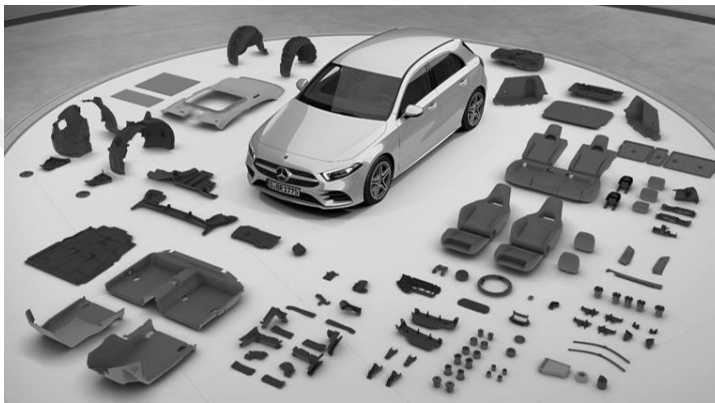


Figure 1.11. Model of the Mercedes-Benz A-class car and its component parts made of natural fiber composite (*New Mercedes-Benz A-Class: Environmental Certificate for the A-Class - Daimler Global Media Site, n.d.*)



2. LITERATURE REVIEW

2.1. Enhancement of Mechanical Properties of Composite material

Since for a long period, scientists are interested to pay their attention on the enhancement of the mechanical properties of composite material. In this chapter, the literature review about enhancement of mechanical properties of composite structures are summarized in four sections according to the extent of the thesis. These are: studies on enhancement of the mechanical properties of composite structures by hybridization, increasing fiber content, changing stacking sequence and fiber surface treatment.

2.1.1. By Hybridization

Many investigations on the influence of additional fiber loading on the mechanical characteristics of natural fiber reinforced hybrid composites have been published. The addition of another natural fiber with comparable high elongation increased the mechanical characteristics of natural fiber reinforced hybrid composites.

2.1.1.1. Natural Fiber- Natural Fiber Reinforced Hybrid Composites

Shanmugam and Thiruchitrambalam examined the mechanical characteristics of a unidirectional palm stalk fiber/jute fiber-reinforced polyester matrix composite. According to the researchers, adding jute fiber to a palm fiber-reinforced polyester composite increased mechanical properties. The hybrid composite P50J50 offers an improvement in tensile and flexural strength of 11 and 28 percent, respectively, as compared to palm polyester composite (Shanmugam and Thiruchitrambalam, 2013).

Alavudeen et al. (2015) investigated the mechanical properties of woven banana/kenaf fiber reinforced polyester composites. The hybridization effect positively detected in the composites. Tensile, flexural and impact strength of hybrid

composites were higher in comparable with either pure banana or kenaf fiber composites.

Venkateshwaran et al. (2011) investigated the mechanical properties of a banana/sisal reinforced epoxy composite. Different weight ratios are used to fabricate composites, with a loading of 30% total fibers in each composite. Tensile strength, flexural strength, and impact strength are all at their highest value in the composite S50B50. In comparison to banana epoxy composite, the composite S50B50 has increased tensile strength, flexural strength, and impact strength by 16, 4, and 35 percent, respectively.

Tensile properties of oil palm empty fruit bunches/jute fiber reinforced epoxy hybrid composites were examined. It was observed in the improvement of tensile strength and tensile modulus of hybrid composite while compared to single empty fruit bunches fiber composites. Excellent interfacial strength was found in hybrid composites than pure oil palm empty fruit bunches composites (Karthi et al., 2019).

Srinivasan et al. (2014) studied the mechanical and thermal properties of banana-flax based epoxy resin hybrid composites developed by hand lay-up technique with a total fiber loading 40%. They found that, hybrid composites exhibited better impact and flexural strength than pure banana/epoxy and flax/epoxy composites.

2.1.1.2. Natural Fiber-Synthetic Fiber Reinforced Hybrid Composites

Karthick et al. (2018) Developed a composite material containing banana fiber, glass fiber, and epoxy resin utilizing a hand lay-up process and evaluated the mechanical properties in terms of tensile, flexural, and impact strength. The fiber length and fiber content of the fabricated composite varied. The result indicated that, properties can be increased with the increase of fiber length and fiber content.

Akil et al.(2010) developed jute/glass/kenaf fiber hybrid composites and investigated the mechanical properties. It was found that, jute-glass based hybrid composite had better flexural strength than kenaf-glass based composite.

Bhoopathi et al. (2015) developed hemp/glass, banana/glass, hemp/banana/glass epoxy laminated hybrid composite material by hand lay-up technique and investigated the mechanical properties in terms tensile, flexural and impact strength. The thickness of composite material was 300 x 300 x 4 mm. The experimental result showed that hemp/banana/glass fiber reinforced epoxy composite exhibited superior mechanical properties than other composite.

Shahzad (2011) showed the effect of hybridization of hemp and glass fiber reinforced unsaturated polyester composite developed by hand lay-up technique followed by compression molding on the tensile and impact properties. The developed composites were such as short hemp fiber mat, chopped strand glass fiber mat and hybrid glass/hemp fiber composite. By Replacing 11% hemp fiber with glass fiber two kinds of fiber configuration were hemp core glass skin and hemp skin glass core were used in the development of composite. The hybrid composite exhibited better tensile properties. Hybrid composite showed increased impact damage tolerance limit.

2.1.2. By Increasing Fiber Content

The mechanical property of composite material depends on fiber loading. In many studies it was observed that mechanical properties of composites increased with the increasement of fiber loading up to certain limit.

Shireesha and Venkata (2019) developed hybrid laminated composite material with jute, banana as reinforcement and epoxy as matrix depending on the varying fiber content. They investigated that tensile and flexural strength of hybrid composite improves as compared to individual banana/jute fiber. The result also showed that in all hybrid and individual fiber composite, tensile and flexural strength gradually increased at 30% fiber loading and turned to decrease at 40% fiber loading.

Dhakal and Keerthi (2017) developed banana fiber reinforced polyester composite material with varying fiber content such as 5%, 10%, 15%, 17.5%, and 20%. The thickness of the composite material was varied as 5mm and 3 mm. The composite material was subjected to tensile, flexural and impact strength. The test result revealed that 5 mm thick composite showed their maximum tensile, flexural and impact strength value at 20% fiber content (Dhakal and Keerthi, 2017).

Sugarcane bagasse fibers were utilized in the polypropylene composite by hot pressing technique to investigate the effect of optimum fiber loading on the mechanical properties such as tensile, flexural and hardness. It was revealed that with the increase of fiber content up to 30%, improvement in mechanical properties were significantly (Subramonian et al., 2016).

Fibers of different length and weight percentage 8, 12, 16 and 20% were used along with epoxy resin to develop composite material. The prepared composites were subjected to tensile, flexural and impact strength test and it was investigated that 15mm fiber length 20% fiber content showed highest tensile, flexural and impact strength (Venkateshwaran et al., 2011).

Krishna (2017) developed composite fabricated of randomly oriented short jute/banana fiber with varying fiber content and effect on tensile, flexural and impact strength were studied. It showed that optimum fiber volume fraction for tensile and flexural strength is found to 15%. Impact strength increase with the increase of fiber content up to 25%.

Rashed et al. (2006) developed jute fiber reinforcement polypropylene composite by hot compression molding technique with the varying process parameter such as fiber condition (treated and untreated), fiber sizes, (1, 2, 4 mm) fiber wt.% (5%, 10% and 15%). The produced composite was characterized by tensile strength. The observation was that the tensile strength increased with the increase of fiber length and fiber wt.% up to certain limit and after that it decreased, and treated fiber had no significant effect. The tensile strength was increased up to

2mm fiber length and 10% fiber wt.% and then it decreased only with the exception of 5% treated fiber.

Komal et al. (2018) developed Polypropylene composite material incorporating untreated and treated (5% NaOH) with varying fiber content (10, 20, 30).They investigated the mechanical properties such as tensile modulus, tensile strength, flexural modulus, flexural strength and observed that tensile modulus increased with the incorporating of fiber loading compared to neat PP, reached to the maximum at the 30% fiber loading. No significant improvement was occurred with the addition of treated fiber. The highest tensile strength gradually decreased with the increases of fiber loading with the marginal improvement in case of treated fiber. They also observed that highest flexural modulus and flexural strength occurred at 30% treated banana fiber.

Jute fiber reinforced polypropylene composites were developed by injection molding technique. Both raw jute and oxidized jute fibers were utilized in composite material at four level of fiber loading such as (20%, 25%, 30% and 35% wt). According to the fiber loading, 30% fiber reinforced composites had optimum mechanical properties (Rahman et al., 2008).

2.1.3. By Changing Stacking Sequences

Venkateshwaran and Elayaperumal (2012) used a hand lay-up method followed by compression molding to develop a jute banana polyester hybrid composite and examined the effects of layering sequences on mechanical parameters like as tensile, flexural, and impact strength. Among various hybrid and individual fiber composite materials, banana jute banana composite material had the highest tensile and flexural strength. The impact strength of the jute banana jute hybrid composite material was higher.

Singh et al. (2019) used compression molding to develop a jute, linen polypropylene hybrid composite with several layering patterns, such as jute-linen-jute, linen-jute-jute, jute-linen-linen, and linen-jute-linen. Linen and jute fibers were

treated by sodium hydroxide. They investigated the maximum tensile and flexural strength of jute-linen-jute/pp and a linen-jute-linen/pp hybrid composite, respectively. The tensile and flexural strength of the jute-linen-linen/pp hybrid composite were the lowest.

Sanjay et al. (2016) developed E glass fabrics/ hemp fabrics reinforced polyester composite by hand lay-up vacuum bagging method. Six different kinds of composite were prepared as per layering sequence. The composites were subjected to mechanical tests such as tensile, flexural and impact strength. The experimental result investigated that hemp glass fabric laminate hybrid composite can increase the mechanical properties.

2.1.4. By Surface Treatment

Many investigations were carried out on the mechanical properties of treated fibers reinforced composites, and it was observed that treated fiber composites had better mechanical properties than its untreated fiber composites counterpart.

Vardhini et al. (2019) investigated the physical, morphological, chemical composition of raw banana fiber and alkali NaOH treated banana fiber at three different concentrations like 10%, 15% and 20%. They observed that density, water absorption gradually decreased till 15% NaOH concentration and after that at 20% concentration it increased. They also observed that cellulose content increased up to 15% concentration and after that it degraded. As a result tenacity and breaking strength increased till 15% concentration and decreased at 20% concentration.

Suardana et.al (2012) investigated the effect of chemical treatment on the hemp fibers and mechanical properties of hemp fiber reinforced composites. Among the tested samples they observed that chemically modified hemp fiber reinforced composite exhibited higher mechanical properties.

Bhoopathi et al. (2015) developed hemp-banana-glass fiber reinforced untreated and NaOH treated hybrid composite with epoxy material by hand layup technique and observed the mechanical properties in terms of tensile, flexural and

impact properties. From the experimental results it has been noted that treated hemp-banana-glass fiber reinforced epoxy hybrid composite exhibited superior mechanical properties.

Vekateshwaran et al. (2013) fabricated composite with banana fiber and reinforced into epoxy matrix. The fibers were treated by NaOH solution at different concentration. They found that flexural strength of treated fiber composite was higher than untreated composite.

Siregar et al. (2009) investigated the effect of NaOH treatment on mechanical properties of pineapple leaf fiber reinforced high impact polystyrene composites in terms of tensile strength, tensile modulus, flexural strength, flexural modulus and impact strength. The fibers were treated at different concentration (0%, 2%, 4%) NaOH solution. Mechanical tests of treated fiber composites were conducted and compared to the untreated composites. It was evaluated that, all mechanical properties of treated PALM fiber composites exhibited higher than that of untreated fiber composites.

Hariprasad et al. (2013) developed banana coir epoxy hybrid composite material by hand lay-up technique and investigated the mechanical properties in terms of tensile, flexural and impact strength. Composite plates were prepared with resin 392 g, coir 54 g and banana 69 g. It was investigated that tensile and impact strength treated hybrid composite were higher than those of untreated composite. The flexural strength of untreated composite was higher than treated composite.



3. MATERIALS AND METHODS

3.1. Materials

Hemp fiber was supplied by local factory in Adana, turkey. Jute fibers was supplied by Erkaplan İplik Halı Tekstil Gıda San ve Tic. Ltd. Şti. /Gaziantep/Türkiye. Unsaturated polyester resin and di-methyl ethyl ketone peroxide as a hardener were purchased from YONTAR T.A.Ş. in Adana/Türkiye, a local chemical shop in Adana, Türkiye.

3.2. Methods

Hemp and jute fibers were used in layer by layer in composite material. The two layers were J/H/H/J and H/J/J/H. Three fiber volume fraction was used to produce the composites such as 30% fiber, (15% jute; 15% hemp) 50% fiber, (25% jute; 25% hemp) 70% fiber (35% jute; 35% hemp). Some fibers were subjected to NaOH treatment prior to composite fabrication. According to the treatment, 3 types of fibers were used for the production of composite plate, such as untreated, 3% NaOH and 5% NaOH treated fibers. Thus 18 composite plates were produced. Table 3.1 shows three different factor such as fiber layering sequence, NaOH percentage, fiber volume fraction and level in each factor. Table 3.2 shows the proportional distribution of jute and hemp fibers within the natural fiber ratio in the composite, separately for each composite sample.

Table 3.1. Production of composites by using fiber layering pattern, fiber volume fraction and NaOH percentage.

Factor Number	Name of the factor	Level	Description
1	Fiber Layering Pattern	2	J/H/H/J, H/J/J/H
2	Fiber volume fraction %	3	30%, 50%,70%
3	Percentage of sodium hydroxide.	3	0% (untreatment),3%NaOH, 5%NaOH

Table 3.2. Composites sample

Sample no	Sample Code	Description
1	U (J/H/H/J)30	Untreated 30% fiber (15%J, 15%H)
2	U (J/H/H/J)50	Untreated 50% fiber (25%J, 25%H)
3	U (J/H/H/J)70	Untreated 70% fiber (35%J, 35%H)
4	U (H/J/J/H)30	Untreated 30% fiber (15%J, 15%H)
5	U (H/J/J/H)50	Untreated 50% fiber (25%J, 25%H)
6	U (H/J/J/H)70	Untreated 70% fiber (35%J, 35%H)
7	3%T (J/H/H/J)30	3% NaOH treated 30% fiber (15%J, 15%H)
8	3%T (J/H/H/J)50	3% NaOH treated 50% fiber (25%J, 25%H)
9	3%T (J/H/H/J)70	3% NaOH treated 70% fiber (35%J, 35%H)
10	3%T (H/J/J/H)30	3% NaOH treated 30% fiber (15%J, 15%H)
11	3%T (H/J/J/H)50	3% NaOH treated 50% fiber (25%J, 25%H)
12	3%T (H/J/J/H)70	3% NaOH treated 70% fiber (35%J, 35%H)
13	5%T (J/H/H/J)30	5% NaOH treated 30% fiber (15%J, 15%H)
14	5%T (J/H/H/J)50	5% NaOH treated 50% fiber (25%J, 25%H)
15	5%T (J/H/H/J)70	5% NaOH treated 70% fiber (35%J, 35%H)
16	5%T (H/J/J/H)30	5% NaOH treated 30% fiber (15%J, 15%H)
17	5%T (H/J/J/H)50	5% NaOH treated 50% fiber (25%J, 25%H)
18	5%T (H/J/J/H)70	5% NaOH treated 70% fiber (35%J, 35%H)

3.2.1. Fibers Treatment by NaOH (Alkali)

Adhesion between the fiber and polymer is one of parameter influencing the mechanical properties of composites (Reddy & Dhoria, 2018). Natural fibers have a number of advantages over synthetic fibers, but one noticeable disadvantage is that they are hydrophilic. Because of this, there is a weak adhesion between the fiber and the matrix, which reduces the composite's properties. This drawbacks can be overcome by chemically altering the fiber surface to make it less hydrophilic (Venkateshwaran, Perumal, et al., 2013).

Several researchers investigated that chemical modifications of natural fibers aimed at improving the adhesion with a polymer matrix (X. Li et al., 2007). When natural fibers are used to reinforce thermoplastics and thermosets, alkaline treatment, also known as mercerization, is one of the most used chemical treatments. The most important change caused by alkaline treatment is the breaking of hydrogen bonding in the network structure, which increases surface roughness. By removing

a certain amount of lignin, wax, and oils from the external surface of the fiber cell wall, this process depolymerizes cellulose and exposes the short length crystallites (Mohanty et al., 2001). The NaOH treatment of jute fibers is one of the ways that is frequently used to improve the mechanical properties of jute reinforced composites. Many studies are reported in the literature about this subject (Doan et al., 2012; Karaduman and Onal, 2013; Liu et al., 2009; Ray et al., 2001; Saha et al., 1999; Sinha and Rout, 2009). Many researcher reported about the positive effect of alkali treated hemp fibers on the mechanical properties of hemp fiber reinforced composites (Kabir et al., 2013; Kabir et al., 2020; Le Troëdec et al., 2011; Serra-Parareda et al., 2020; Shah et al., 2021; Vilaseca et al., 2020). In alkaline treatment, fibers are immersed in NaOH solution for a given period of time (X. Li et al., 2007). Different duration time were reported by the researchers in the literature. Ray et al. treated jute and sisal fibers with 5% aqueous NaOH solution for 2 h up to 72 h at room temperature (Ray et al., 2001). Similar treatments were also attempt to treat jute and sisal fibers by Mishra et al. (Mishra et al., 2001).

In this work, first, required amount of NaOH was taken in a big beaker containing distilled water and continue stirring to make 3% or 5% NaOH stock solution. From that solution, necessary amount was taken into a small beaker to immerse fibers to maintain M:L ratio 1:20. Materials were kept in this condition for 1 hour. Meanwhile pH was measured by pH meter, and it was found 10. After that, materials were rinsed thoroughly by distilled water. Materials were then immersed into 1% acetic acid solution, for 10 minutes, while maintaining pH=7, washed thoroughly by running water to remove remaining acetic acid adhered to the fiber. Excess water was squeezed out from the fiber followed by drying in a woven dryer while increasing the temperature gradually until it reaches at 80°C. It was 80 mins to continue the machine to ensure proper drying.

3.2.2. Single Fiber Tensile Test

The mechanical performance of individual fiber was evaluated using the fiber tensile tester (Instron 1185 load cell 10 N) at the crosshead speed of 2 mm/ min at room temperature ($20 \pm 2^\circ\text{C}$) and ($65\% \pm 4$) relative humidity. The gauge length was 20mm. 20 fibers were tested, and the average value was reported. Both treated and untreated jute and hemp fibers were subjected to tensile strength.

3.2.3. Carding

After drying, treated fibers and untreated fibers were subjected to Miniature carding Machine. This machine is used in the laboratory for sample sliver production. The application of this machine is appropriate only when 5-10 g weight of the material is vital important for experimental work.

Untreated fibers remained in disentanglement state. In the treated fibers disentanglement increased due to severe rinsing and washing. Fibers were not state and parallel in the sample. Due to this lack of parallelization, Uniform mixing of fiber and matrix would not be possible resulting in improper fabrication of composites materials. For this reason, a miniature carding machine was used to

- To make the fiber alignment
- To even out fiber density
- To ensure uniform length distribution
- To produce continuous sheet of fibers.
- To increase fiber volume
- To produce web of fiber.

Several researchers reported about the positive effect of fiber carding on the mechanical properties of fiber reinforced composites. Yin et al. reported about the positive influence of carding process on the mechanical properties of carbon fiber

reinforced thermoplastics composites (Yin et al., 2017). Park et al. fabricated 10 to 90% increased lyocell fiber with the polylactic matrix through a surface hydrophobic treatment using phenyl silane and fiber carding/melt pressing. The fibers in the composites were oriented using the carding procedure. To confirm the impact of the fiber directionality of the composite on its mechanical properties, cross direction (CD) and machine direction (MD) samples were created. The MD samples outperformed the CD samples in performance (Park et al., 2019). Lee et al. developed bio-composites comprised of kenaf fiber reinforced polylactide are fabricated by carding followed by treatment with a 3-glycidoxypropyl trimethoxy silane and hot-pressing (Lee et al., 2009).



Figure 3.1. Miniature carding machine

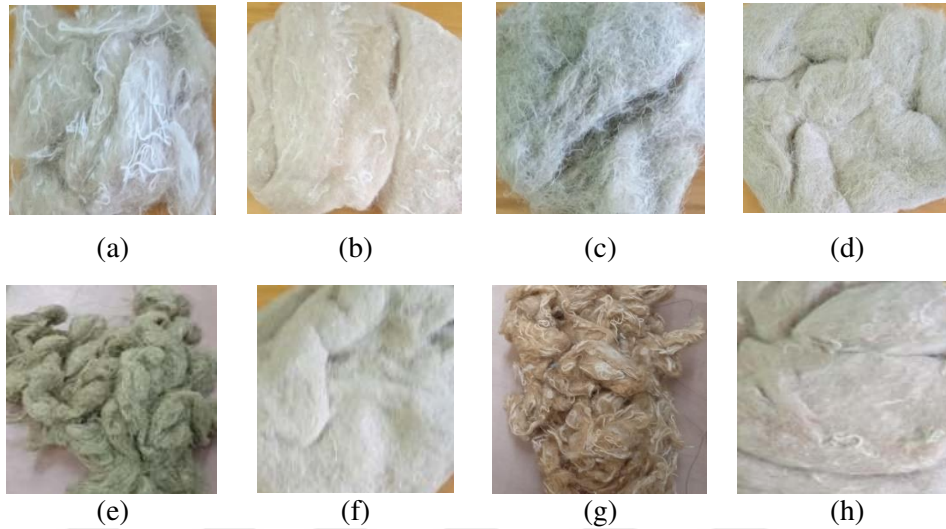


Figure 3.2. (a)Untreated jute fibers. (b)Untreated carded jute fibers (c)Untreated hemp fibers. (d)Untreated carded hemp fibers. (e)NaOH treated hemp fibers (f)NaOH treated carded hemp fibers(g)NaOH treated jute fibers. (h)NaOH treated carded jute fibers.

3.2.4. Fabrication of Composites

There are numerous fiber-reinforced plastic manufacturing technique. The choice of process depends on many factors, such as type of reinforcement and matrix materials, size, shape, quantity, and cost. There are many specialized processes available, but hand lay-up method, cheapest, easiest, only the most commonly used commercial process, used here in the preparation of the composites. This method have been used by many researchers (Alzebdeh et al., 2019; Arun et al., 2012; Balaji et al., 2015; Chaudhary et al., 2018; Dharmalingam et al., 2018; Hasan et al., 2016; Hossen et al., 2017; Jawaaid et al., 2013; Kumar and Srivastava, 2017; Manjunath and Rao, 2013; Mbeche and Omara, 2020; Munikenche Gowda et al., 1999; Panneerdhass et al., 2014; Patel et al., 2009; Sanjay et al., 2016; Sci, 2009; Yahaya et al., 2015). 18x18x4cm steel mould was used to fabricate the composite material. First 1% hardener thoroughly mixed to the resin and stirred properly until to form paste coat in a jar. The time was taken to form paste was 5 minutes. Care was taken

to avoid formation of bubble. Mold releasing paper placed over the bottom surface of the mold. A little portion of resin paste applied to the paper slowly. Then ply of jute fibers placed on the matrix layer which was covered by matrix layers poured again slowly on the fiber surface. Then ply of hemp fibers were placed on the matrix layer. Subsequently, a steel hand roller was used to roll the wet composite to ensure an enhanced interaction between the reinforcement and the matrix, to facilitate a uniform resin distribution, to remove any entrapped air between fiber and resin and to obtain the required thickness. By this way four layers of fibers were laminated. At last, remaining large portion of resin applied to the fibers to fabricate stacking laminated composites (J/H/H /J). Another stacking laminated composite (H/J/J/H) was produced similarly. Another mould releasing paper placed over the top fiber layer in the mold. Upper part of the mould placed on the top with a weight 10 kg for uniform pressure and fabricated composite was allowed to cure at room temperature for 4-5 hours. Some of the polyester and hardener mixture was squeezed out under the application of pressure. When preparing composites, special care was taken to account for this loss. The lamination was 9.5-10 mm thick on average.

