PRODUCTION, CHARACTERIZATION, AND EVALUATION OF HIGH POROUS AND FLEXIBLE CARBON FABRIC FROM DENIM WASTE FABRIC AS A RESIN REINFORCED COMPOSITE MATERIAL

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Master's Thesis

Department of Chemical Engineering

Programme in Reactor Design

Supervisor: Assist. Prof. Dr. Murat KILIÇ

Eskişehir

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SUPERVISOR APPROVAL

Master's student Gülşen Yağmur KAYALAR, whom I supervise, has completed this thesis titled PRODUCTION, CHARACTERIZATION, AND EVALUATION OF HIGH POROUS AND FLEXIBLE CARBON FABRIC FROM DENIM WASTE FABRIC AS A RESIN REINFORCED COMPOSITE MATERIAL. According to my inspections, the work is scientifically and ethically appropriate for the student to the thesis defense exam.

Supervisor

Assist. Prof. Dr. Murat KILIÇ

ABSTRACT

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The need for alternative materials in the world increases together with the need for light, high strength, and many unique features used in different sectors are also increasing. Carbon-based material-reinforced composites, which maintain their place among composite materials and whose research, and applications are increasing every year; they are resistant to thermal shocks, have high strength, are lightweight, and have low density. The biggest disadvantage of carbon fiber reinforced composite materials is the high raw material and production costs.

Evaluation of waste management for the production of materials that are so important to the world is the basis for high savings in possible costs. In this study, flexible activated carbon cloth (ACC) that can be used as an alternative to high-cost carbon fiber/resin composites were produced by applying carbonization and chemical activation processes to waste denim fabrics in the textile industry. For production, it has been tried with different techniques and parameters in the literature, and in this way, the optimum production conditions have been determined by applying characterization studies. Activated carbon cloths obtained were used to strengthen a construction structure.

When the results were evaluated, flexible activated carbon cloth produced from the waste of cotton denim was seen as a low-cost, environmentally friendly alternative effective material to be used as a carbon/resin composite material.

Keywords: Waste utilization, Activated carbon cloth, Carbon composite materials, Resin confining.

ÖZET

ATIK DENİM KUMAŞLARDAN YÜKSEK GÖZENEKLİ VE ESNEK YAPILI KARBON KUMAŞ ÜRETİMİ, KARAKTERİZASYONU VE REÇİNE TAKVİYELİ KOMPOZİT MALZEME OLARAK DEĞERLENDİRİLMESİ

Gülşen Yağmur KAYALAR

Kimya Mühendisliği Anabilim Dalı

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Dünyada her gün artmakta olan malzeme ihtiyacıyla birlikte farklı sektörlerde kullanılan hafif, yüksek dayanımlı ve kendine özgü birçok özellikler barındıran kompozit malzeme ihtiyacı da artmaktadır. Kompozit malzemeler arasında yerini koruyan ve her geçen yıl araştırmaları ve uygulamaları artmakta olan karbon esaslı malzeme takviyeli kompozitler; termal şoklara karşı dirençli, dayanımı yüksek, hafif ve düşük yoğunluğa sahiptirler. Karbon fiber takviyeli kompozit malzemelerin en büyük dezavantajı kullanılan hammaddelerinin ve üretimlerinin yüksek maliyetli olmasıdır.

Dünya için bu denli bir öneme sahip malzeme üretimi için atık yönetimi değerlendirmesi yapmak olası maliyetlerde yüksek bir tasarruf elde etmenin temelini oluşturmaktadır. Bu çalışmada, ülkemizin en önemli sektörlerinden biri olan tekstil sektöründeki atık kot kumaşlara karbonizasyon ve aktivasyon proseslerini uygulayarak, ileriye dönük yüksek maliyetli karbon fiber/reçine kompozit malzemelere alternatif kullanılabilecek esnek yapılı aktif karbon kumaşlar (AKK) üretilmiştir. Üretim için, literatürde yer alan farklı teknik ve parametrelerle denenmiş ve bununla birlikte karakterizasyon çalışmaları uygulanarak optimum üretim koşulları belirlenmiştir. Elde edilen aktif karbon bezleri, inşaat yapılarının güçlendirilmesi için kullanılmıştır.