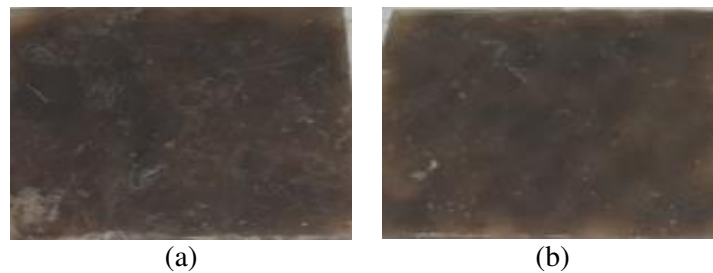


Figure 3.3. (a)J/H/H/J layered composites. (b)H/J/J/H layered composites

3.2.5. Thickness Testing

Produced composites were subjected to thickness tester to measure the thickness. 10 measurements were taken from different place of the composite and average result was reported.

3.2.6. Mechanical Properties

The mechanical properties of materials, such as tensile properties, flexural properties, impact properties, are always used to describe the composites' performance. These properties are vital in determining material capabilities, particularly under extreme and critical situations, which are linked to engineering performance (H. M. Akil et al., 2011).

3.2.6.1. Tensile Strength Test

The most basic type of mechanical test done on any material is a tensile test, often known as a tension test. Tensile tests are simple, relatively inexpensive, and fully standardized. As the material is being pulled, it is possible to establish its strength together with how much it will elongate. The material's point of failure is of great interest, and it's usually referred to as its "Ultimate Tensile Strength" (UTS) (T. Hariprasad, Dharmalingam and Raj, 2013). Tensile testing provides these useful data: tensile yield strength, ultimate tensile strength, Young's modulus, and elongation at yield and break.

The tensile strength (σ) is given by

$$\sigma = \frac{F}{bh}$$

where F: load

b: width of the sample

h: thickness of the sample

Strain or elongation is defined as:

$$\varepsilon = \Delta l / l_0$$

where Δl : is the extension

l_0 : the initial gauge length

The Young's modulus in tension (E_t) is the slope of the stress vs. strain curve evaluated at small strains, where the response is linear.

The tensile test specimens are prepared, and testing of the composite laminates are carried out as per ASTM D 638 standards and procedures. There are four specimens are used from each laminate for testing tensile behavior of hybrid laminates. The test has been carried out on the Zwick/Roell Z010 (BL-GRWO10K) universal testing machine, by means of applying load on the specimen until it gets failure. The test speed was 5mm/min. The gauge length was 115 mm. Same approaches are applied to the other specimens in the same composite laminate as well as other laminate specimens to calculate the mean tensile strength.



Figure 3.4. Tensile test specimen

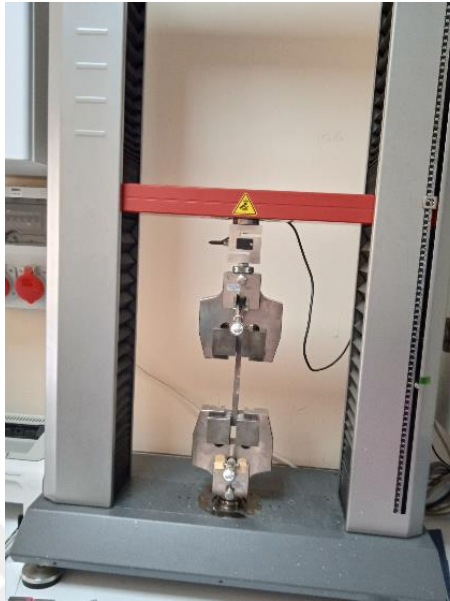


Figure 3.5. Tensile strength testing

3.2.6.2. Flexural Strength Test

The ability of a material to resist deformation under load is known as flexural strength. It's a three-point bend test that usually leads to inter-laminar shear failure (Santhosh, 2014). The modulus of rupture, commonly known as bend strength or fracture strength, is a mechanical parameter for brittle materials. The most common test is the transverse bending test, which involves bending a rod-shaped specimen with a circular or rectangular cross section until it fractures using a three-point flexural test procedure. A material's flexural strength is the maximum stress it can withstand at the time of rupture. The flexural strength would be the same as the direct tensile strength if the material was homogeneous. Most materials, in fact, have minor or substantial flaws that cause stresses to concentrate locally, resulting in localized weaknesses. When a material is bent, only the extreme fibers are subjected to the maximum stress; thus, assuming those fibers are defect-free, the flexural strength will be dictated by the strength of those intact fibers. If the same material is exposed to direct tension, all of the fibers in the material are subjected to the same stress, and

failure will initiate when the weakest fiber reaches its limiting tensile stress. As a result, flexural strengths for the same material are frequently higher than direct tensile values. on the other hand, A homogeneous material with only surface defects, have a higher direct tensile strength than flexural strength (Hariprasad et al., 2013).

The bending modulus (E_b) and bending strength (σ_b) are calculated the following equations:

$$E_b = \frac{mL^3}{4bh^3}$$

$$\sigma_b = \frac{3FL}{2bh^2}$$

where E_b : bending modulus

σ_b : bending strength

F: load

m: initial slope of the load vs. deflection curve

L: specimen length between two support points

The flexural test specimens are prepared as per the standard methods of ASTM D 790. The four test specimens of each laminate hybrid reinforced polyester composites are prepared and tested by applying the 3 point flexural load with the help of same universal testing machine, Zwick/ Roell Z010 (BL-GRWO10K). The test speed was 2mm/min. The specimen length between two supporting point was 100mm. The average result of the four specimen was taken into consideration. The result of each sample is observed for result comparison.

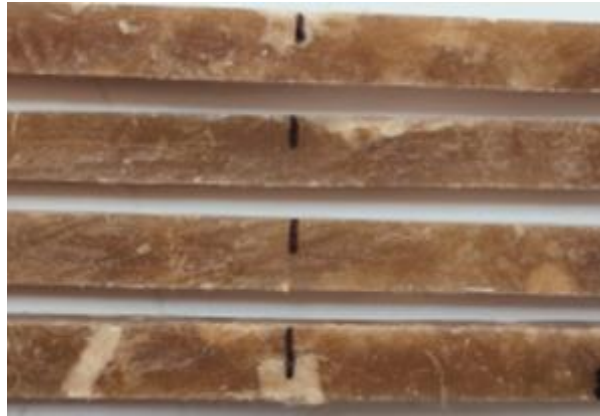


Figure 3.6. Specimen for flexural strength



Figure 3.7. Flexural strength testing

3.2.6.3. Impact Strength Test

The ability of a material to resist breaking under shock loading or to withstand fracture under high-speed stress is known as impact resistance. One of the most stated mechanical features of engineering materials is impact behavior (Srinivasa & Bharath, 2011).

The impact test is used to determine how a specimen of a known material will react to a sudden force. The test determines if the material is fragile or tough. In most cases, a notched test piece is used, and the Izod and Charpy tests are the most commonly used methods. The result is usually reported as the energy in ft.lbs. or KJ required to fracture the test piece. The impact performance is affected by molded-in stresses, polymer orientation, weak points (e.g., weld lines or gate sections), and part geometry. When additives, such as coloring agents, are added to plastics, their impact properties change as well.

$$a_c U = \frac{W}{hb} \times 10^3$$

Where W: energy

b: width of the sample

h: thickness of the sample

Impact strength testing was performed using the Zick/Roell (HIT5.5P) Izod impact strength tester. The four specimens were made from each composite laminate to test the material's impact load bearing capabilities. The test specimens were made from a hybrid composite laminate in accordance with ASTM D 256, and the specimens' edges were neatly finished, with small "v" notches were provided using a RAY-RAN motorized notch cutting machine. During the test, the maximum energy that can be stored to break the specimens is recorded for analysis of result.

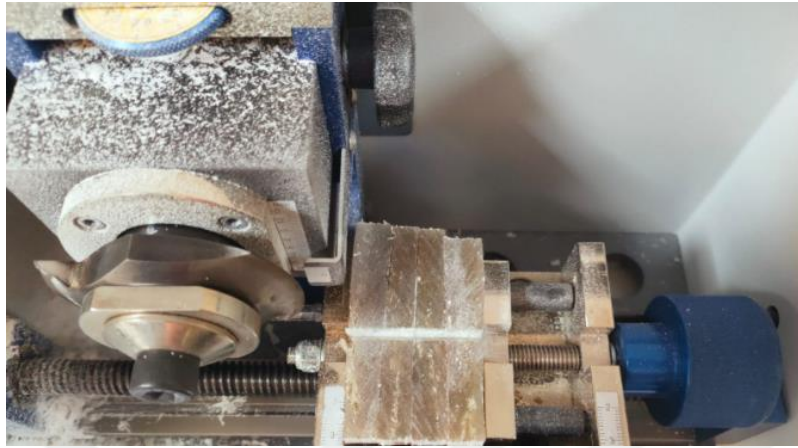


Figure 3.8. RAY-RAN motorized notch cutting machine.



Figure 3.9. Specimen for impact strength testing



Figure 3.10. Impact strength testing

3.2.7. ANOVA and t-test

Analysis of variance test was done to observe the overall effects of various factor Like fiber content and NaOH% on tensile properties, flexural properties and impact properties. The implementation of t-test was done to observe the difference between two layering sequences. Experimental analysis of ANOVA and t-test were implemented by using SPSS software. These analyses were undertaken for a level of confidence of significance of 5%.

3.2.8. Scanning Electron Microscopy (SEM)

SEM analysis is used to inspect and analyze morphological analyses in composites such as interfacial bonding, matrix dilation, fiber pulling, and fracture between the fiber and matrix (Prabhu et al., 2019). Morphological studies were performed on the untreated and treated hemp and jute fiber. SEM examination also performed on the fractured surface of all mechanically tested samples.

A scanning electronic microscope (SEM) (FEI Quanta FEG 650) was used to examine the cross-section and fiber surface at a voltage of 20 kV. The sample was coated with a thin layer of gold to provide electrical conductivity, which significantly affects the resolution of the images. In a scanning electron microscope (SEM), a highly focused scanning electron beam bombards the surface, producing a huge number of secondary electrons to be created, the intensity of which is governed by surface topography. The process can be used on any material, although nonconducting materials must have a thin conductive layer of sputtered gold applied to them, which can modify or hide the true surface shape. Topographical features have a resolution of about 5 nanometers. Before using more sophisticated techniques, SEM is frequently used to survey a surface.



Figure 3.11. FEI Quanta model FEG 650 seam analyzer.

3.2.9. Energy Dispersive X-ray Spectroscopy Analysis (EDX)

The EDX requires an analytical technique to investigate the chemical composition formed by fiber surfaces. This approach can detect the key chemical components of fiber, such as C and O, as well as Na, Al, Si, and Mg. However, it is unable to detect H, which is one of the most important natural fiber elements (Ali et

al., 2018; Rashid et al., 2016). It is based on an interaction between an X-ray source and a sample. Its characterization powers are largely attributable to the fundamental premise that each element has a unique atomic structure, resulting in a distinct collection of peaks on its electromagnetic emission spectrum. (Which is spectroscopy's main principle.) Mosley's law accurately predicts peak positions, far better than the experimental resolution of a common EDX equipment.

The chemical composition of both treated untreated hybrid composites were measured on the same machine (FEI Quanta FEG 650) used for SEM analysis. The analysis was carried out at an accelerating voltage of 20 kV.

3.2.10. XRD

XRD is a rapid and non-destructive analytical technique for figuring out the chemical composition and crystallographic structure of natural fibers. By bombarding a substance with incoming X-rays, the XRD technique measures the X-rays' intensities and scattering angles as they leave the object (Y. Liu & Hu, 2008; Madhu et al., 2019; Mannan, 1993; *What Is X-Ray Diffraction Analysis (XRD) and How Does It Work?*, 2022).

X-ray crystallography is an experimental science that identifies the atomic and molecular structure of a crystal by diffracting an X-ray beam incident on it in a variety of directions due to the crystal's structure. By calculating the angles and intensities of these diffracted beams, a crystallographer can produce a three-dimensional representation of the density of electrons within the crystal. Using this electron density, one may determine the average locations of the atoms in the crystal as well as their chemical bonds, crystallographic disorder, and other details (Contributors to Wikimedia projects, 2022).

The natural fibers (processed) can be scanned in a 2θ range, ranging from 10° to 50° , using X-ray diffraction. The diffraction peaks of the amorphous and crystalline areas can be seen in the spectrum obtained from the measurements for a certain fiber. The crystallinity index (CI) is computed from the resulting X-ray

diffractogram using the formula: $CI = (1 - \frac{I_{AM}}{I_{000}}) \times 100$ where I_{000} and I_{AM} is the intensity of crystalline phase, and the amorphous phase respectively. The crystallite size (CS) could be calculated using the equation; $CS_{000} = \frac{0.89\lambda}{\beta_{000} \cos\theta}$, where β is the full-width at half-maximum of the peak, while θ is the Bragg angle (Seki et al., 2013). X-Ray machine panalytical model empyrean was used to identify the image of crystallography.



Figure 3.12. X-Ray machine (Panalytical model Empyrean)

4. RESULTS AND DISCUSSION

4.1. Single Fiber Tensile Strength

The measurement of tensile strength of single fiber is important because it affects the mechanical properties of the composite. The attribution of poor mechanical properties in the composite results from the reinforcement of weak fiber and vice-versa. Table 4.1 showed the result of single fiber tensile strength.

It was observed from the Table 4.1 that, untreated jute and hemp fibers showed lower tensile strength than treated jute and hemp fibers. Because of the increased cellulose% percentage and crystallinity in the treated fiber due to removal of hemicellulose, lignin and waxy layer from the fiber surface by alkali treatment.

Similar result was found by several researchers. Sawpan et al. (2011) treated hemp fibers by NaOH and observed the fiber structure and mechanical properties. They found that, treated hemp fibers exhibited higher tensile strength than untreated fiber.

The physio-chemical characteristics of jute fibers treated with alkali (NaOH) solution were examined by Saha et al. (2010) The treatments were carried out at room temperature and at increased temperatures, including high-pressure steaming. According to the findings, soaking the fibers in a 0.5 percent alkali solution for 30 minutes followed by 30 minutes of alkali-steam treatment increases the tensile strength by up to 65 percent.

Table 4.1. Result of single fiber tensile strength testing

Fiber type	Tensile strength (cN)	CV%
Untreated jute	96.4	44.2
3% treated jute	120	65.8
5% treated jute	116.5	60.5
Untreated hemp	76.2	48.7
3% treated hemp	105	56.7
5% treated hemp	98.3	51.8

4.2. Thickness Testing

Table 4.2 shows the average value of thickness in mm at ten different places of the composite.

Table 4.2. Average value of thickness measurement (mm)

Sample no	Measuring points										
	1	2	3	4	5	6	7	8	9	10	Average value (mm)
1	9.14	11.44	9.88	9.14	10.28	11.5	11.05	8.17	10.22	11.4	10.222
2	12.27	11.75	11.08	10.05	8.28	10.43	11.46	10.33	10.28	9	10.493
3	11.88	12.31	11.91	10.42	10.63	9.94	9.77	10.68	11.55	11.14	11.023
4	10.08	10.93	10.96	10.72	11.16	11.33	11.89	11.41	11.85	12.32	11.265
5	10.06	12.2	10.4	9.41	9.94	10.94	10.67	10.66	10.2	11.3	10.578
6	8.14	10.53	10.06	10.25	10.96	11.72	12.4	11.4	12.09	13.3	11.085
7	10.5	10.26	10.71	9.7	9.2	9.3	9	9	9.3	9.28	9.625
8	9.45	9.43	9.39	10.43	10.21	10	9.5	9.98	10.7	10.17	9.97
9	9.94	9.78	9.21	9.28	9.2	9.46	9.86	9.78	9.26	9.3	9.507
10	10.26	10.8	11.25	9.31	10	9.36	8.5	7.84	8.83	9.34	9.54
11	10.89	10.14	9.75	9.43	9.68	9.76	10.28	9.34	9	8.75	9.702
12	10.7	10	9.5	10.07	10.63	11.05	11	8.68	11.01	10.38	10.30
13	10	11.1	12.26	13.68	12.62	11.09	10.1	9.06	10.28	11.86	11.20
14	9.46	9.56	9.22	9.03	9.24	9.6	10.07	10.04	9.84	10.85	9.69
15	9.66	11.73	10.27	9.55	9.03	10.14	11.62	11.53	11.09	10.43	10.50
16	11.03	10.35	10.18	10.74	10.05	10.39	10.35	9.5	10	9.38	10.19
17	11.38	10.43	10.58	9.91	10.31	10.88	11.15	11.71	11	10.07	10.74
18	11	11.17	11.09	10.05	10.79	10.7	9.77	9.57	9.79	10.5	10.44

4.3. Tensile Properties of The Composites

Tensile properties is the resistance of a material when it is subjected to a certain amount of axial load. Tensile strength and tensile modulus are the significant tensile properties for composite material. Choosing a right application area depends on the selection of appropriate tensile properties of the composite.

4.3.1. Tensile Strength

Tensile strength is a important tensile properties which is the ratio of breaking load to the cross-sectional area of the composite. The fiber content%, type of surface treatment, concentration% of applied chemical agent, alteration of fiber layering

pattern are the important process parameter which influences tensile strength. Table 4.3 represented the mean tensile strength in which extreme left, middle and extreme right columns were used for defining sample number, sample code and tensile strength. In defining sample code, U, 3%T, 5% T stands for 0% NaOH, 3% NaOH and 5% NaOH treatment respectively. J/H/H/J goes for Jute/Hemp/Hemp/Jute and H/J/J/H for Hemp/Jute/Jute/Hemp. 30, 50 and 70 defined the fiber volume fraction in percentage. These notations were followed by every table where composite plate is defined against different mechanical properties

Table 4.3. Mean tensile strength of the composite plates

Sample no	Sample code	Tensile strength (MPa)
1	U (J/H/H/J)30	23.40
2	U (J/H/H/J)50	16.44
3	U (J/H/H/J)70	14.49
4	U (H/J/J/H)30	17.02
5	U (H/J/J/H)50	12.84
6	U (H/J/J/H)70	10.07
7	3%T (J/H/H/J)30	29.06
8	3%T (J/H/H/J)50	20.91
9	3%T (J/H/H/J)70	18.32
10	3%T(H/J/J/H)30	20.88
11	3%T(H/J/J/H)50	18.74
12	3%T(H/J/J/H)70	13.46
13	5%T (J/H/H/J)30	25.29
14	5%T (J/H/H/J)50	18.45
15	5%T (J/H/H/J)70	16.33
16	5%T(H/J/J/H)30	18.38
17	5%T(H/J/J/H)50	14.87
18	5%T(H/J/J/H)70	11.64

4.3.1.1. Effect of Fiber Content on The Composites' Tensile Strength

Composite containing 30% fiber showed highest tensile strength among other composites contained 50% and 70% fiber. In the above Figure 4.1 and 4.2, J/H/H/J and H/J/J/H layered composites contained 30% fiber reached highest tensile strength among others respectively. In the untreated J/H/H/J and H/J/J/H layered composites, 30% fibers revealed 42% and 61%, 32% and 69% more tensile strength

than 50% and 70% fibers contained composites respectively. In case of 3% treated and 5% treated composite, in the J/H/H/J layered and H/J/J/H layered, 30% fibers showed 18%, 38% and 11%, 55% and 37%, 54% and 23%, 57% more strength than same counterpart respectively. This is due to the fact that, composites contained higher amount fiber were not properly wetted by resin for which load was not properly distributed among the fibers in the resin.

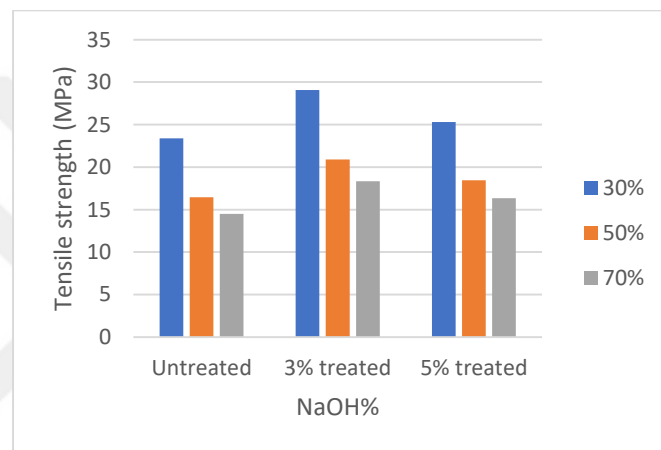


Figure 4.1. Tensile strength of different fiber content of (J/H/H/J) layered composite.

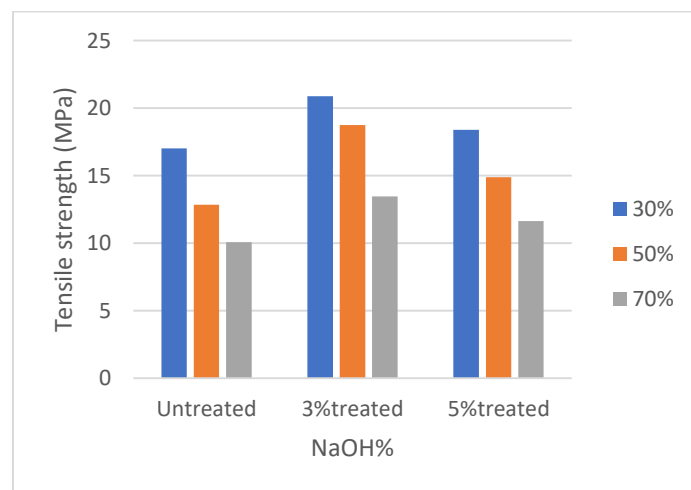


Figure 4.2. Tensile strength of different fiber content of (H/J/J/H) layered composite.

4.3.1.2. Effect of NaOH% on The Composites' Tensile Strength

In the Figure, 4.3 and 4.4, in all fiber content, 3% treated composite achieved highest tensile strength. In 4.3 Figure, at 30, 50 and 70% fiber content, 3% treated composites exhibited 24% and 14%, 27% and 13%, 26% and 12% more tensile strength than untreated and 5% treated composites respectively. In the Figure 4.4, at 30, 50 and 70% fiber content, 3% treated composites exhibited 22% and 13%, 45% and 26%, 33% and 29% more tensile strength than untreated and 5% treated composites respectively.

According to the literature, due to the presence of non-cellulosic compound in natural fibers, untreated fibers and their composites had low tensile strength, (Goriparthi et al., 2012) due to poor wettability and incompatibility of NFs with matrix phase (Gomes et al., 2007).

Alkali treatment increased the surface roughness of fiber. Rough surface enriched wettability of fiber. Surface treatment creates interlocking of fiber to the matrix. After treatment, diameter reduced which caused better penetration of matrix into the fiber. The achievement of greater strength in the treated fiber may be attributed due to the above-mentioned reasons.

Akhtar et al. reported that, treatment of kenaf fibers with 6 wt% NaOH solution improved the fiber roughness of fiber surface (Akhtar et al., 2016). In addition, Subasinghe et al. conducted a study in which, kenaf fibers were treated with NaOH solution and observed that, treated kenaf/PP composite leads to increment in tensile strength and modulus by 1.34% and 40% respectively as compared to untreated Kenaf/PP composites respectively (Subasinghe et al., 2015).