Sonuçlar değerlendirildiğinde, pamuklu denim atıklarından üretilen esnek yapılı aktif karbon bezlerinin, reçine takviyeli karbon kompozit malzemelerde kullanılmak için düşük maliyetli, çevreye dost alternatif bir etkin malzeme olduğu görülmüştür.

Anahtar Sözcükler: Atık değerlendirme, Aktif karbon kumaş, Karbon kompozit malzemeleri, Reçine kaplama.

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Gülşen Yağmur KAYALAR

08/06/2022

STATEMENT OF COMPLIANCE WITH ETHICAL PRINCIPLES AND RULES

I hereby truthfully declare that this thesis is an original work prepared by me; that I have behaved in accordance with the scientific ethical principles and rules throughout the stages of preparation, data collection, analysis and presentation of my work; that I have cited the sources of all the data and information that could be obtained within the scope of this study, and included these sources in the references section; and that this study has been scanned for plagiarism with "scientific plagiarism detection program" used by Eskişehir Technical University, and that "it does not have any plagiarism" whatsoever. I also declare that, if a case contrary to my declaration is detected in my work at any time, I hereby express my consent to all the ethical and legal consequences that are involved.

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1. INTRODUCTION

The production and use of composite materials led the world, especially in the aerospace and defense industry. It has great importance with its different and wide application areas, such as transportation, electrical-electronic parts production, wind turbines in the energy sector, and tank-pipe production, especially in the construction industry. Carbon fiber composites have lots of advantages such as lightweight, high strength, high level of elastic modulus, and temperature resistance. The most important of disadvantages is that products produced with new technologies show up as production cost. A material production, which is not as strong as these products' structure but has high strength, could be used in various sectors. This study shows that new and advanced carbon materials are to be obtained by waste material utilization in the textile industry, which is one of the most important sectors of Turkey. It aims to recover the energy, raw material, and cost used in the production of textile products and contribute to economic development with innovative engineering products obtained with this product. Cotton denim fabric, which is one of the special products of the textile industry, is used for production. The manufacturing industry products are in the first three in the world's textile trade sectoral structure. On the other hand, the textile sector constitutes about 10% of Turkey's gross national product (GNP). The textile sector is also among the most significant in Turkey as well as in the world. In the last 30 years, textile/off-the-rack production, which has rapidly increased from past to present, has appreciated tenfold. Textile materials with increasing production show an increase in the same ratio in both pre-production wastes and post-production wastes, as well as the waste in the production process. While the total textile product is about 3.873.842 tons in 2009 in Turkey, the amount of the total industrial waste is about 485.485 tons/years. The amount of the waste, which happens during the production of textile materials is about 10% of the production in the industry. These waste product should be evaluated in different application areas, such as targeting the reduction of environmental pollution, ensuring the protection of natural resources, or zero waste management to ensure energy resource and cost savings. In this regard, various projects are being implemented in many countries around the world. Cotton denim fabric is determined as the raw material used in production. Denim fabric, one of the most important products of the textile industry, shows as a good raw material because of its close texture and wearproof. In this study, activated carbon fabric was produced as an alternative material for prospective high-cost carbon fiber/resin

composite applications by physical and chemical activation from cotton denim fabric. Besides, strengthening the mechanical properties of the fabric with resin reinforcement to the activated carbon fabric and to strengthen the columns of this structure in the construction sector was aimed. The results obtained in this study shows that the low cost, high mechanical and temperature resistant, light and flexible activated carbon fabric composites obtained with the two-phase reinforcing member can be evaluated as an alternative to carbon fiber/resin composite materials.

2. TEXTILE, TEXTILE WASTES, AND WASTE UTILIZATION METHODS

Textile includes textile products, textile fibers, semi-finished textile products, and all the products obtained using them. The textile industry has a wide range of products, including not only yarn and fabric production and the resulting apparel but also the industry's supply chain and other areas. The products obtained as a result of production are generally according to their usage areas; divided into two groups as ready-made clothing, ready-made home materials, and technical textiles (Kozak 2010; İlter 2015).