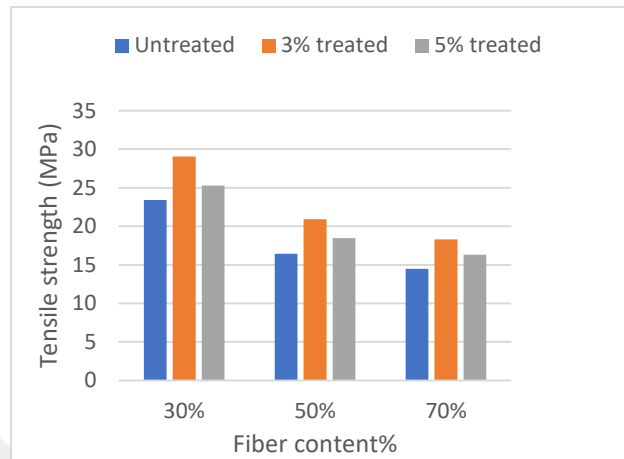


Figure 4.3. Tensile strength of (J/H/H/J) layered composites at different NaOH%.

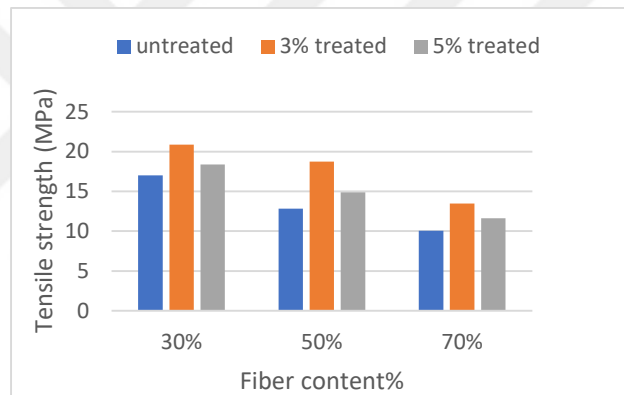


Figure 4.4. Tensile strength of (H/J/J/H) layered composites at different NaOH%.

4.3.1.3. Effect of Layering Sequence on The Composites' Tensile Strength

In all fiber content, J/H/H/J layered composites possess higher tensile strength than H/J/J/H composites. At 30% fiber content, in the untreated, 3% treated and 5% treated composites, J/H/H/J stacking sequences obtained 37%, 39% and 37% more tensile strength than H/J/J/H stacking sequence respectively. In the Figure 4.6 and 4.7, in case of untreated, 3% treated, 5% treated composites, J/H/H/J layered composites possess 28%, 11%, 24% and 43%, 36%, 40% more strength than H/J/J/H composites.

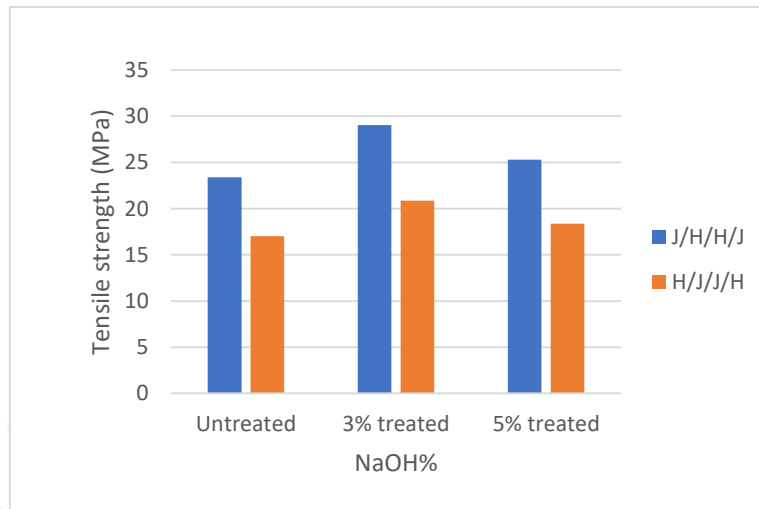


Figure 4.5. Tensile strength of different layered composites at 30% fiber content.

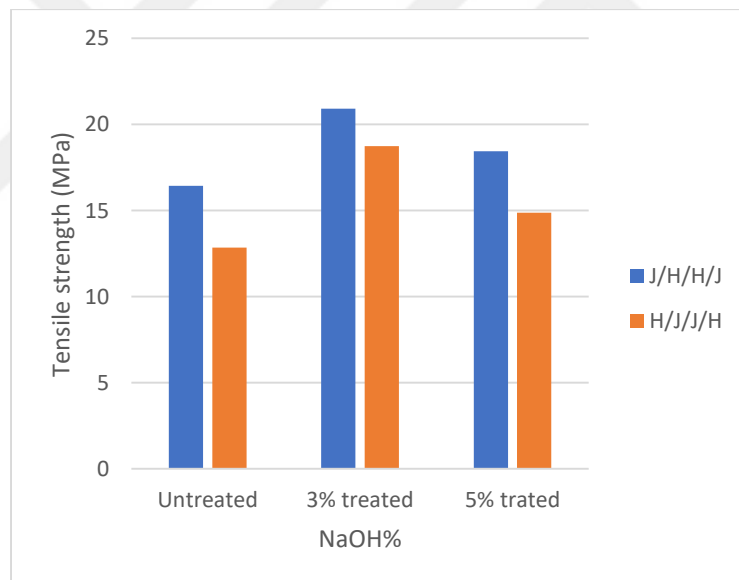


Figure 4.6. Tensile strength of different layered composites at 50% fiber content

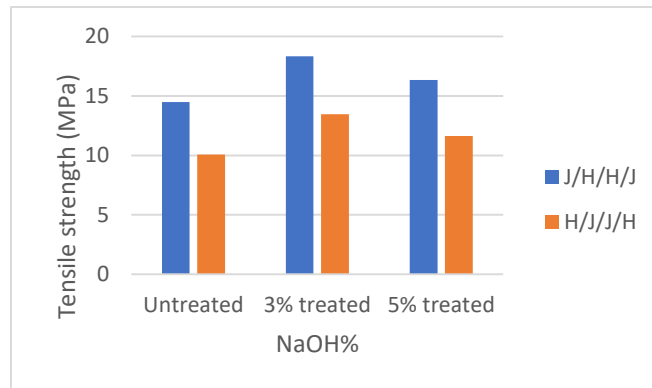


Figure 4.7. Tensile strength of different layered composites at 70% fiber content

4.3.2. Tensile Modulus

Tensile modulus is an important mechanical property which is used to evaluate the stiffness of a material. It is the ratio of stress (force per unit area) to the strain (relative deformation). Higher the tensile modulus, higher the stiffness of a material. Higher tensile modulus indicates that more force is required to deform the material. Like tensile strength, tensile modulus also changed according to the changing of fiber content, fiber surface treatment and fiber layering sequence. Table 4.4 showed the mean tensile modulus value of the composite.

Table 4.4. Mean tensile modulus of the composite plates.

Sample no	Sample code	Tensile modulus (MPa)
1	U (J/H/H/J)30	433.86
2	U (J/H/H/J)50	405.78
3	U (J/H/H/J)70	346.65
4	U (H/J/J/H)30	402.38
5	U (H/J/J/H)50	328.27
6	U (H/J/J/H)70	241.75
7	3%T (J/H/H/J)30	733.56
8	3%T (J/H/H/J)50	723.95
9	3%T (J/H/H/J)70	715.62
10	3%T(H/J/J/H)30	549.82
11	3%T(H/J/J/H)50	444.89
12	3%T(H/J/J/H)70	329.03
13	5%T (J/H/H/J)30	560.68
14	5%T (J/H/H/J)50	450.34
15	5%T (J/H/H/J)70	368.82
16	5%T(H/J/J/H)30	523.04
17	5%T(H/J/J/H)50	423.64
18	5%T(H/J/J/H)70	310.46

4.3.2.1. Effect of Fiber Content on The Composites' Tensile Modulus

Composite containing 30% fiber showed highest tensile modulus among other composites contained 50% and 70% fiber. In the Figure 4.8 and 4.9, J/H/H/J and H/J/J/H layered composites contained 30% fiber reached highest tensile modulus among others respectively. In the untreated J/H/H/J and H/J/J/H layered composites, 30% fibers exhibited 6% and 25%, 22% and 66% more tensile modulus than 50% and 70% fibers contained composites respectively. In case of 3% treated and 5% treated composite, in the J/H/H/J layered and H/J/J/H layered, 30% fibers showed 1%, 2% and 23%, 67% and 24%, 52% and 23%, 68% more strength than same counterpart respectively.

In the Figure 4.8, for 3% treated composite, 30% fiber content achieved 1.3% more strength than 50% fiber loading and 2.5% more strength than 70% fiber loading.

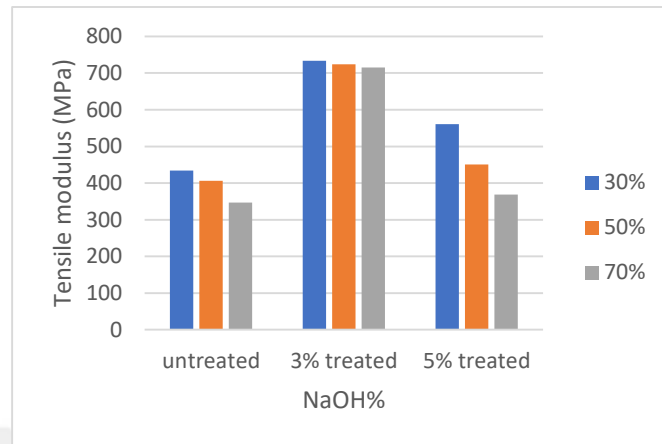


Figure 4.8. Tensile modulus of different fiber content of (J/H/H/J) layered composites.

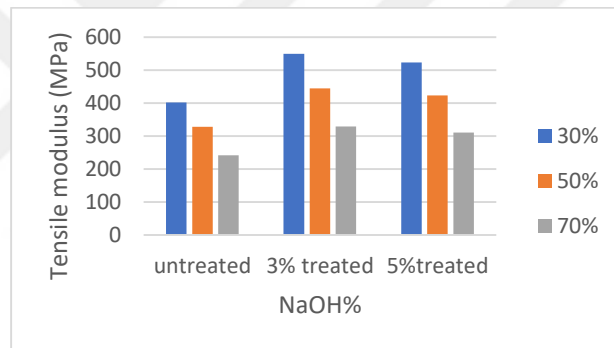


Figure 4.9. Tensile modulus of different fiber content of (H/J/J/H) layered composites.

4.3.2.2. Effect of NaOH% on The Composites' Tensile Modulus

In the Figure, 4.10 and 4.11, in all fiber content, 3% treated composite achieved highest tensile modulus. In 4.10, at 30, 50 and 70% fiber content, 3% treated composites exhibited 69% and 30%, 78% and 60%, 206% and 194% more tensile modulus than untreated and 5% treated composites respectively. In 4.11, at 30, 50 and 70% fiber content, 3% treated composites exhibited 36% and 5%, 35% and 5%, 36% and 5% more tensile modulus than untreated and 5% treated composites respectively.

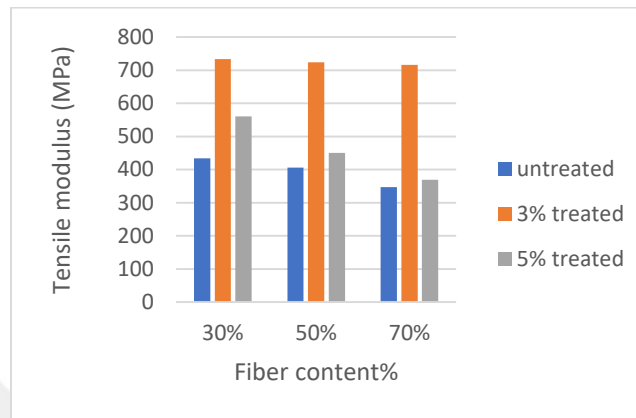


Figure 4.10. Tensile modulus of (J/H/H/J) layered composites at different NaOH%

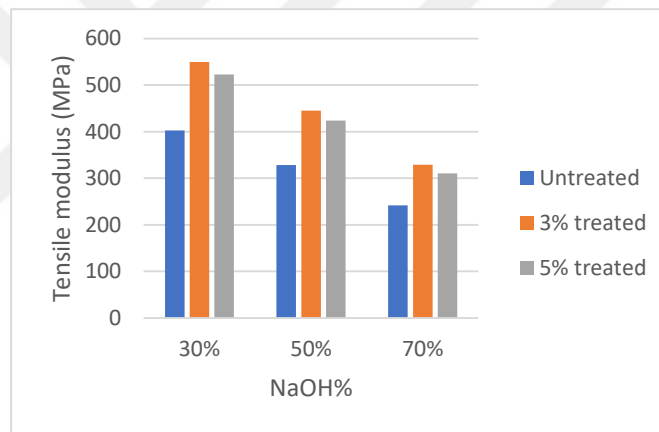


Figure 4.11. Tensile modulus of (H/J/J/H) layered composites at different NaOH%

4.3.2.3. Effect of Layering Sequence on The Composites' Tensile Modulus

In all fiber content, J/H/H/J layered composites possess higher tensile modulus than H/J/J/H composites. At 30% fiber content, in the untreated, 3% treated and 5% treated composites, J/H/H/J stacking sequences obtained 7%, 33% and 7% more tensile modulus than H/J/J/H stacking sequence respectively. In the Figure 4.13 and 4.14, in case of untreated, 3% treated, 5% treated composites, J/H/H/J layered

composites possess 23%, 62%, 6% and 43%, 217%, 18% more modulus than H/J/J/H composites.

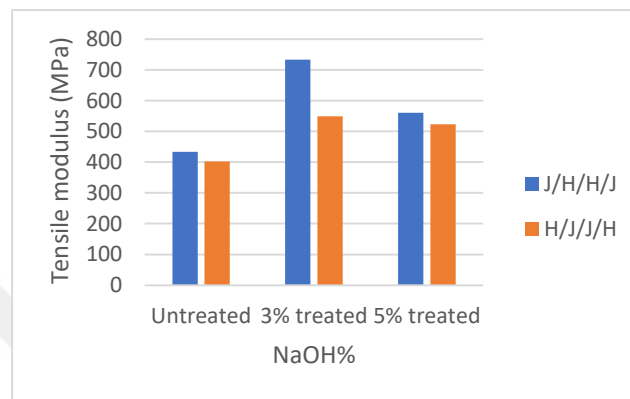


Figure 4.12. Tensile modulus of different layered composite at 30% fiber content.

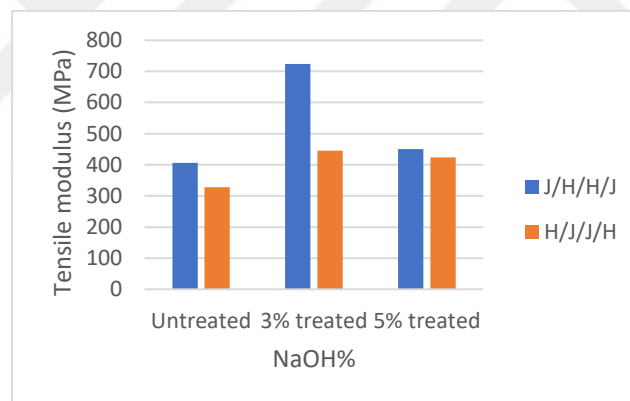


Figure 4.13. Tensile modulus of different layered composite at 50% fiber content.

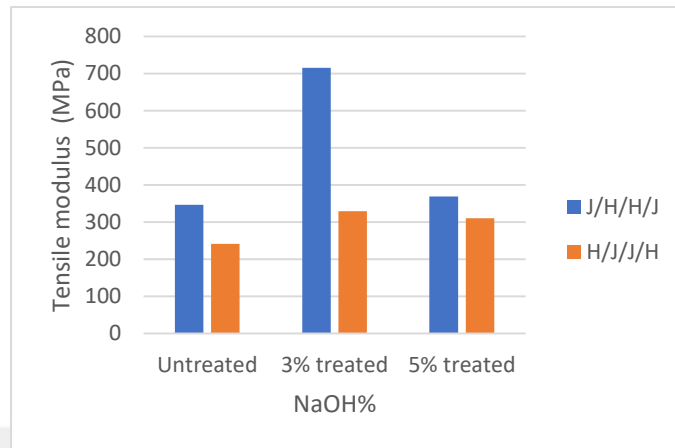


Figure 4.14. Tensile modulus of different layered composite at 70% fiber content.

4.4. Flexural Properties

Flexural property is used to describe the force required to bend a material under three point bending conditions. Flexural strength and flexural modulus are the important flexural properties. It indicates a composite material's load bearing capability without flexing. The application area of the composite material is governed by this property.

4.4.1. Flexural strength

Flexural strength of composite material is governed by amount of fiber content, interfacial adhesion between fiber and matrix and fiber layering sequences. Interfacial adhesion also depends on fiber surface treatment and concentration% of chemical agent used in that treatment. Table 4.5 shows values of flexural strength of the composites.

Table 4.5. Flexural strength of the composite plates.

Sample no	Sample code	Flexural strength MPa
1	U (J/H/H/J)30	50.89
2	U (J/H/H/J)50	55.61
3	U (J/H/H/J)70	59.23
4	U (H/J/J/H)30	48.87
5	U (H/J/J/H)50	52.70
6	U (H/J/J/H)70	55.41
7	3%T (J/H/H/J)30	55.17
8	3%T (J/H/H/J)50	58.61
9	3%T (J/H/H/J)70	62.55
10	3%T(H/J/J/H)30	50.00
11	3%T(H/J/J/H)50	54.76
12	3%T(H/J/J/H)70	58.41
13	5%T (J/H/H/J)30	53.10
14	5%T (J/H/H/J)50	56.95
15	5%T (J/H/H/J)70	61.23
16	5%T(H/J/J/H)30	49.00
17	5%T(H/J/J/H)50	53.70
18	5%T(H/J/J/H)70	56.51

4.4.1.1. Effect of Fiber Content on The Composites' Flexural Strength

Composite containing 70% fiber showed highest flexural strength among other composites contained 50% and 30% fiber. In the Figure 4.15 and 4.16, J/H/H/J and H/J/J/H layered composites contained 70% fiber reached highest flexural

strength among others respectively. In the untreated J/H/H/J and H/J/J/H layered composites, 70% fibers showed 6% and 16%, 5% and 13% more flexural strength than 50% and 30% fibers contained composites respectively. In case of 3% treated and 5% treated composite, in the J/H/H/J layered and H/J/J/H layered, 70% fibers showed 6%, 13% and 6%, 16% and 7%, 15% and 5%, 15% more strength than same counterparts respectively.

Joseph et al. developed composites using abaca fiber with varying fiber loading and found that, flexural modulus increased by 45% up to 25% fiber loading. The flexural strength also revealed good enhancement, for increasing fiber loading (S. Joseph et al., 2002).

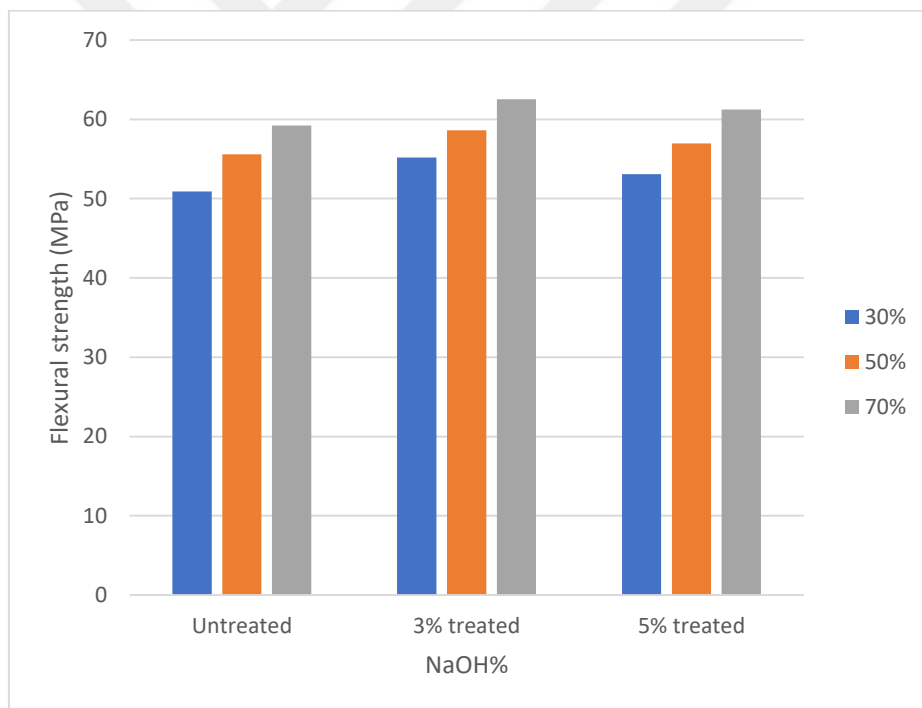


Figure 4.15. Flexural strength of different fiber content of J/H/H/J layered composites

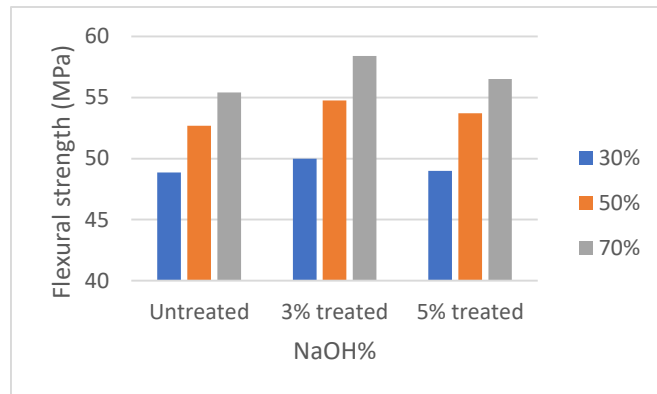


Figure 4.16. Flexural strength of different fiber content of H/J/J/H layered composites.

4.4.1.2. Effect of NaOH% on The Composites' Flexural Strength

In the Figure, 4.17 and 4.18, in all fiber content, 3% treated composite achieved highest flexural strength. In Figure 4.17, at 30, 50 and 70% fiber content, 3% treated composites exhibited 8% and 3%, 5% and 2%, 5% and 5% more flexural strength than untreated and 5% treated composites respectively. In the Figure 4.18, at 30, 50 and 70% fiber content, 3% treated composites exhibited 2% and 2%, 3% and 1%, 5% and 3% more flexural strength than untreated and 5% treated composites respectively.

Alfa fibers were treated with 1, 5, and 10 wt% NaOH solution and results revealed that, treated alfa fibers composites had increment in flexural strength (Rokbi et al., 2011).

Wenberg et al. developed unidirectional flax/epoxy composites with treated and untreated fibers. They treated fibers with (1, 2, 3% NaOH) and observed the effect of treatment on mechanical properties and found that, treated fibers composites had 30% greater flexural strength than untreated composites (Van de Weyenberg et al., 2003).