Ready-made clothes are the ones you buy in stores or online that come in standard sizes. Ready-made cloth is ready-to-wear clothing that can be purchased from store shelves or online. They are not specifically tailored to measurements but generalized to anthropometric studies. They are made from many different fabrics and threads.

The other group, technical textiles, is defined as textile materials and products that are primarily consumed for their functional properties and technical performances, rather than their aesthetic and decorative properties. However, today, aesthetic, and decorative features can be at the forefront in technical textiles as in-car upholstery. For this reason, it is possible to define technical textiles as textile products other than clothing products and home textiles. Synthetic and natural fibers such as polyester, polypropylene, nylon, viscose, cotton, glass, and aramid are used in the production of technical textiles (Can 2008).

2.1. Classification of Textile Fibers

The raw material used in textile is called fiber. Fiber is a group of colored or colorless fibers that can be bent (twisted) with pull-off resistance, adhered to each other, and whose length is very long compared to its width.

As seen in Figure 2.1, fiber types are divided into two by their chemical origin as natural and manufactured fibers.

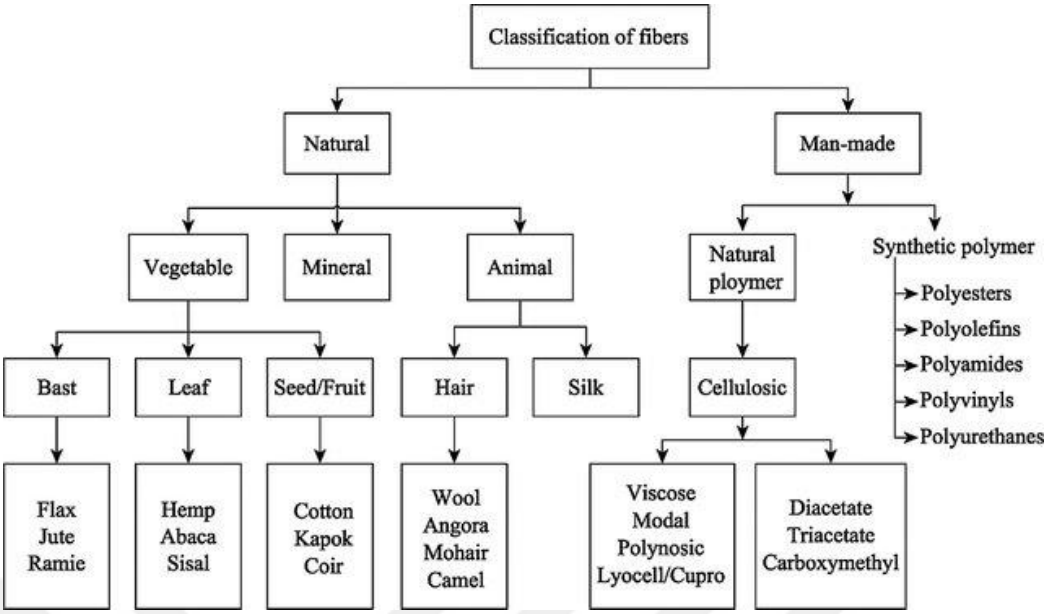

Figure 2.1. *Classification of fiber according to their sources (Jiang et al. 2020).*

2.1.1. Natural fiber

Natural fibers are those found in nature in fiber form. Traditionally, natural fiber sources are divided into 3 major groups as animal, plant, or mineral. Fibers from plant or vegetable sources are more accurately called cellulose-based and can be further classified by plant source. Natural fibers have very definite characteristics in each type, e.g., hair fibers will have features in common, as well as bast fibers; it is very difficult to tell a cashmere fiber from very fine wool, or flax hemp or ramie, but the difference is subtle (Jiang et al. 2020; Yimer and Ababa 2013).

2.1.1.1. Animal fibers

Animal fibers are natural fibers that consist largely of certain proteins. Animal fiber means that fiber is produced from animals such as sheep, alpaca, camels, goats, silkworms, etc. Silk, angora, cashmere, sheep's wool, etc. are given as examples of animal fibers.