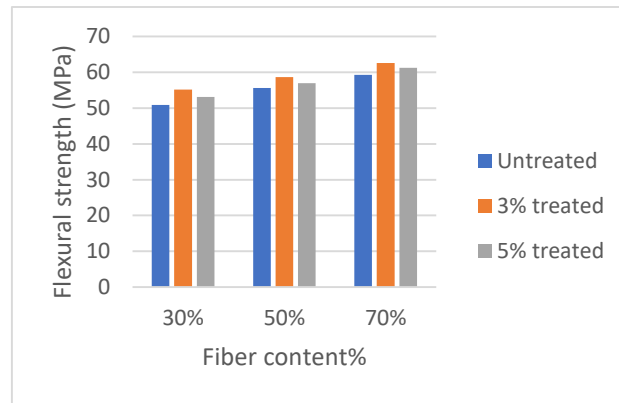


Figure 4.17. Flexural strength of J/H/H/J layered composites at different NaOH%

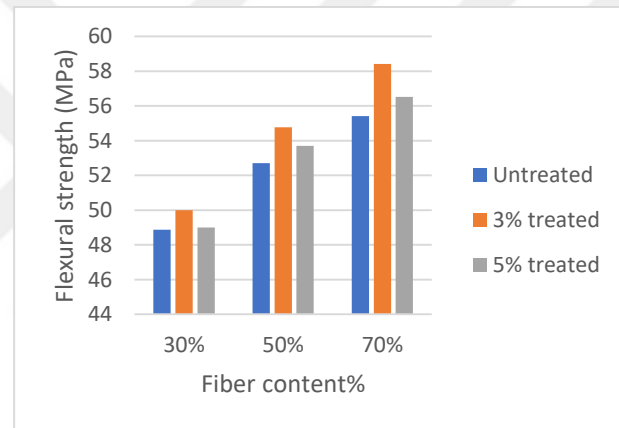


Figure 4.18. Flexural strength of H/J/J/H layered composites at different NaOH%

4.4.1.3. Effect of Stacking Sequence on The Composites' Flexural Strength

In all fiber content, J/H/H/J layered composites possess higher flexural strength than H/J/J/H composites. At 30% fiber content, in the untreated, 3% treated and 5% treated composites, J/H/H/J stacking sequences obtained 4%, 10% and 8% more flexural strength than H/J/J/H stacking sequence respectively. In the Figure 4.20 and 4.21, in case of untreated, 3% treated, 5% treated composites, J/H/H/J layered composites possess 5%, 7%, 6% and 6%, 7%, 8% more strength than H/J/J/H composites.

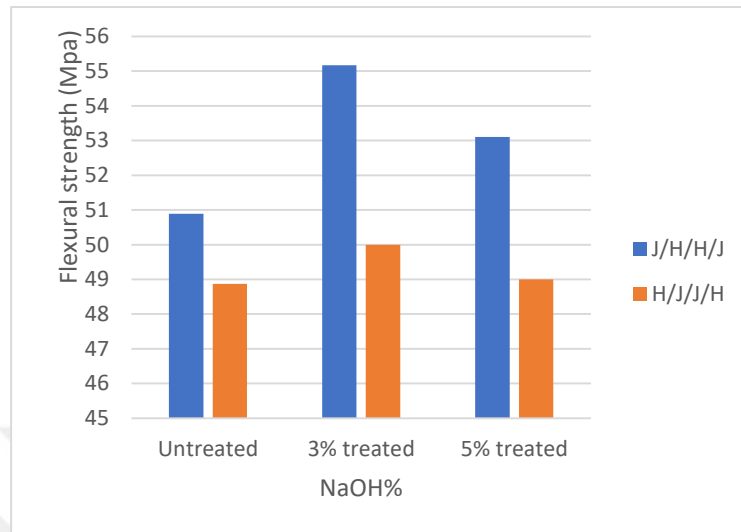


Figure 4.19. Flexural strength of different layered composites at 30% fiber content.

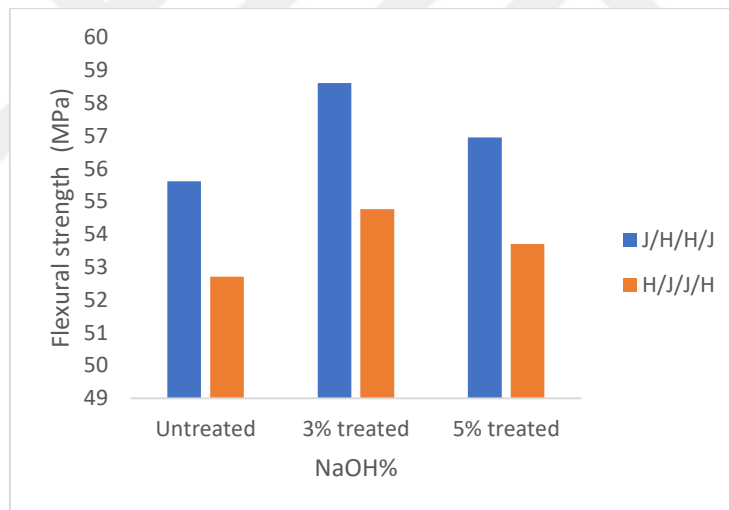


Figure 4.20. Flexural strength of different layered composites at 50% fiber content.

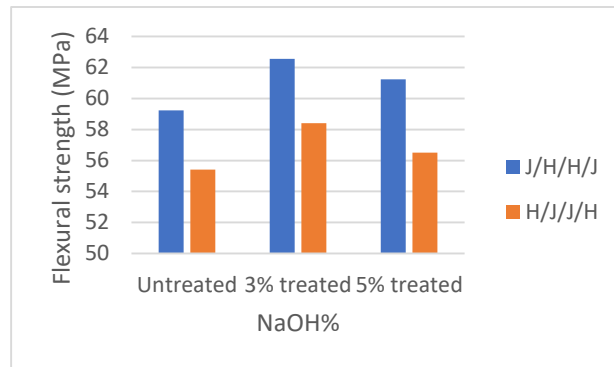


Figure 4.21. Flexural strength of different layered composites at 70% fiber content.

4.4.1.4. Flexural Modulus

Flexural modulus is a property that measures stiffness of a material when 3 point bending load is applied on it. It is a ratio of bending strain and bending stress. It is a amount of deformation of a material expected while bending load is applied. Higher the flexural modulus, higher the stiffness of material without flexing. Flexural modulus alters according to the alteration of fiber content, fiber surface treatment and fiber layering sequences.

Table 4.6. Flexural modulus of the composite plates.

Sample no	Sample code	Flexural modulus in GPa
1	U (J/H/H/J)30	1.99
2	U (J/H/H/J)50	2.04
3	U (J/H/H/J)70	2.9
4	U (H/J/J/H)30	1.8
5	U (H/J/J/H)50	1.9
6	U (H/J/J/H)70	2.07
7	3%T (J/H/H/J)30	2.75
8	3%T (J/H/H/J)50	3.00
9	3%T (J/H/H/J)70	3.04
10	3%T(H/J/J/H)30	2.6
11	3%T(H/J/J/H)50	2.7
12	3%T(H/J/J/H)70	2.9
13	5%T (J/H/H/J)30	2.6
14	5%T (J/H/H/J)50	2.8
15	5%T (J/H/H/J)70	2.95
16	5%T(H/J/J/H)30	2.4
17	5%T(H/J/J/H)50	2.6
18	5%T(H/J/J/H)70	2.65

4.4.1.5. Effect of Fiber Content on Flexural Modulus

Composite containing 70% fiber showed highest flexural modulus among other composites contained 50% and 30% fiber. In the Figure 4.22 and 4.23, J/H/H/J and H/J/J/H layered composites contained 70% fiber reached highest flexural modulus among others respectively. In the untreated J/H/H/J and H/J/J/H layered composites, 70% fibers showed 42% and 52%, 9% and 15% more flexural strength than 50% and 30% fibers contained composites respectively. In case of 3% treated and 5% treated composite, in the J/H/H/J layered and H/J/J/H layered, 70% fibers showed (1.3%, 10.5% and 5%, 13%) and (6%, 10% and 2%, 10%) more strength than same counterparts respectively.

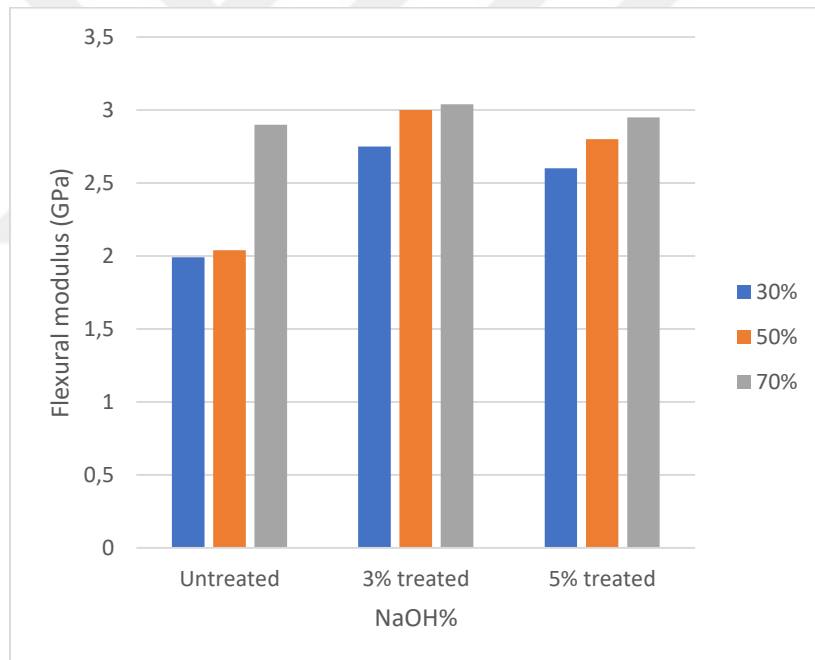


Figure 4.22. Flexural modulus of different fiber content of J/H/H/J layered composites

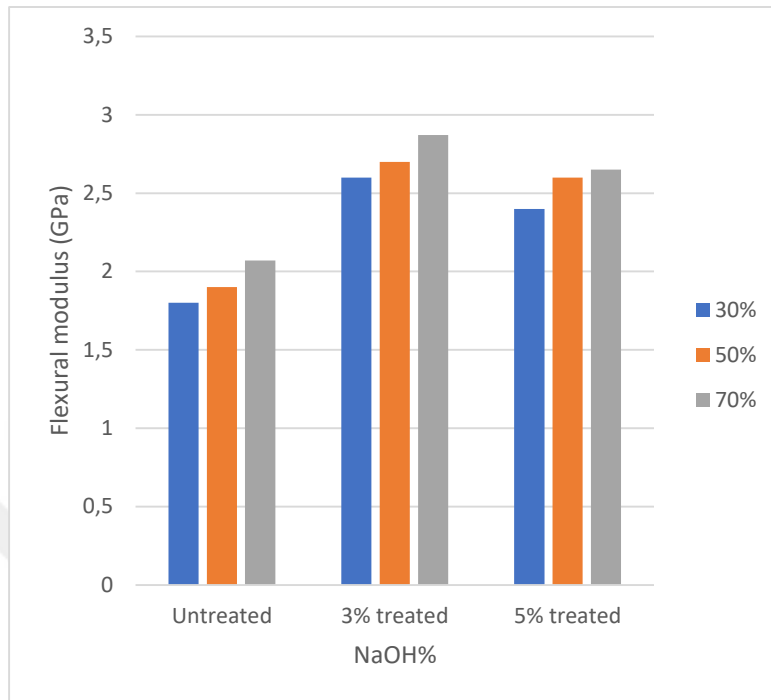


Figure 4.23. Flexural modulus of different fiber content of H/J/J/H layered composites

4.4.1.6. Effect of NaOH % on Flexural Modulus

In the Figure, 4.24 and 4.25, in all fiber content, 3% treated composite achieved highest flexural modulus. In Figure 4.24, at 30, 50 and 70% fiber content, 3% treated composites exhibited 38% and 5%, 47% and 7%, 4% and 3% more flexural modulus than untreated and 5% treated composites respectively. In the Figure 4.25, at 30, 50 and 70% fiber content, 3% treated composites exhibited 44% and 8%, 42% and 4%, 38% and 8% more flexural modulus than untreated and 5% treated composites respectively.

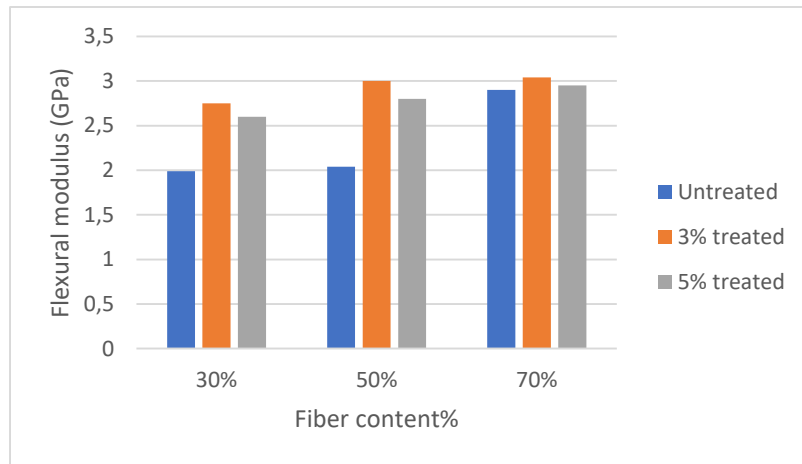


Figure 4.24. Flexural modulus of J/H/H/J layered composites at different NaOH%

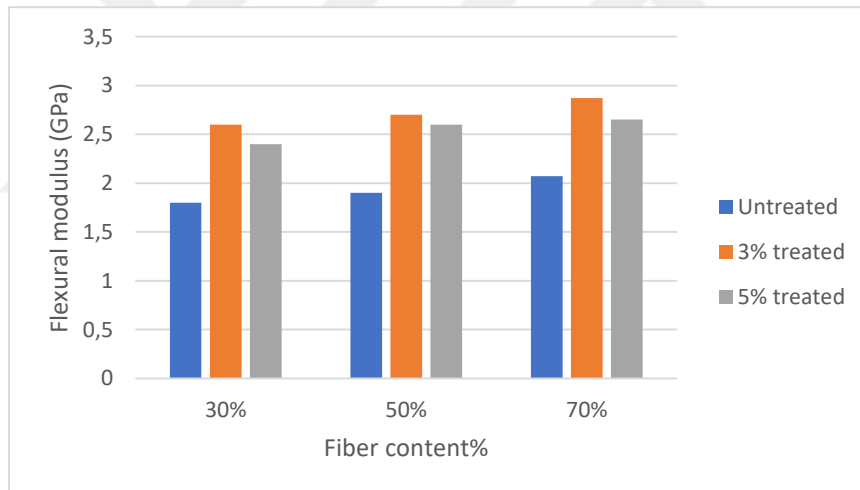


Figure 4.25. Flexural modulus of H/J/J/H layered composites at different NaOH%

4.4.1.7. Effect of Stacking Sequences on The Composites' Flexural Modulus

In all fiber content, J/H/H/J layered composites possess higher Flexural modulus than H/J/J/H composites. At 30% fiber content, in the untreated, 3% treated and 5% treated composites, J/H/H/J stacking sequences obtained 10%, 6% and 8% more flexural modulus than H/J/J/H stacking

sequence respectively. In the Figure 4.27 and 4.28, in case of untreated, 3% treated, 5% treated composites, J/H/H/J layered composites possess 7%, 11%, 7% and 40%, 4%, 11% more modulus than H/J/J/H composites.

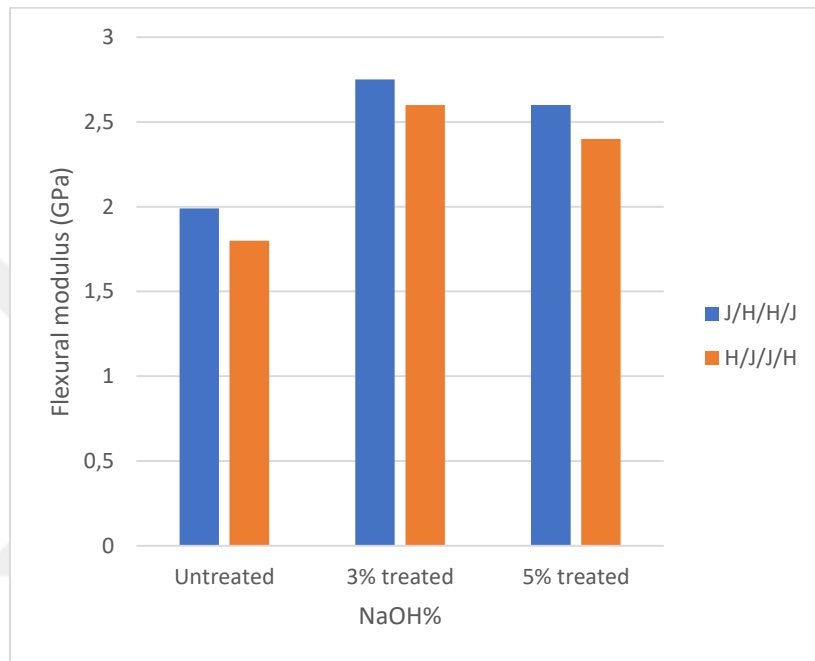


Figure 4.26. Flexural modulus of different layered composites at 30% fiber content.

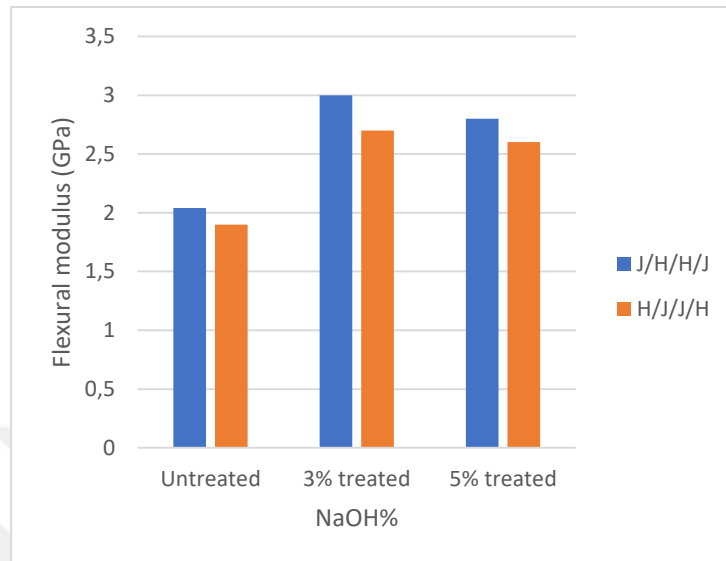


Figure 4.27. Flexural modulus of different layered composites at 50% fiber content.

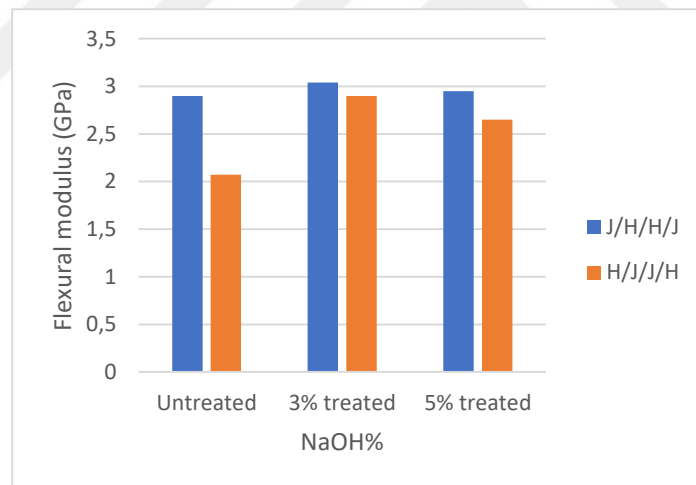


Figure 4.28. Flexural modulus of different layered composites at 70% fiber content.

4.5. Impact Properties

It is a resistance of a material when sudden load is applied on it. The load is applied in milliseconds or less. The near instantaneous load causes the material to absorb the energy. When applied load exceeds the limit that it can absorb, the material undergoes to fracture, tear or damage. Impact strength or impact toughness is a significant impact property.

4.5.1. Impact Strength

Impact strength of composite is significant when it goes to the area of application where material experiences against sudden load. So, it is important to measure impact strength whether it is applicable for expected end uses or not. The impact strength of the material is measured in two ways such as charpy impact test and izod impact test. This property of material can be changed according to the changes of fiber content, varying concentration% of alkali and altering fiber layering sequences. Table 4.7 shows the mean impact strength of the composite plates.

Table 4.7. The values of Impact strength of the composite plates

Sample no		Sample code	Impact strength
1		U (J/H/H/J)30	1.86
2		U (J/H/H/J)50	2.03
3		U (J/H/H/J)70	2.21
4		U (H/J/J/H)30	2.05
5		U (H/J/J/H)50	2.24
6		U (H/J/J/H)70	2.40
7		3%T (J/H/H/J)30	2.10
8		3%T (J/H/H/J)50	2.30
9		3%T (J/H/H/J)70	2.43
10		3%T(H/J/J/H)30	2.31
11		3%T(H/J/J/H)50	2.50
12		3%T(H/J/J/H)70	2.6
13		5%T (J/H/H/J)30	1.93

14		5%T (J/H/H/J)50	2.10
15		5%T (J/H/H/J)70	2.30
16		5%T(H/J/J/H)30	2.15
17		5%T(H/J/J/H)50	2.3
18		5%T(H/J/J/H)70	2.5

4.5.1.1. Effect of Fiber Content on The Composites' Impact Strength

Composite containing 70% fiber showed highest impact strength among other composites contained 30% and 50% fiber. In the above Figure 4.29 and 4.30, J/H/H/J and H/J/J/H layered composites contained 70% fiber reached highest impact strength among others respectively. In the untreated J/H/H/J and H/J/J/H layered composites, 70% fibers exhibited 18% and 8%, 17% and 7% more impact strength than 30% and 50% fibers contained composites respectively. In case of 3% treated and 5% treated composite, in the J/H/H/J layered and H/J/J/H layered, 70% fibers showed (15%, 5% and 12%, 4%) and (19%, 9% and 16%, 8%) more strength than same counterparts.

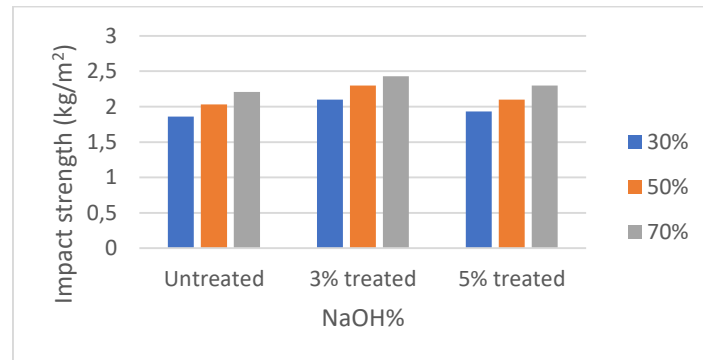


Figure 4.29. Impact strength of different fiber content of J/H/H/J layered composite

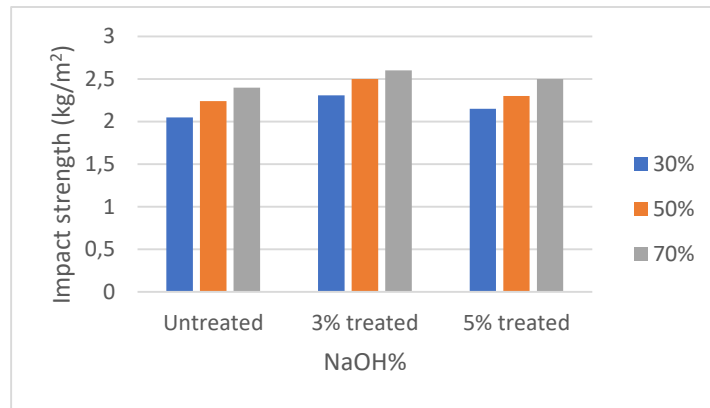


Figure 4.30. Impact strength of different fiber content of H/J/J/H composite.

4.5.1.2. Effect of NaOH% on Impact Strength

In the Figure, 4.31 and 4.32, in all fiber content, 3% treated composite achieved highest impact strength. In Figure 4.31, at 30, 50 and 70% fiber content, 3% treated composites exhibited 12% and 8%, 13% and 9%, 9% and 5% more impact strength than untreated and 5% treated composites respectively. In the Figure 4.32, at 30, 50 and 70% fiber content, 3% treated composites exhibited 12% and 7%, 11% and 8%, 8% and 4% more impact strength than untreated and 5% treated composites respectively.

Venkateshwaran et al. (2013) treated banana fibers with various concentration of NaOH solution and developed treated banana/epoxy composites. They observed that 1% NaOH treated fibers composite revealed the highest impact strength of banana/epoxy composite as compared to 0.5, 2, 5, 10, 15, and 20 wt% NaOH.

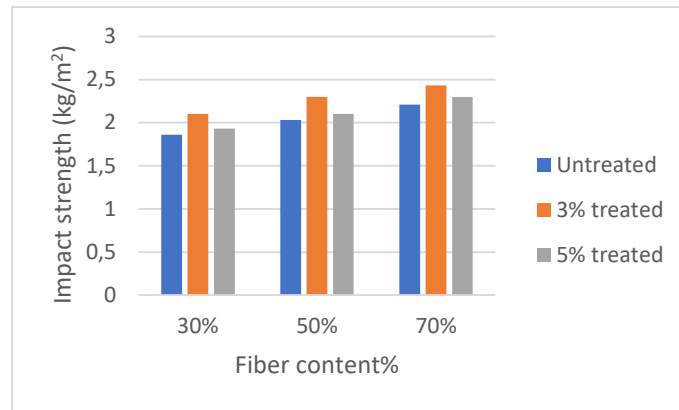


Figure 4.31. Impact strength of J/H/H/J layered composites at different NaOH%

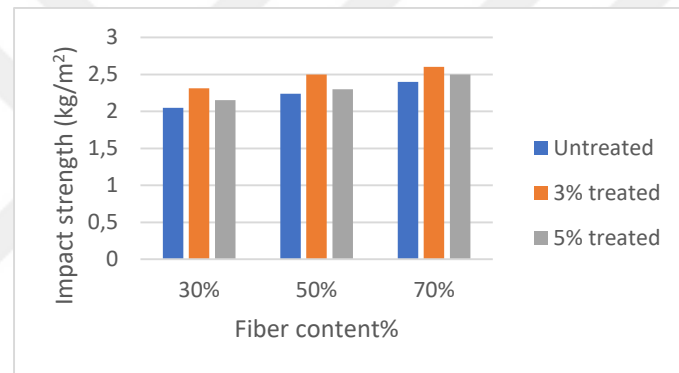


Figure 4.32. Impact strength of H/J/J/H layered composites at different NaOH%

4.5.1.3. Effect of Stacking Sequence on Impact Strength

In all fiber content, H/J/J/H layered composites possess higher impact strength than J/H/H/J layered composites. At 30% fiber content, in the untreated, 3% treated and 5% treated composites, H/J/J/H stacking sequences obtained 10%, 10% and 11% more impact strength than J/H/H/J stacking sequence respectively. In the Figure 4.34 and 4.35, in case of untreated, 3% treated, 5% treated composites, H/J/J/H layered composites possess 10%, 8%, 9% and 14%, 6%, 8% more strength than J/H/H/J composites.

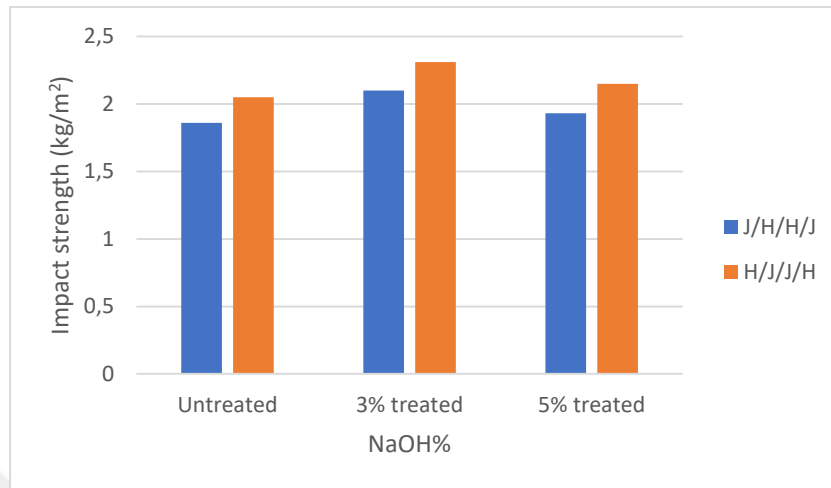


Figure 4.33. Impact strength of different layered composites at 30% fiber content.

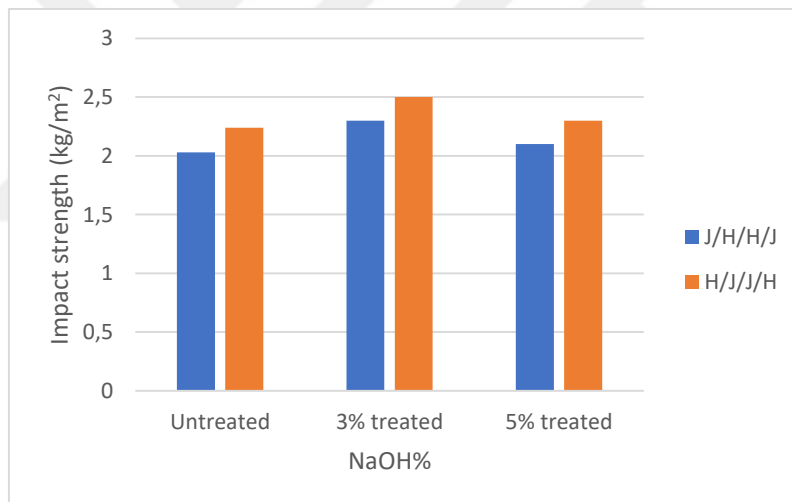


Figure 4.34. Impact strength of different layered composites at 50% fiber content.

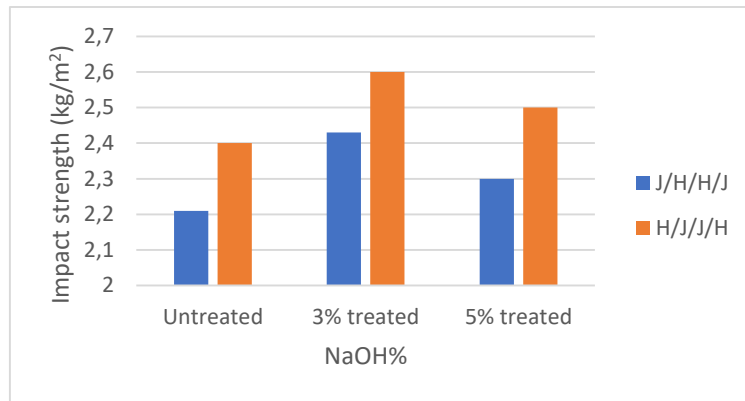


Figure 4.35. Impact strength of different layered composites at 70% fiber content

4.6. Analysis of ANOVA Test

From the Table 4.8, it was observed, ($p=0.004$), indicating that, fiber content significantly influenced tensile strength but had no significant effect on tensile modulus due to higher value of p , ($p > 0.05$). Similarly, fiber content also affected flexural strength and impact strength significantly but in case of flexural modulus, there was no effect due to higher value ($p > 0.05$).

Table 4.8. ANOVA test result for the effect of fiber content

		Sum of Sq	df	Mean Sq	F	Sig.
Tensile strength	Between Groups	211.327	2	105.664	8.373	0.004
	Within Groups	189.285	15	12.619		
	Total	400.613	17			
Tensile modulus	Between Groups	2327106.494	2	1163553.247	1.431	0.270
	Within Groups	12194833.239	15	812988.883		
	Total	14521939.733	17			
Flexural strength	Between Groups	179.229	2	89.615	14.691	0.000
	Within Groups	91.503	15	6.100		
	Total	270.732	17			
Flexural modulus	Between Groups	358573.506	2	179286.753	1.157	0.341

	Within Groups	2324599.875	15	154973.325		
	Total	2683173.381	17			
Impact strength	Between Groups	0.347	2	0.174	7.151	0.007
	Within Groups	0.364	15	0.024		
	Total	0.711	17			

From the Table 4.9, there were no significant difference among the effect of NaOH treatment with 0%, 3%, 5% concentration on tensile strength, flexural strength and impact strength due to p Value is greater than 0.05. On the other hand, having lower p value than 0.05, tensile modulus and flexural modulus were influenced by altering the NaOH treatment with various concentration.

Figure 4.36. ANOVA test result for the effect of NaOH treatment.

		Sum of Sq.	df	Mean Sq.	F	Sig.
Tensile strength	Between Groups	61.984	2	30.992	1.371	0.284
	Within Groups	339.078	15	22.605		
Tensile Modulus	Between Groups	125555.793	2	62777.896	4.926	0.023
	Within Groups	191170.632	15	12744.709		
Flexural strength	Between Groups	23.534	2	11.767	0.714	0.506
	Within Groups	247.198	15	16.480		
Flexural modulus	Between Groups	1627867.124	2	813933.562	11.570	0.001
	Within Groups	1055262.615	15	70350.841		
Impact strength	Between Groups	0.181	2	0.091	2.567	0.110
	Within Groups	0.530	15	0.035		

4.7. Analysis of t-test

It was a observation from the Table 4.11 for ρ value against the tensile strength, tensile modulus, flexural strength, flexural modulus and impact strength were 0.217, 0.517, 0.426, 0.488, 0.528 respectively, meant that the value was greater than 0.05. It was concluded that, there was no significant difference between the effect of two layering sequence for all mechanical properties.

Table 4.9. Group statistics for fiber layering.

Properties	Fiber layering	N	Mean	Std. Deviation	Std. Error Mean
Tensile strength	J/H/H/J	9	19.2644	5.50969	1.83656
	H/J/J/H	9	16.3611	3.87743	1.29248
Tensile modulus	J/H/H/J	9	483.9989	152.42952	50.80984
	H/J/J/H	9	437.3944	146.03672	48.67891
Flexural strength	J/H/H/J	9	55.9256	4.08606	1.36202
	H/J/J/H	9	54.3744	3.97395	1.32465
Flexural modulus	J/H/H/J	9	2592.6644	434.74905	144.91635
	H/J/J/H	9	2457.7789	368.98550	122.99517
Impact strength	J/H/H/J	9	2.2078	0.21288	0.07096
	H/J/J/H	9	2.2711	0.20325	0.06775

Table 4.10. t-test result for the effect of fiber layering.

		Levene's test for equality of variances		t-test for equality of means		
		F	Sig.	t	df	ρ value
Tensile strength	Equal variances assumed	1.324	0.267	1.293	16	0.214
	Equal variances not assumed			1.293	14.363	0.216
Tensile modulus	Equal variances assumed	0.120	0.734	0.662	16	0.517
	Equal variances			0.662	15.971	0.517

	not assumed					
Flexural strength	Equal variances assumed	0.000	0.996	0.816	16	0.426
	Equal variances not assumed			0.816	15.988	0.426
Flexural modulus	Equal variances assumed	0.110	0.745	0.710	16	0.488
	Equal variances not assumed			0.710	15.588	0.488
Impact strength	Equal variances assumed	0.008	0.930	0.646	16	0.528
	Equal variances not assumed			0.646	15.966	0.528

4.8. Analysis of SEM (Scanning electronic microscope)

Scanning electron microscopy contributes an excellent technique for identification of fiber surface morphology and fractured surface of fiber composites.

The SEM assessments were applied for the untreated and NaOH treated jute and hemp fibers characterizations. Untreated jute and hemp fibers shown in Figure 4.36 (a) and Figure 4.37 (a) respectively. One can see a usual structure due to the presence of chemical compounds like lignin, cellulose, hemicelluloses, and wax. After NaOH treatment of jute and hemp fibers using 3, 5% NaOH, the abovementioned components were removed partially. The presence of a few supplementary fibers may be due to the modification on the outside. As shown, the untreated jute and hemp fiber had noticeable pores on the surface than the NaOH treated fibers. It can be seen from the Figure that the untreated jute and hemp fibers

are attached with each other with the presence of lignin, oil, wax, and surface impurities. As a result of alkali treatment, it is observed that the adhesion between the fibers seems to get demolished.

By using alkali treatment, the fiber surface appeared irregular texture and relatively cleaned as compared to untreated fiber, as shown in Figure 4.36 and 4.37 respectively. It is clearly observed in the Figure that diameter also have been decreased of treated fibers as compared to untreated ones due to removal of impurities and shrinkage of lumen. Cai has reported that the alkali treatment reduced the diameter of the fibers due to removal of surface contaminants and shrinking lumens (Cai et al., 2015). Alkali treatment alters fiber surface morphology and creates a linking mechanism with the surface of composites, modifying the natural surface and improving mechanical properties (Noorunnisa Khanam et al., 2011; Orue et al., 2015).

4.8.1. SEM Images of The Fibers

SEM analysis was applied to visually examine the effect of NaOH alkali treatment on the fiber morphology of jute and hemp fibers. Figure 4.36 and Figure 4.37 show scanning electronic microscopic view of (a) untreated (b) 3% treated (c) 5% treated fiber for jute and hemp.

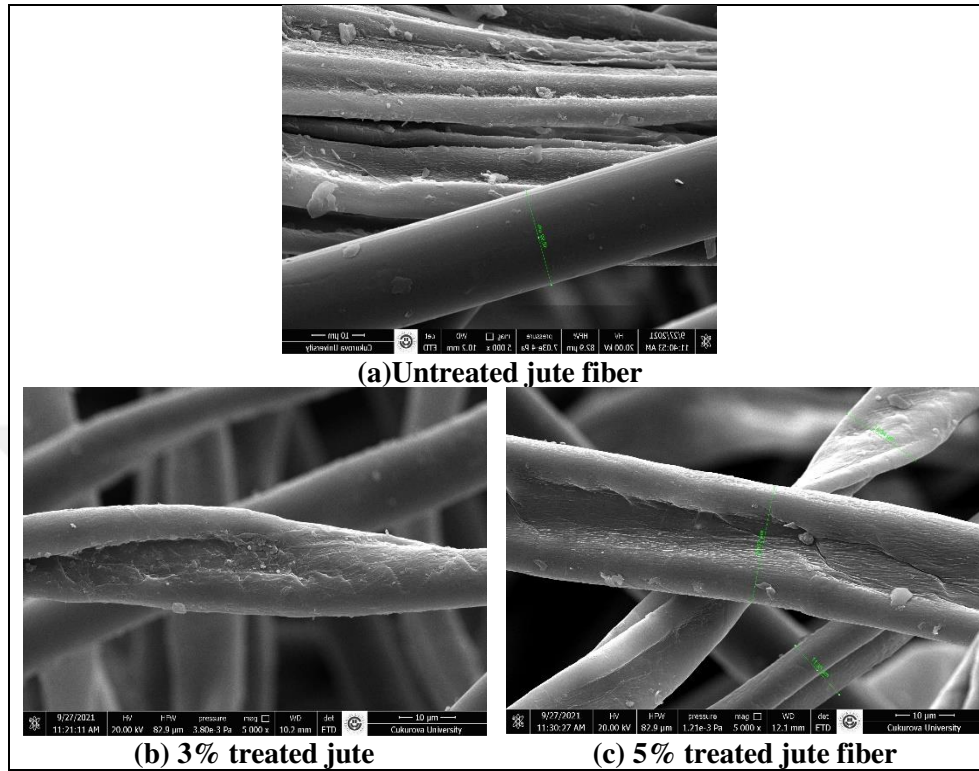


Figure 4.37. Scanning electronic microscopic view of (a) untreated (b) 3% treated (c) 5% treated jute fiber

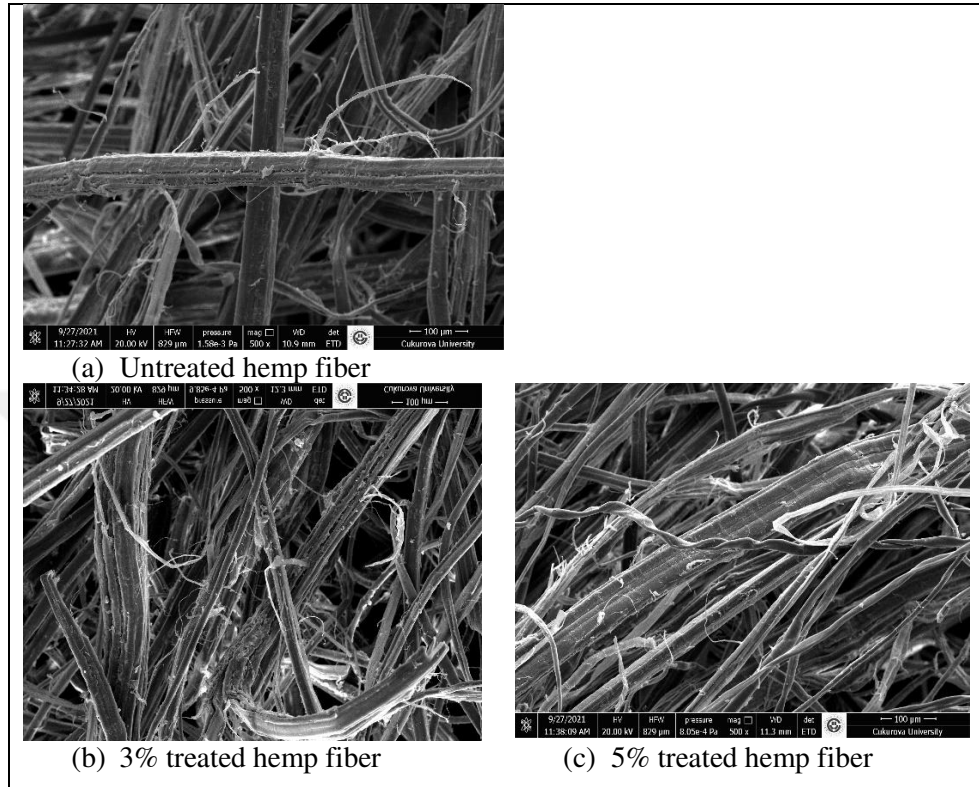


Figure 4.38. Scanning electron microscopic view of (a) untreated (b) 3% treated (c) 5% treated hemp fiber

4.8.2. SEM Images of The Composites

The morphological analysis such as interfacial bonding, dilation of the matrix, fiber pulling or fracture between the fiber and matrix in the composites are examined and analyzed using SEM analysis. Some SEM analyses results were given Appendix-1. From the Figure 4.39, 4.40 and 4.41 SEM micrographs of untreated fiber reinforced composites shows that there is a low bonding between the fiber and matrix laying path to low interfacial strength and bigger cracks in the polyester matrix that happened due to fiber pull out at the interphase and porous is developed because of less effectiveness in interfacial wetting. In case of alkali treated hybrid composite, the stress transfer between fiber and polyester resin is effective. Due to

lesser fiber pull out, and homogeneous mixture of fiber in polyester resin and fiber breakage develops very frequently in the compressive region which leads to excellent adhesion at the interphase region.

From the Figure 4.39 it is cleared that, lesser fiber pull out was happened in the 3% treated composites than untreated and 5% treated composites due to higher adhesion between fiber and matrix, indicating the highest tensile strength.

SEM micrographs of broken tensile test specimens exhibited finer fracture and fiber pull-out, which were essential elements of the fracture mechanism during the produced composite's tensile testing (Öztürk, 2010). After tensile testing, used SEM analysis was used to detect the failure process of a napier/polyester composite. Fiber extrusion occurred as a result of fiber pull-out, which resulted in matrix cracking, fiber fracture, and fiber debonding, as shown by SEM micrographs (Haameem et al., 2016).

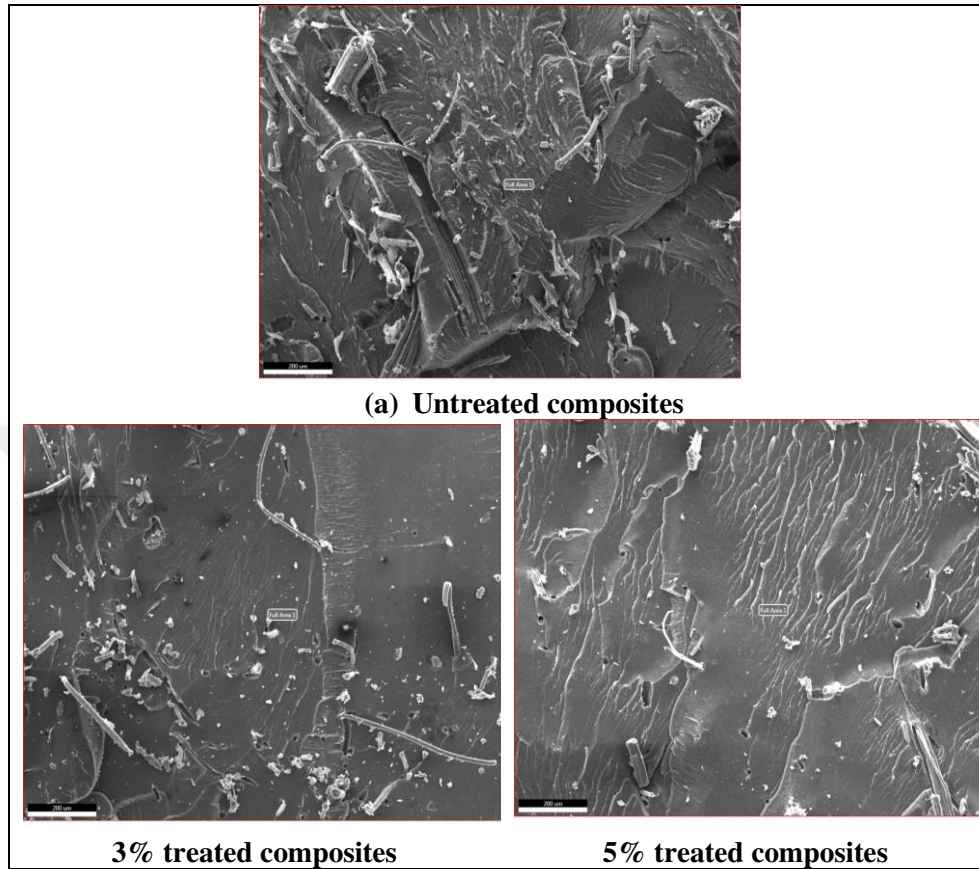


Figure 4.39. SEM image of (a) untreated (b) 3% treated (c) 5% treated composites before mechanical testing.

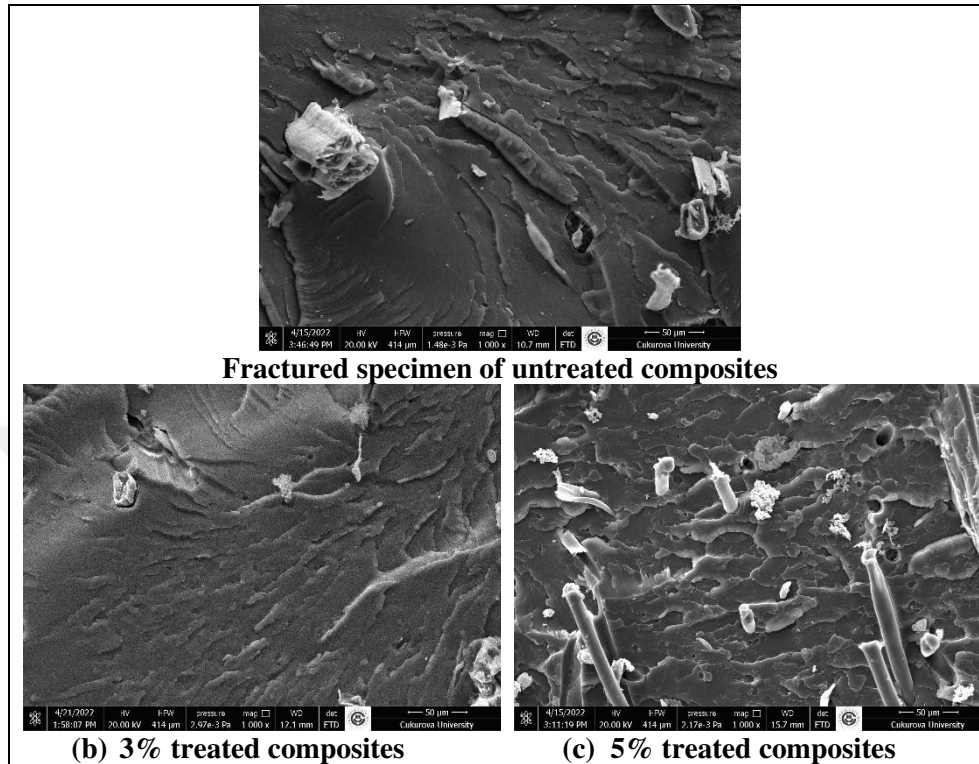


Figure 4.40. SEM image of (a) untreated (b) 3% treated (c) 5% treated composites after tensile testing.