2.1.1.2. Plant fibers

Plant fibers are natural fibers that consist largely of complex organic comprising three main components cellulose, hemicellulose, and lignin often in combination. Examples include bamboo, cotton, flax, hemp, jute, wood, sisal, etc. Plant fibers are divided into three main groups according to their sources as bast, leaf, and seeds of plants

(Popescu 2017; Qin 2016). Table 2.1. is shown as the chemical contents of some plant fiber. Each example has a different ratio component therefore each fiber type has its specific characterization properties.

Table 2.1. *Chemical contents (%) of some plant fibers (Ekundayo and Adejuyigbe 2019; Yimer and Ababa 2013).*

Fiber Type	Cellulose	Hemicellulose	Lignin	Pectin
Bagassa	32-48	21	19,9-24	10
Bamboo	26-43	15-26	21-31	-
Cotton	82-96	2-6	0.5-1	5-7
Flax	60-81	14-19	2-3	0.9
Hemp	70-92	18-22	3-5	0.9
Jute	51-84	12-20	5-13	0.2
Wood	45-50	23-30	27	2-2.5

Cotton fiber is one of the members of the seed-hair main group. Cotton fiber, soft and comfortable cotton, is known as the most widely used fiber with its use in clothing, home furnishings, medical and surgical materials, and many other industrial products. It contains 82-96% cellulose, 1.5-5.0% protein and pectin, 1.0-1.2% inorganic substances, 2.0-3.5% moisture and 0.5-0.6% waxes and oils. What is important here is that cellulose is a carbohydrate that forms a large part of the cell structure in plants (Can 2008; Ekundayo and Adejuyigbe 2019; Erkan 3013; Yimer and Ababa 2013).

Cellulose

Cellulose is the most abundant complex carbohydrate produced by living organisms, is found in main cell walls of plant fibers (Popescu 2017). The biopolymer is shown in Figure 2.2., whose generic name is poly-(1,4-β-d-glucose), is biodegradable thanks to its suitable mechanical properties. It can be produced in high amounts and contains a high amount of carbon element. Due to its high carbon content, materials containing high cellulose composition are suitable materials for the production of carbonaceous materials.

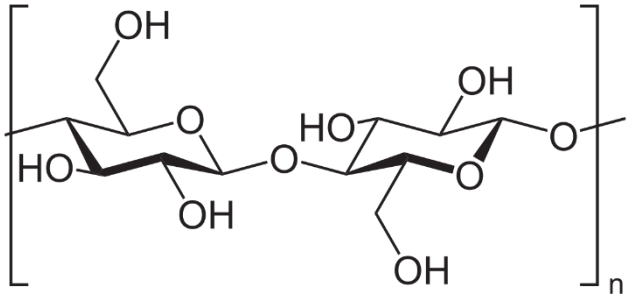

Figure 2.2. *The chemical structure of cellulose (Qin 2016)*

Hemicellulose

Hemicellulose is the common name of some complex carbohydrates or polysaccharides found together with cellulose and pectin in the walls of annual and perennial plant cells. It is classified into four groups as xylans, mannans, mixed linkage β-glucans, and xyloglucans (Reise and Waller 2009).

Lignin

Lignin, which is found in the cell wall of plants, provides the woody structure and durability of the plant together with cellulose. It fills the spaces in the cell wall between cellulose, hemicellulose, and pectin components. (Saake and Lehnen 2000, Blackwell 2014).

2.1.2. Manufactured fiber

Manufactured fibers, whose name is man-made fibers are produced by chemical reactions controlled by people instead of occurring naturally. Common synthetic fibers include nylon, acrylic, olefin, polyester. Synthetic fibers stated in other words manufactured fibers are formed by combining monomers to form a long chain structure. This process is called polymerization.

2.2. Textile Waste

All kinds of substances that we do not need and remove can be defined as waste. It can be categorized as solid, liquid, and gas. Solid waste is both decomposable and non-degradable materials, including semi-solid sludges from domestic, commercial, or industrial processes, including mining, agricultural operations, and water treatment units.

Textile wastes are wastes that come out during the production process in textile factories or after consumption by consumers. In this context, textile wastes are classified as either pre-consumption or post-consumption.