From the Figure 4.40 and 4.41, lesser fiber pull out, lesser dilation of the matrix, less fiber fracture, comparatively smooth and clean surface was observed in the 3% treated composites than other counterpart, governing the achievement of highest flexural and impact strength respectively. Untreated composites had higher fiber matrix debonding than 3% treated and 5% treated composite, hence indicating less flexural and impact strength achievement.

Flexural strength significantly depends upon Fiber/matrix surface adhesive characteristics, appropriate resin penetration inside fiber bundles, which increases interfacial bonding between fiber/matrix interfaces and lowers fiber pull-out, and fiber debonding (Yousif et al., 2012). Vijaya Ramnath studied about the fractured specimen of flexural testing. He observed that, existence of voids, inter-phase

delamination and matrix breakage are the regulating characteristics of the fractured specimen during flexural testing (Vijaya Ramnath et al., 2013).

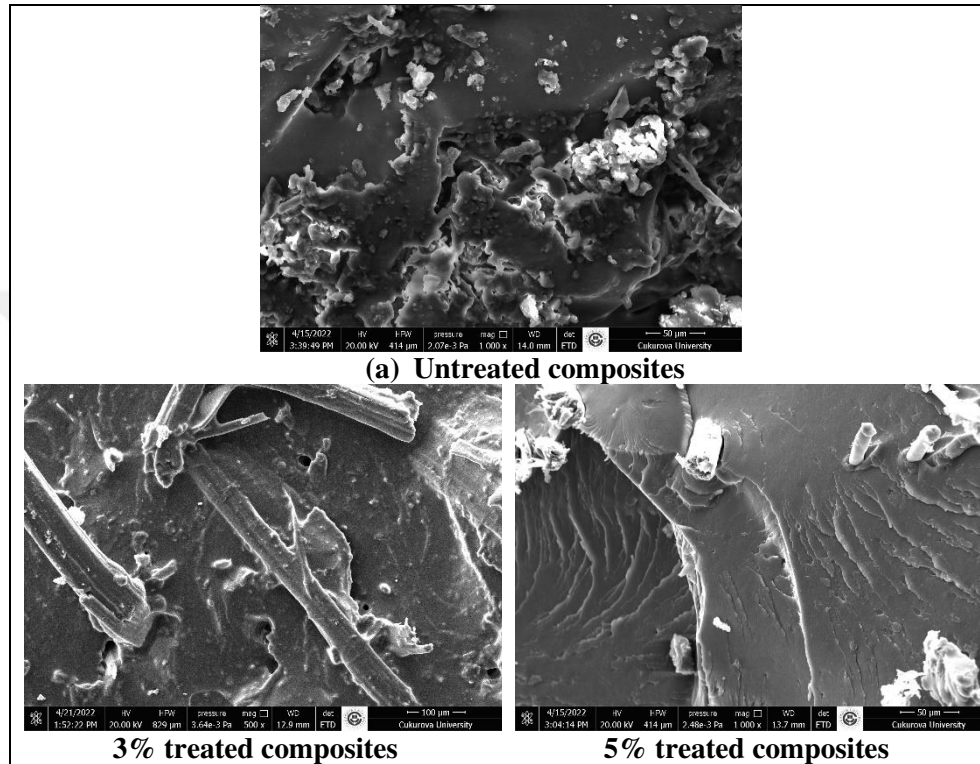


Figure 4.41. SEM image of (a) untreated (b) 3% treated (c) 5% treated composites after flexural testing.

Debonding between fiber-matrix interfaces, severe polyester matrix rupture and cracking, fiber fracture, and total pull-out are some of the reasons for the failure of the developed composite laminates under impact testing, as shown in Figure 4.41. The brittle nature of laminate failure is readily demonstrated by SEM images, which exhibit a sharp and clean matrix fracture. Mader et al., (2016) observed the impact properties of cellulose epoxy composite due to the effect of adhesion between fiber and matrix. The author suggested that fiber pull-out causes a weaker interfacial shear strength that reduced the impact strength of the developed composites. Similar study was conducted by (Mader et al., 2016) on glass/epoxy composite and he found that,

matrix cracking, fiber debonding and fiber pull-out are the factors to reduce impact strength.

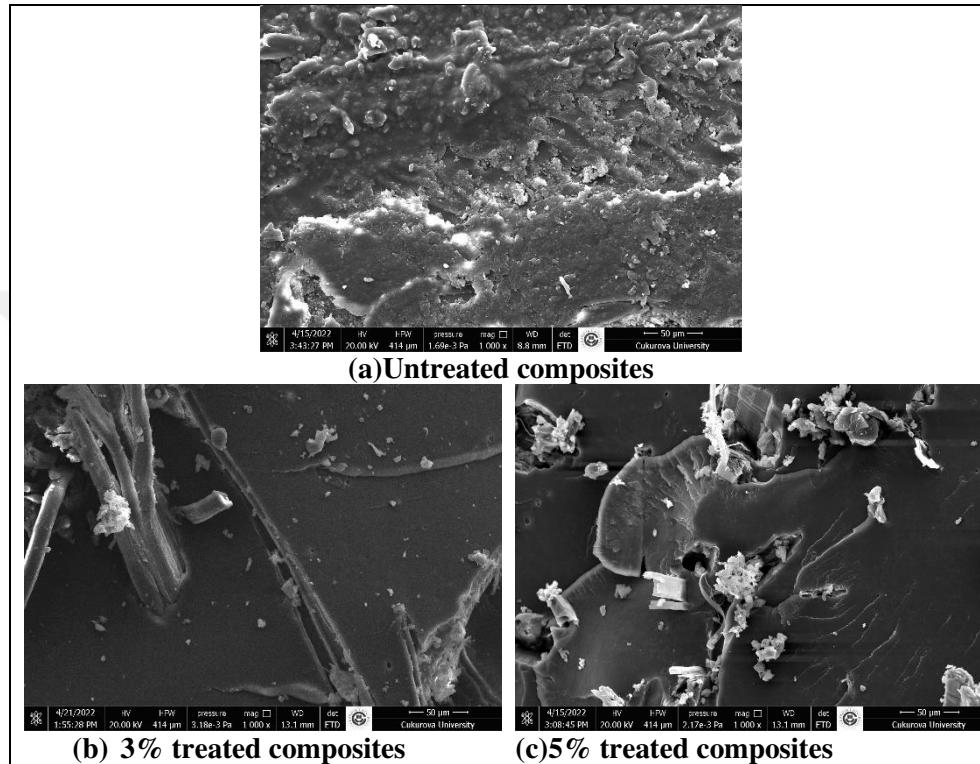


Figure 4.42. SEM image of (a) untreated (b) 3% treated (c) 5% treated composites after impact testing.

4.9. Analysis of EDS Image of The Composites

EDX is a useful technique for chemical analysis. This method relies on a considerable interaction between the sample and the X-ray source. Essentially, each element has its own atomic structure, resulting in a distinct set of peaks on each X-ray emission spectrum. The surface topography with elemental analysis of the untreated, 3 percent treated, and 5 percent treated hybrid composites was observed and investigated using an EDX analysis and SEM. Some EDX results were given in Appendix-2 section. The results are shown in Figure 4.42, 4.43, and 4.44,

respectively, revealed that jute and hemp fiber are primarily composed of carbon and oxygen. The C and O elements, which are the major components of lignocellulose fiber structure, appear to be the most dominating in the EDX spectra. Basically, no significant change was observed in the chemical elements in treated and untreated fiber hybrid composites.

Similar result was reported by Rashid et.al. They analyzed EDS spectra of sugar palm untreated and treated fiber with sea water and alkali and observed carbon, oxygen and silicone molecule in both fibers (Rashid et al., 2016)

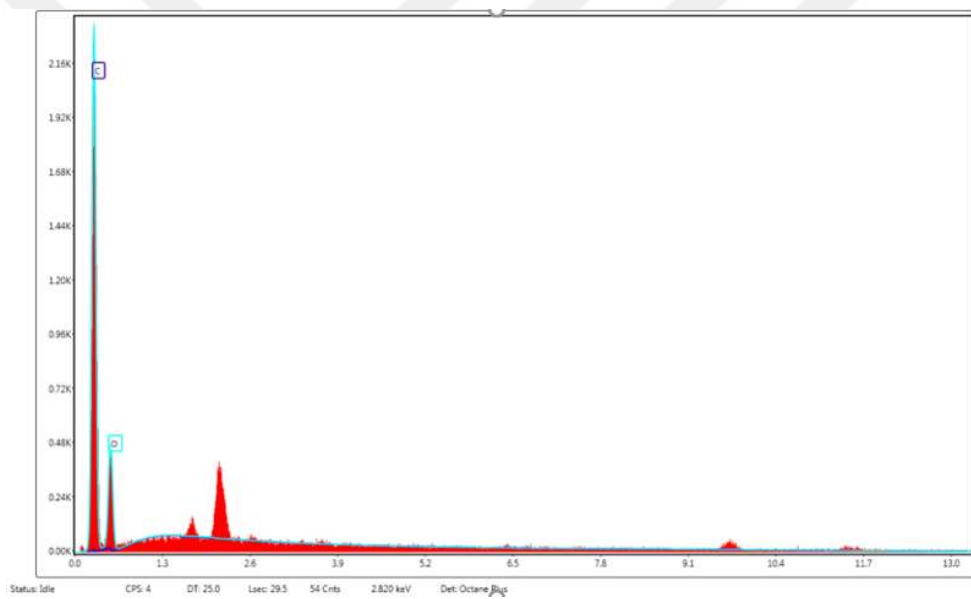


Figure 4.43. EDS image of untreated composites.

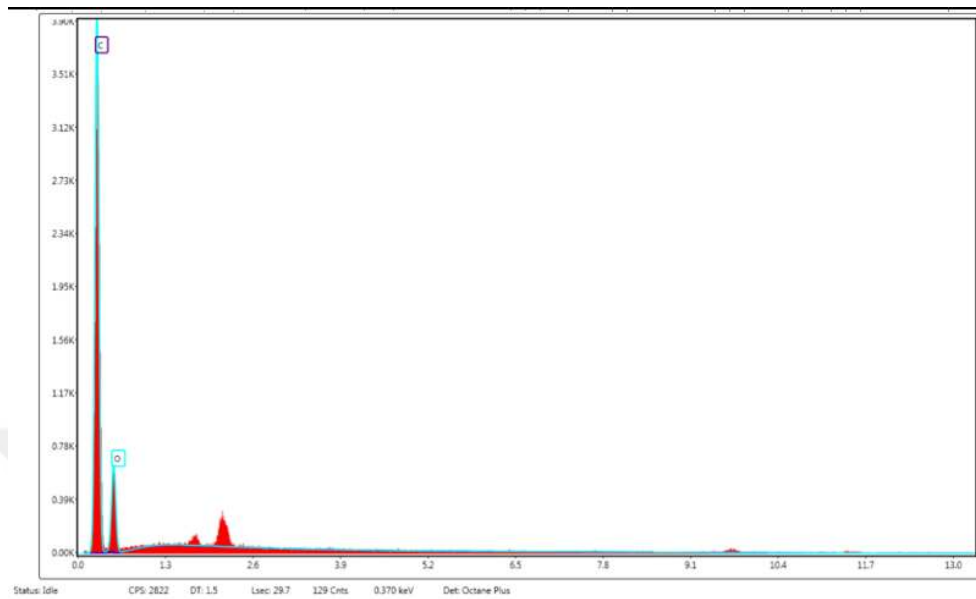


Figure 4.44. EDS image of 3% treated composites

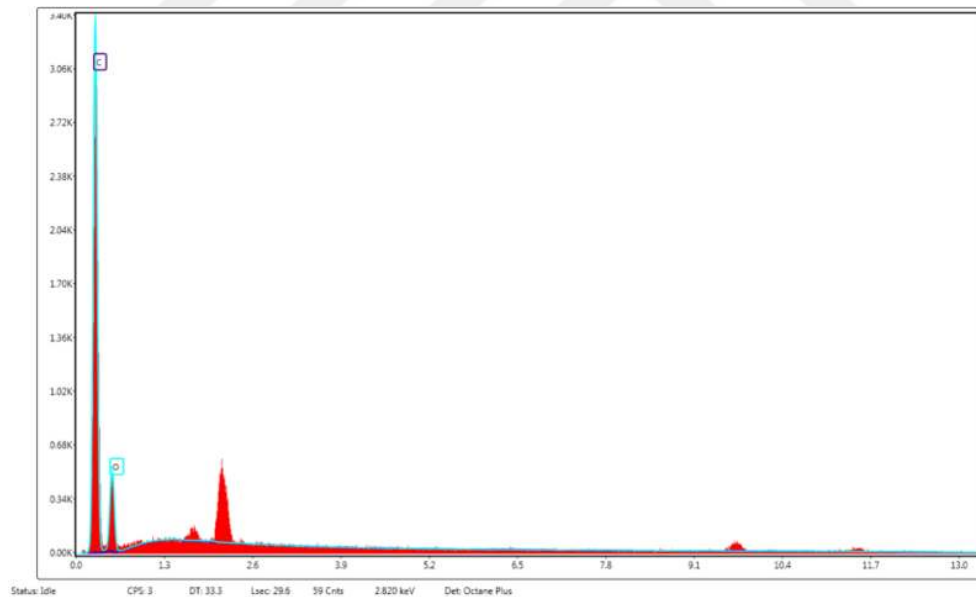


Figure 0.1. EDS image of 5% treated composites.

4.10. Analysis of XRD Image of The Composites

From the Figure 4.45, 4.46 and 4.47 it was observed that, crystallinity index of treated composites was higher than untreated composites. The elimination of the amorphous contents (hemicellulose and lignin) of the fibers by the alkali treatment could have determined the rise in the crystallinity index of the fibers, also contributing to improved mechanical properties. Some XRD results were given in Appendix-3 section.

Several researchers explained the similar result. The XRD spectra of untreated, 2 percent alkali treated, and 5 percent alkali treated *Acacia planiform* fibers were studied by Senthamaraikannan et al. (2019) The treatment reduced the amorphous fraction and increased the crystallinity index. Untreated, 2 percent alkali-treated, and 5 percent alkali-treated fibers had crystallinity indexes of 65.38 percent, 69.94 percent, and 74.76 percent, respectively.

Sinha et.al (2009) characterized jute fiber and its composites through X-ray diffraction studies. They treated jute fibers with 5% alkali at room temperature and investigated that 5% treated fibers exhibited higher crystallinity index% than untreated fibers.

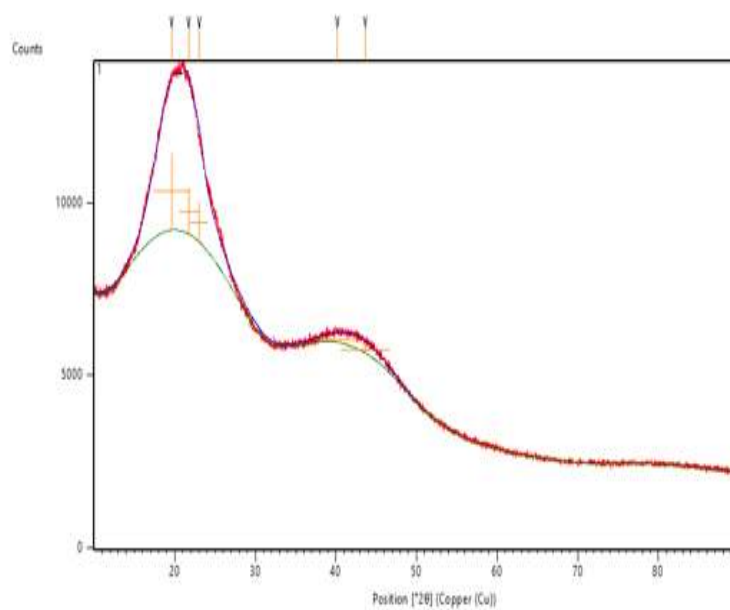


Figure 4.45.XRD of untreated composites

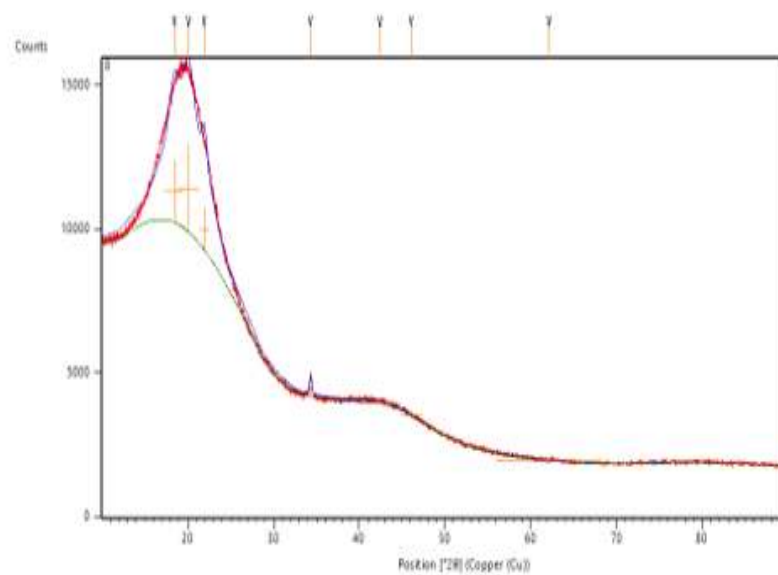


Figure 4.46. XRD of 3% treated composites

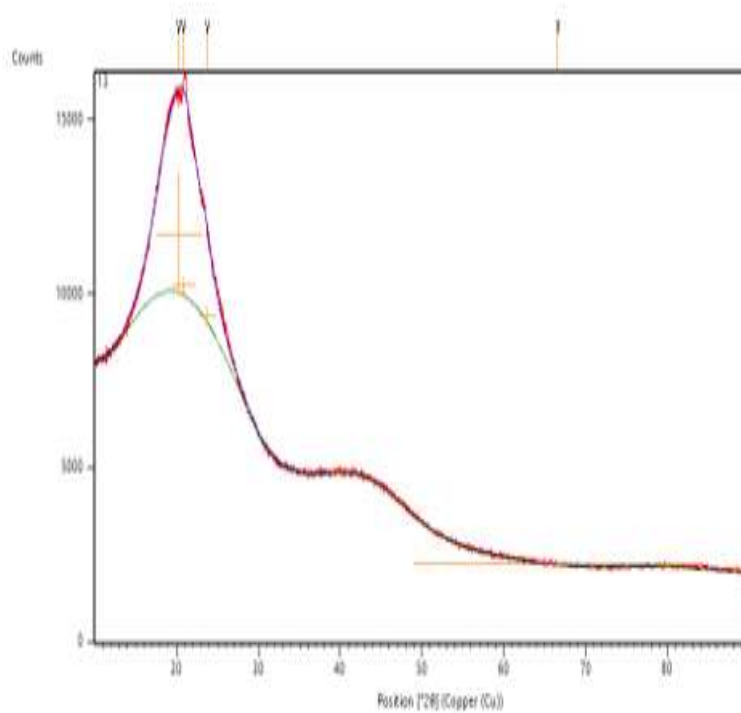


Figure 4.47. XRD of 5% treated composites.

4.11. Cost analysis of The Study

A small study on the cost of the composites produced in this study was also made and given here. September 2022 exchange rate has been taken into account for TL conversion.

It was calculated for the composite plate's size, 18x18x4 cm and average weight of the composite plate, 350 g and given below.

For example considering the composite containing 70% fiber by volume.

- 20 g weight of fibers covered 70% fiber by volume.
- Density of polyester is about 1g/cm^3

4. RESULTS AND DISCUSSION

Md Zahidul ISLAM

350 g weight contained (350x0.2) =70 g fibers in which there were 35g
jute, 35 g hemp and (350x.3) =105 g polyester resin.

1 kg composite plate contained 300 g polyester resin

1 kg composite plate contained (1000x0.2) =200 g fiber in which 100 g hemp and
100 g jute fiber.

Price of 1 ton hemp fiber	=260 \$
Price of 1 kg hemp fiber	=0.26 \$=4.8 TL
Price of 1 kg jute fiber	=0.8 \$=14.83 TL
Price of 1 kg polyester	=70 TL
Price of 300 g polyester	=21 TL
Price of 100 g hemp fiber	=0.48 TL
Price of 100 g jute fiber	=1.5 TL

So total cost per kg of composite = (21+0.48+1.5) = 22.98 TL/kg

The composite plate's cost is 23 TL/kg (for September 2022)

5. CONCLUSION AND RECOMMENDATION

Natural fiber reinforced composites are attracting attention from the researchers, academicians due to their environmentally friendly, sustainability and renewability. Higher specific strength and stiffness, lower density, ease in processing have made it attractive alternative to the synthetic fiber counterpart. The mechanical properties of natural fiber reinforced composites could be enhanced by several factors such as by hybridizing, using optimum level of fiber content, increasing fiber matrix adhesion through chemical treatment as well as altering sequence of fiber layering.

Fiber content is an important factor influences the mechanical properties of hybrid composite. By increasing fiber content gradually up to certain limit, mechanical properties reach to its optimum level. After exceeding that limit, mechanical properties exert to decrease.

Fiber matrix adhesion force governs the mechanical properties. Mechanical properties increase with the increase of fiber matrix interfacial force. Due to incompatibility of hydrophilic natural fibers with hydrophobic matrix, natural fiber reinforced polymer composite should be subjected to surface treatment prior to its fabrication. Alkali treatment increases surface roughness which increases fiber matrix interfacial force, in the long run, higher mechanical properties.

- **Tensile properties:** 30% fiber loading exhibited higher tensile strength and tensile modulus than 50% and 70% fiber loading. 50% fiber loaded composite showed higher strength and modulus than 70% fiber loaded composite. 3% treated composite showed highest tensile strength and tensile modulus among untreated and 5% treated composite. 5% treated composites surpassed untreated composites in terms of tensile strength and modulus. J/H/H/J layered composite showed higher strength and modulus, than H/J/J/H layered composite.

- **Flexural properties:** 70% fiber loaded composites revealed highest flexural strength and flexural modulus among others. 50% fiber loaded composite exhibited higher flexural strength, and flexural modulus than 30% fiber loaded composite. 3% treated composites revealed highest flexural strength and flexural modulus, among others. 5% treated composites showed higher flexural strength and flexural modulus than untreated composites. J/H/H/J layered composite showed higher flexural strength and flexural modulus than H/J/J/H layered composite.
- **Impact properties:** 70% fiber loaded composites exhibited highest impact strength among others. 50% fiber loaded composite exhibited higher strength than 30% fiber loaded composite. 3% treated composites showed highest strength among others. 5% treated composites exhibited higher impact strength than untreated composites. H/J/J/H layered composites surpassed J/H/H/J composite in terms of impact strength.

The longitudinal views of treated, untreated jute and hemp fibers by using SEM analysis showed that, treated fibers were rough, clean and less porous than untreated fiber. Alkali treatment removed hemicellulose, lignin, wax and oil which made the fiber surface rough and clean. Surface roughness of treated fibers increased interfacial force between fiber and matrix, which obviously an agreement of superior mechanical properties of treated fibers than untreated ones.