- Pre-consumption wastes consist of textile, fiber, and cotton industry by-product materials that are regenerated for automotive, aerospace, home building, furniture, bedding, coarse yarn, household goods, paper, clothing, and other industries.
- Post-consumption waste is defined as household items made from any clothing or textile materials that the consumer no longer needs and decides to throw away. These materials are discarded either because they are too old and worn out or because they are outdated.

Recycling and waste utilization are seen as the best solution for both natural and synthetic-based textile wastes, for reasons such as reducing the environmental impacts in the production processes, preventing the problems that will occur when they are thrown into nature (Altun 2016; Kozak 2010).

2.3. Textile Waste in Turkey and World

The textile sector constitutes approximately 10% of the gross national product in Turkey and is one of the most important sectors in Turkey as well as around the world. The utilization of textile wastes, which have reached high levels both in Turkey and in the world, within the scope of waste management is as important as other waste management (Altun 2016; Kozak 2010).

As seen in Table 2.2., the total amount of textile product production in 2009 was 3,873,842 tons/year. The total amount of industrial waste is 485,485 tons/year. Approximately 10% of waste textile products are formed during industrial productions. Considering that the textile production index has increased since 2009, it can be said that the amount of waste generated has also increased.

Table 2.2. *Generated waste ratios and waste amounts in textile processes (Altun 2012).*

Production Type	Production amount in 2009, tons/year	Waste ratio, %**	Waste amount, average value, tons/year
(Cotton) carded yarn including ring spinning	288,940	12-20.6	46,230
(Cotton) combed yarn including ring spinning	79,836	23-50.6	29,939
(Cotton) open-end yarn	303,607	10-19.2	43,944
Worsted yarn	68,283	21-33	18,436
Woolen yarn	302,005	>40	120,802
Polypropylene yarn	310,00 (SUSEB)*	14	43,400
Acrylic yarn	308,00 (SUSEB)*	10-13	35,420
Polyester yarn	360,00 (SUSEB)*	3***	10,800
Polyamide yarn	77,600 (SUSEB)*	3	2,328
Chenile, fancy yarns, hand knitting yarns, embroidery yarns	293,806	8-13	30,849.5
Cotton weaving	26,214	Yarn waste: 1-2/ Trimming: 1-6	Yarn waste: 393 Trimming: 917
Wool weaving	600	Yarn waste: 1-2/ Trimming: 1-6	Yarn waste: 9 Trimming: 21
Man-made yarn weaving	121,314	Yarn waste: 1-2/ Trimming: 1-6	Yarn waste: 1,819 Trimming: 4,245
Knitting	581,632	2-4	29.081
Knitting garment	70,000	3-23	9,100
Nonwoven	55,625	~ 5	2,781
Cloth dyeing	188,184	0.5-2	2,351.5
Ready-made (except garment)	301,083	2-15	10,537
Apparel garment	137,113	2-20	15,082
Total	3,873,842		458,485

*Data obtained from Association of Man-Made Fibre Producers, in Turkey (SUSEB).

**Waste ratios were determined in factory studies and shrink values declared by local chambers of industry. Database of the Gaziantep Chamber of Industry, Karapınar Chamber of Industry, and Adana Chamber of Industry were used.

***Excluding hard waste and texturing waste; during the polymerization process approx. 0.06% of hard waste; during texturing processes approx. 1 - 2% of yarn waste occurs.

The second important source of textile waste is the waste generated after its use. Waste product per person in Turkey is 1.77 kg/day according to TUIK 2012 data. The ratio of textile products in this amount is 1.14% (Altun 2012).

The total amount of textile waste in households in Turkey is calculated as follows (Turkish population in 2009 was 76,561,312):

$$Textile\ waste\ in\ households = \frac{0.014 \times 1.14\ kg\ \times\ 365\ day \times\ 76{,}561{,}312}{1000} =\ 564{,}657\ yons/year$$

According to the producer survey, the most consumed raw materials are cotton at 29% and polyester at 24%. The amount of industrial solid waste disposed of by industrial groups and disposal methods of used and waste textile products are shown in Table 2.3.