The morphological properties of fractured specimen by using SEM analysis exhibited that, fiber pull out, fiber fracture, matrix cracking, matrix breakage, fiber breakage, debonding are the reason for failure mechanism during different mechanical testing. Treated composites always showed higher mechanical properties than untreated composites, which was supported by morphological study. Fiber fracture, fiber pull out, debonding were less in treated composite than untreated

composites. 5% treated composite always showed higher fiber fracture, fiber pull-out, matrix cracking, debonding, matrix breakage than 3% treated composite.

To solve the challenges with NFRPCs such as moisture absorption, insufficient hardness, and decreased long-term stability for outdoor applications, more research is needed. Therefore, research must concentrate on creating materials with a balance between structure and composite properties that are more affordable and easily could be manufactured.

However, natural fibers are inconsistent in their properties resulting a obstacle to develop high quality hybrid composite. The inconsistency of natural fibers depends on growth condition such as improper climate, unavailability of quality pesticide etc. No doubt, research should be carried out on the improvement of quality of natural fibers which can revolutionize their utilization in the development of composite product. On the other hand, incorporation of micro or nano-size particles can overcome some of their limitations.



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BIOGRAPHY

MD Zahidul Islam started his undergraduate education in Textile Engineering under the College of Textile Technology (1995 – 1999). He was working as a production officer in Beximco knitting LTD from 2000 to 2003. He was working as a lecturer in the College of Textile Technology from 2003 to 2013. He was working as a Assistant Professor in the Bangladesh University of Textiles from 2013 to till now. He started his Master's program in Yarn Engineering Department under the Bangladesh University of Textiles (2012-2015). He started his Doctor of Philosophy (Ph.D) program in Textile Engineering Department, Institute of Science and Technology at Çukurova University (2018-).





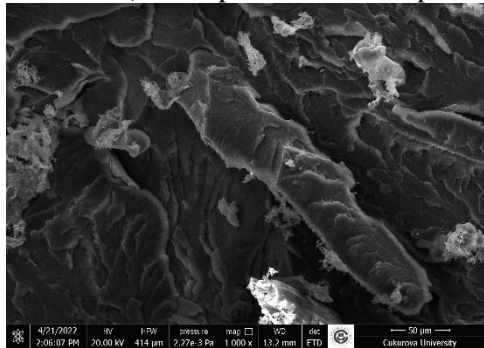
APPENDICES



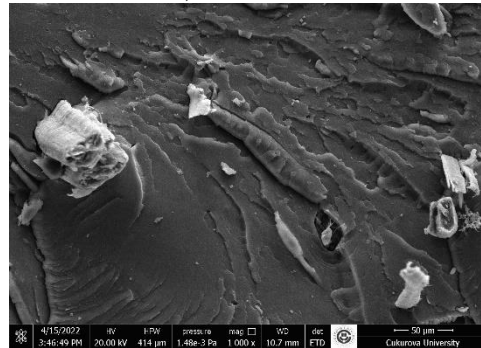
Appendix-1: SEM Image of Fractured Specimen of Mechanical Test.

1.1 SEM image of fractured specimen of tensile strength test.

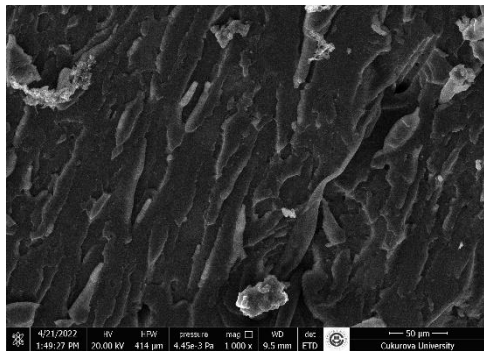
1.1.1. SEM image of fractured specimen of tensile strength test for S-1, S-2, S-3, S-4, S-5, S-6 (S: Sample number in experimental date tables)



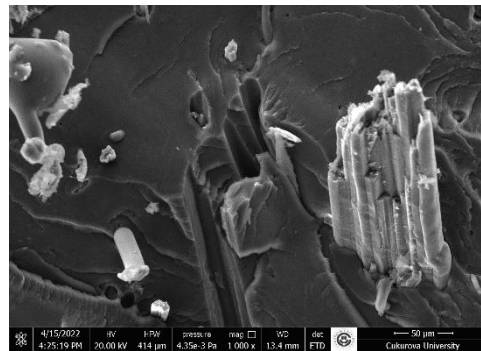
S-1



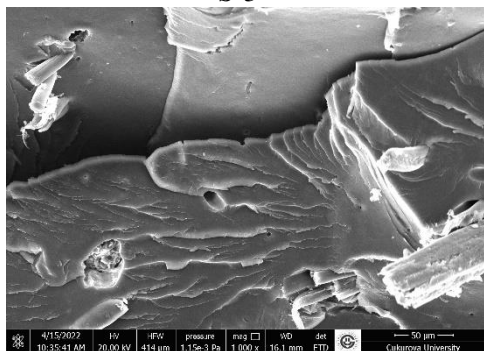
S-2



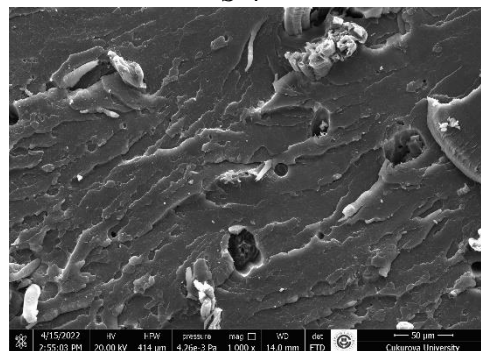
S-3



S-4



S-5



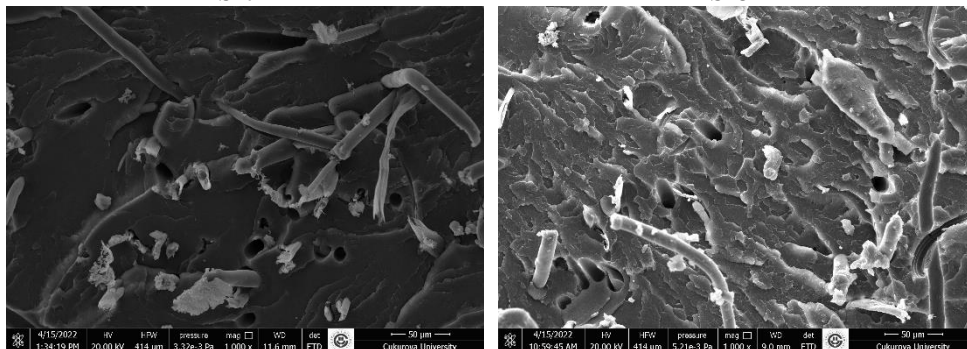
S-6

1.1.2. SEM image of fractured specimen of tensile strength test for S-7, S-8, S-9, S-10, S-11, S-12 (S: Sample number in experimental date tables)



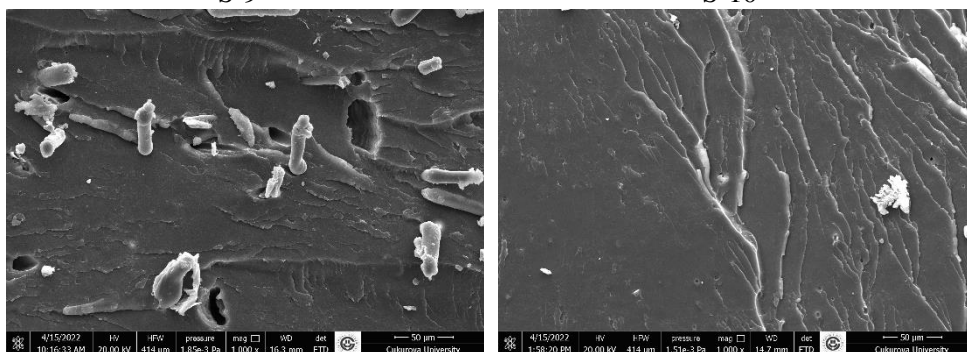
S-7

S-8



S-9

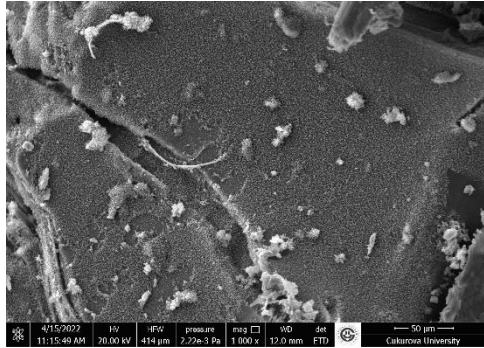
S-10



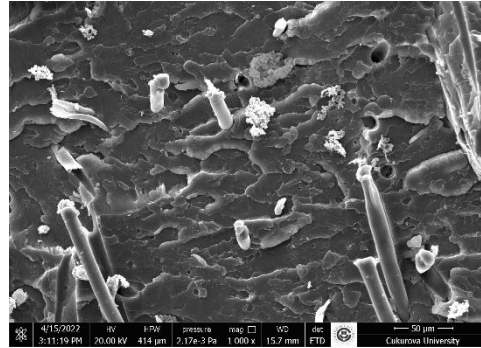
S-11

S-12

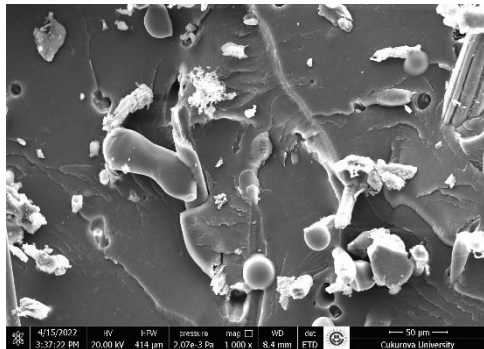
1.1.3. SEM image of fractured specimen of tensile strength test for S-13, S-14, S-15, S-16, S-17, S-18 (S: Sample number in experimental date tables)



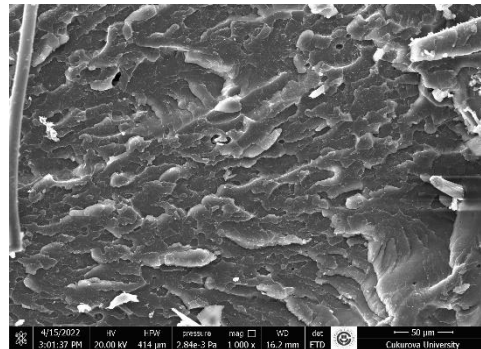
S-13



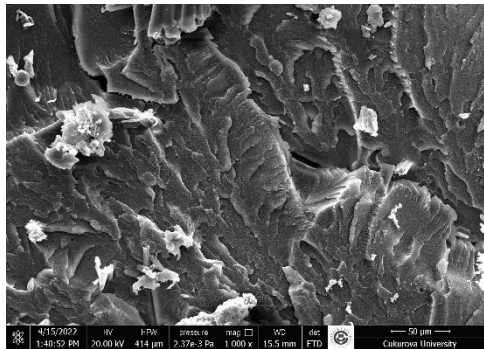
S-14



S-15



S-16



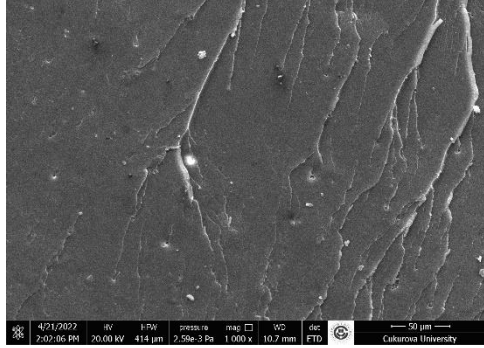
S-17



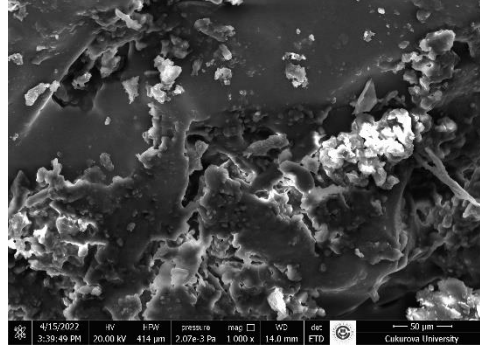
S-18

1.2.SEM image of fractured specimen of flexural strength test.

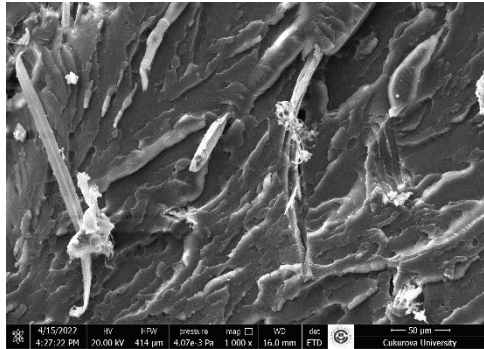
1.2.1. SEM image of fractured specimen of flexural strength test for S-1, S-2, S-3, S-4, S-5, S-6 (S: Sample number in experimental date tables)



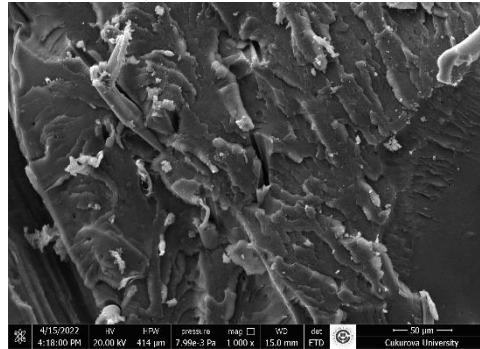
S-1



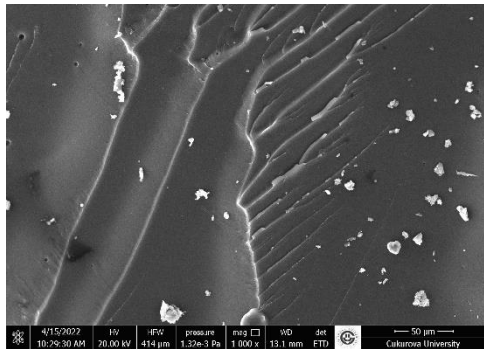
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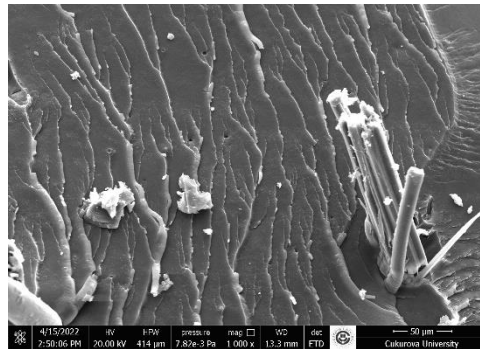
S-3



S-4

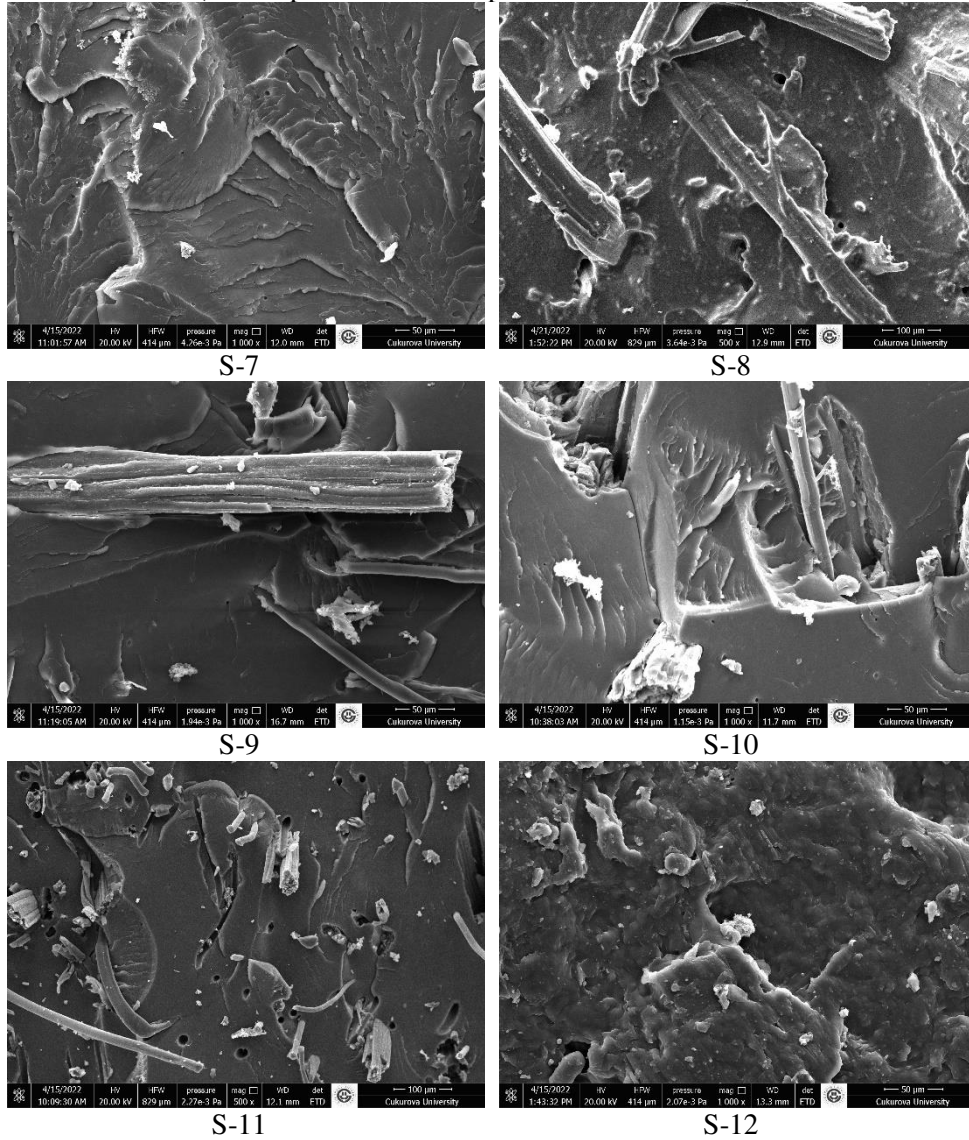


S-5

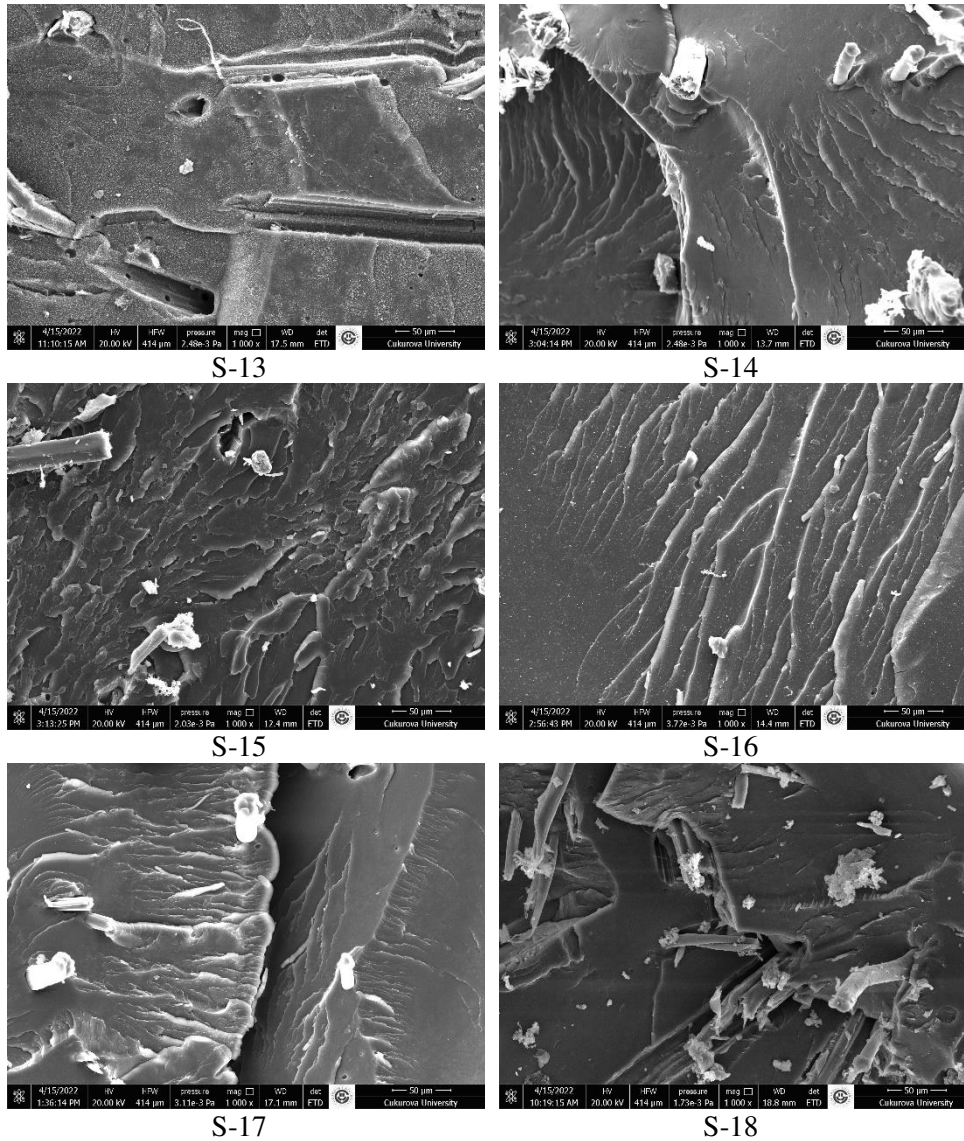


S-6

1.2.2. SEM image of fractured specimen of flexural strength test for S-7, S-8, S-9, S-10, S-11, S-12 (S: Sample number in experimental date tables)

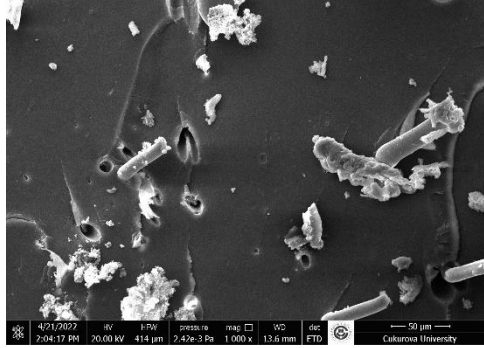


1.2.3. SEM image of fractured specimen of flexural strength test for S-13, S-14, S-15, S-16, S-17, S-18 (S: Sample number in experimental date tables)

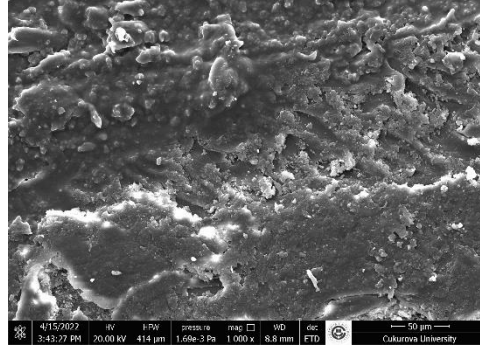


1.3.SEM image of fractured specimen of impact strength test.

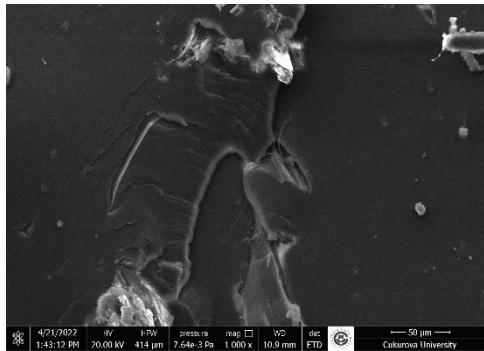
1.3.1. SEM image of fractured specimen of impact strength test for S-1, S-2, S-3, S-4, S-5, S-6 (S: Sample number in experimental date tables)



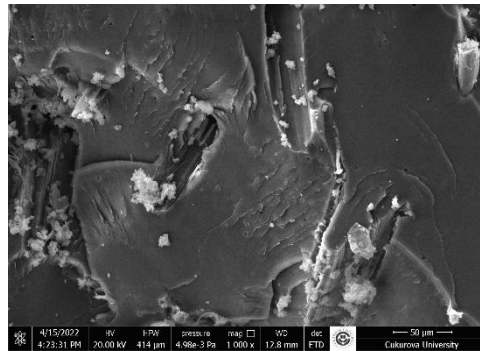
S-1



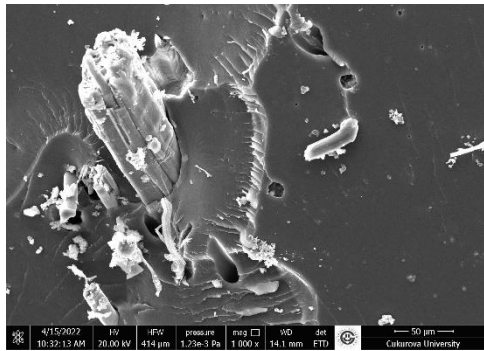
S-2



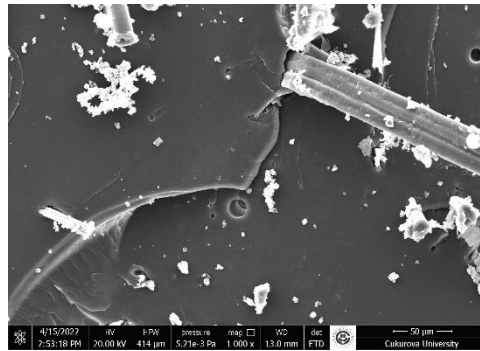
S-3



S-4

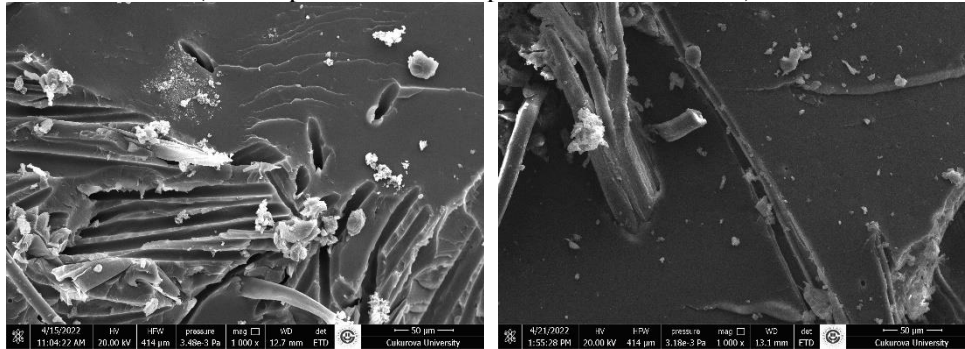


S-5



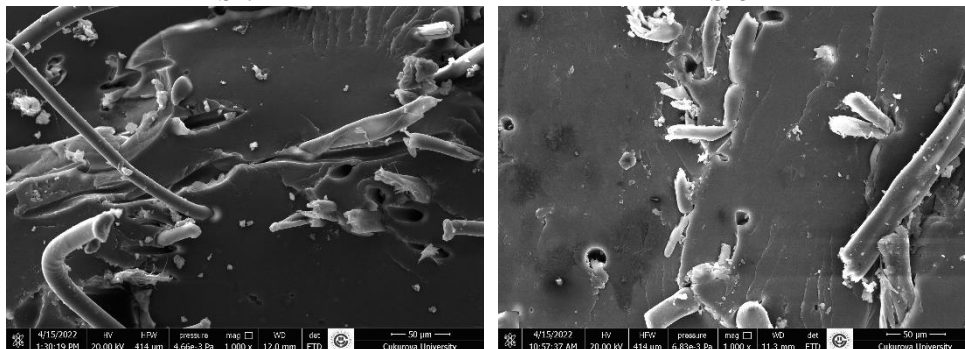
S-6

1.3.2. SEM image of fractured specimen of impact strength test for S-7, S-8, S-9, S-10, S-11, S-12 (S: Sample number in experimental date tables)



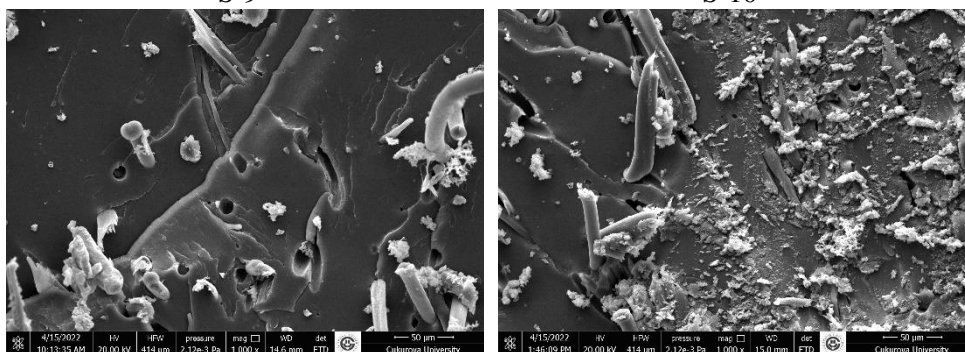
S-7

S-8



S-9

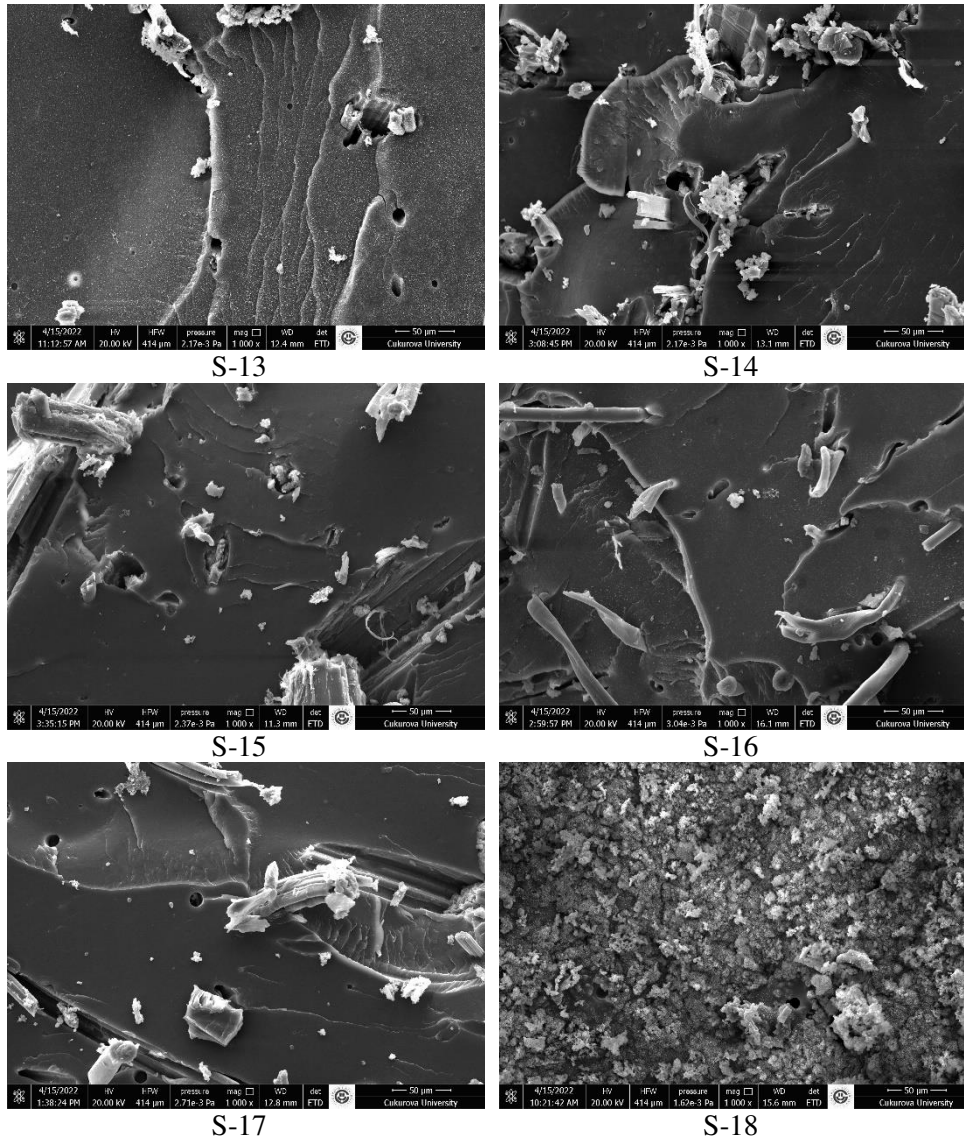
S-10



S-11

S-12

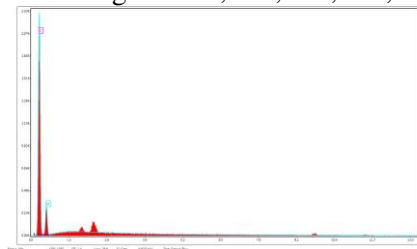
1.3.3. SEM image of fractured specimen of impact strength test for S-13, S-14, S-15, S-16, S-17, S-18 (S: Sample number in experimental date tables)



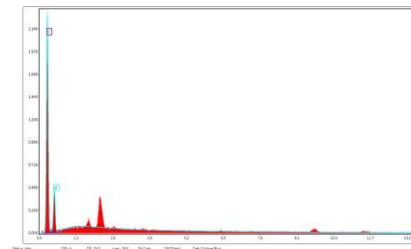
Appendix-2: Energy Dispersive X-ray Spectroscopy (EDS) of Composite

S: Sample number in experimental date tables

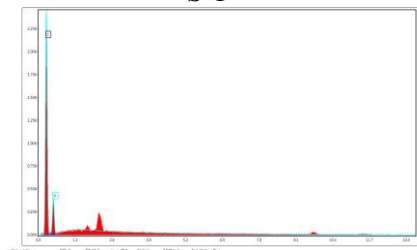
2.1 EDS image of S-1, S-2, S-3, S-4, S-5, S-6



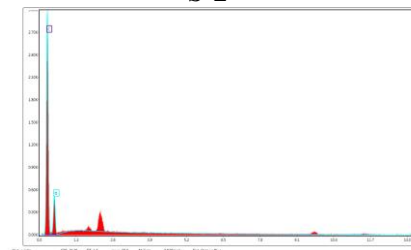
S-1



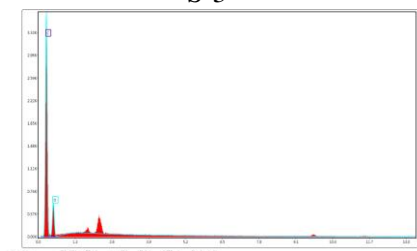
S-2



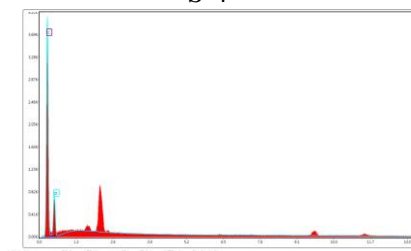
S-3



S-4

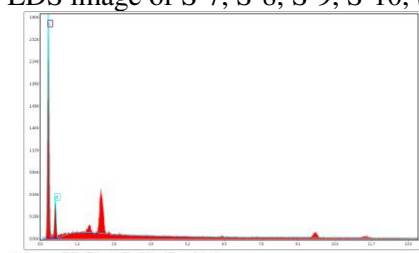


S-5

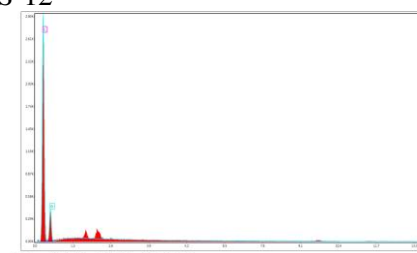


S-6

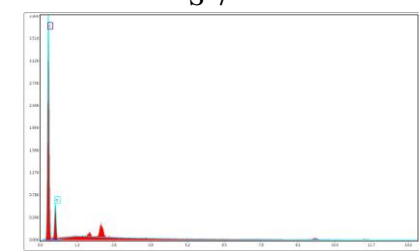
2.2 EDS image of S-7, S-8, S-9, S-10, S-11, S-12



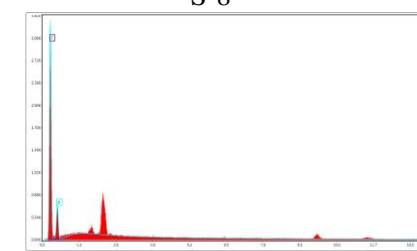
S-7



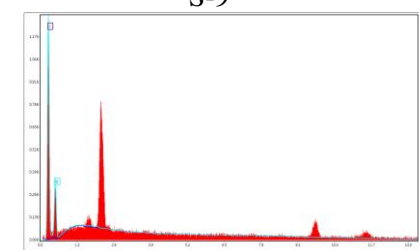
S-8



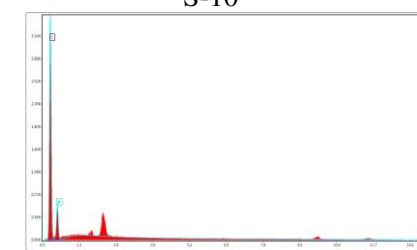
S-9



S-10

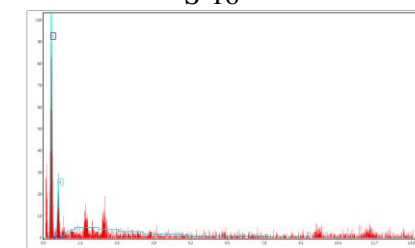
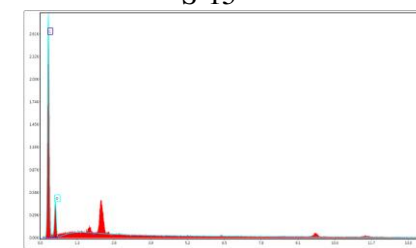
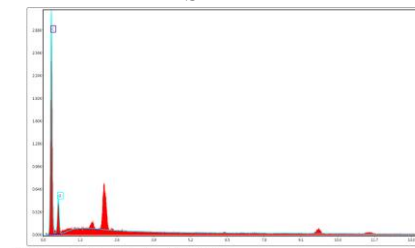
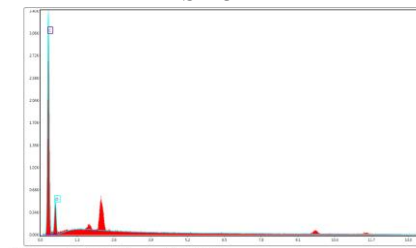
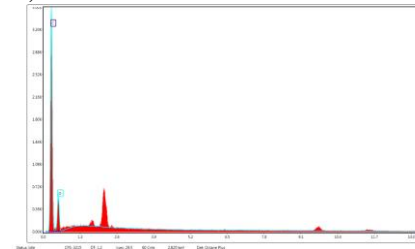
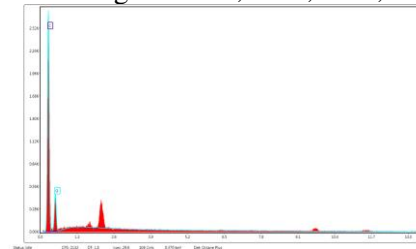


S-11



S-12

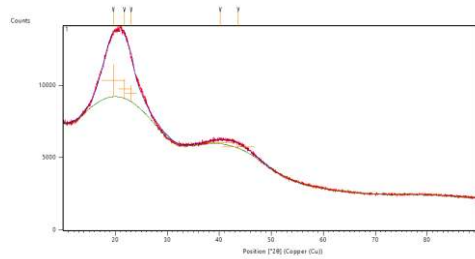
2.3 EDS image of S-13, S-14, S-15, S-16, S-17, S-18



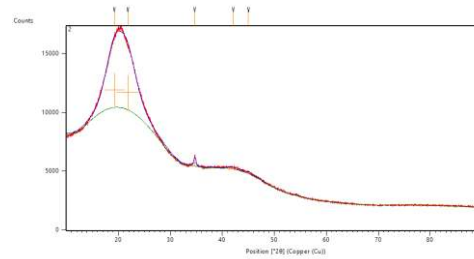
Appendix-3:X-ray Diffraction (XRD) of Composites

S: Sample number in experimental date tables

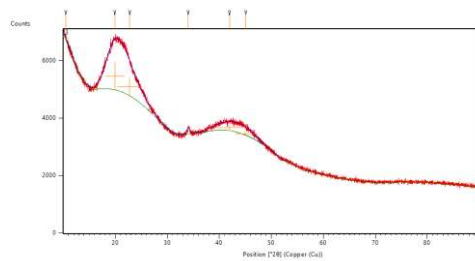
3.1.XRD image of S-1, S-2, S-3, S-4, S-5, S-6



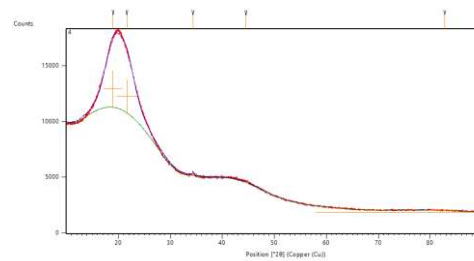
S-1



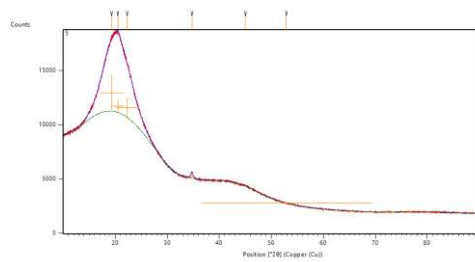
S-2



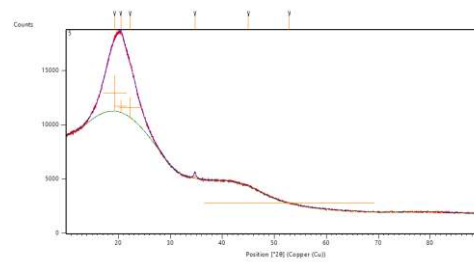
S-3



S-4

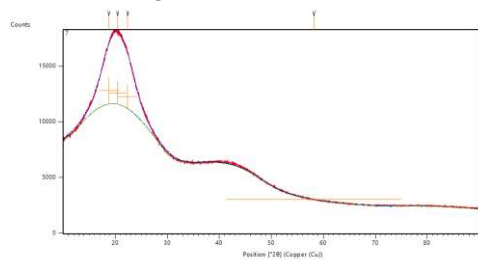


S-5

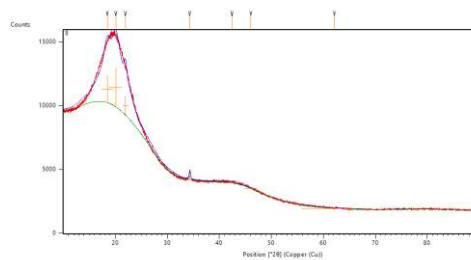


S-6

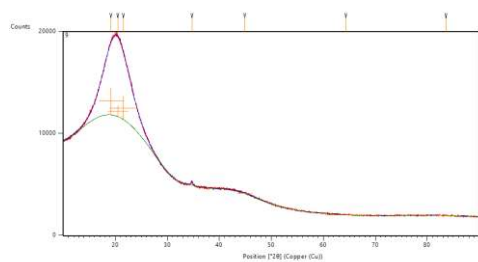
3.2.XRD image of S-7, S-8, S-9, S-10, S-11, S-12



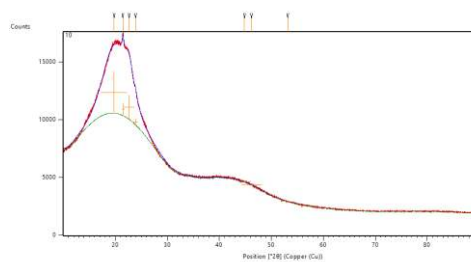
S-7



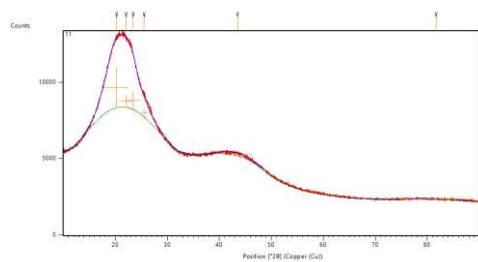
S-8



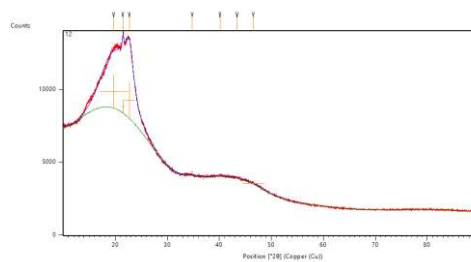
S-9



S-10

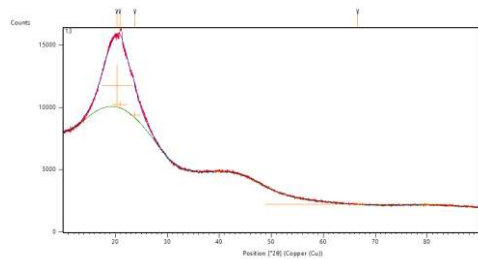


S-11

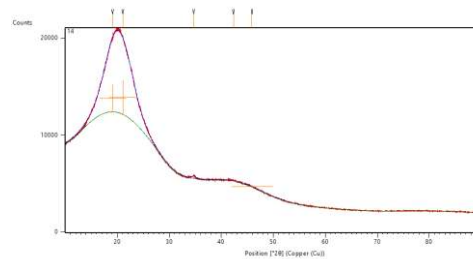


S-12

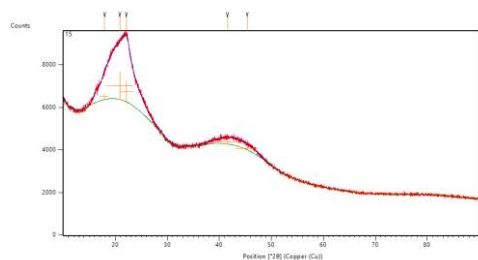
3.3.XRD image of S-13, S-14, S-15, S-16, S-17, S-18



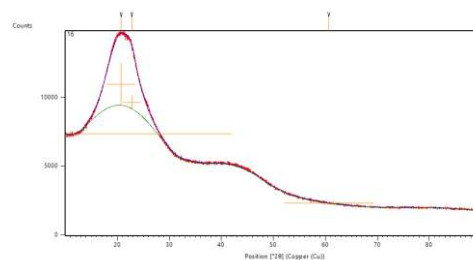
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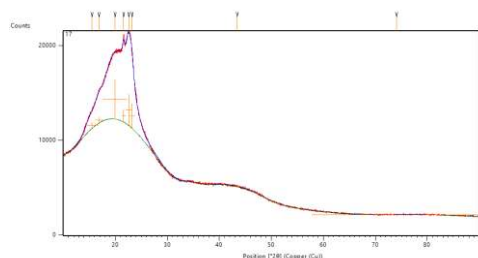
S-14



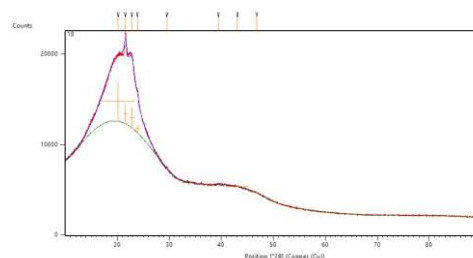
S-15



S-16



S-17



S-18