Table 2.3. *Ultimate disposal methods for industrial textile waste according to Turkstat (Altun 2012).*

Disposal methods	**%**
Dumping site	46.64
Controlled landfill	37.94
Incineration	7.62
Composting	-
Stored within the establishment	4.60
Used as filling material	2.26
Dumping in an open area	0.83
Dumping into the sea, river, and lake	0.02
Others	0.09

In the European Union (EU), an average of 5.8 tons of textile products are thrown away annually by consumers. Of these textile products, only 1.5 million tons, or 25%, are recycled by charitable organizations and industrial enterprises, and the remaining 4.5 million tons of waste is sent to landfill or waste incineration plants (Koszewska 2018; Üçgül and Turak 2015).

Table 2.4. shows the extent of textile consumption and, subsequently, the volumes of disposal of fashion waste in Europe according to the European Commission's Institute for Prospective Technological Studies in 2020 (European Commission's Institute 2022).

Table 2.4. *The volumes of disposal of fashion waste in Europe on 22 Jan. 2020 (European Commission's Institute 2022).*

Country	Population	Yearly Total Textile waste, tons	Yearly textile waste per person, kg	Yearly landfilled textile waste per person, kg
Italy	60,359,546	465.925	7.7	4.4
Portugal	10,276,617	81.715	8.0	4.6
Austria	8,858,775	62.446	7.0	4.0
United Kingdom	66,647,112	206.456	3.1	1.7
Belgium	11,467,923	169.949	14.8	8.4
Czech Republic	10,649,800	108.273	10.2	5.8
Denmark	5,806,081	18.134	3.1	1.8
Spain	46,934,632	98.881	2.1	1.2
Finland	5,517,919	14.934	2.7	1.5
Germany	83,019,213	391.752	4.7	2.7
Netherlands	17,282,163	102.261	5.9	3.4
France	67,028,048	210.001	3.1	1.8
Ireland	4,904,226	22.944	4.7	2.7
Poland	37,972,812	103.683	2.7	1.6
Hungary	9,772,756	23.190	2.4	1.4

Developing waste evaluation methods and creating alternative new methods can reduce waste textile products both in Turkey and across the world, creating a more sustainable future.

2.4. Waste Utilization Methods

Waste utilization methods of textile wastes are generally grouped under the following categories:

- Mechanical methods
- Thermo-mechanical methods
- Thermal methods
- Chemical methods
- Energy obtaining methods
- Other methods

With these methods, both recycling and disposal processes of waste materials can be carried out (Altun 2016).

Wastes are brought into a fibrous form that can be used in the production of yarn, fabric, and nonwovens by using mechanical methods. In the thermo-mechanical method,

it is remelted into granules and the obtained granules are used in the production of plastics and fibers. In the chemical method, especially synthetic-based wastes are generally recycled to raw material or intermediate products through chemical depolymerization methods, and the obtained products are used in various fields, such as textile finishing materials, fibers, unsaturated resins.

Thermal methods, on the other hand, refer to heat treatments. This method aims to dispose of textile waste. There are 4 applications for thermal methods: incineration, gasification, liquefaction, and pyrolysis.

For incineration, combustion is the rapid chemical combination of flammable elements in the fuel with the oxygen of the air and the release of heat. For burning, it is necessary to form a rapid chemical composition by reacting with the oxygen of combustible gases. There are three essential conditions for combustion to occur: (1) the temperature required to initiate the combustion reaction, (2) the air/fuel turbulence required for combustion to occur, and (3) the sufficient time for combustion.

Gasification is based on the high-temperature degradation of solids such as carbon-containing biomass. With the gasification technique, a gaseous fuel can be obtained from biomass with a high efficiency to be used in turbines that provide power and heat working.

With the liquefaction method, biomass can be converted into 2-10% gas, 40% liquid product, and 5-10% solid product under conditions where pressure, high temperature, aqueous medium, and catalyst are most suitable.

Pyrolysis is the separation of organic substances into gas, solid, and liquid products by heating them in an oxygen-free environment. In this process, the theoretically required amount of heat should be at a level that will disrupt the chemical structure of the organic matter and enable the formation of new chemical substances (Üçgül and Elibüyük 2014).

3. ACTIVATED CARBON CLOTH

Nowadays, materials which have low-density, light, and economical are constantly increasing. Studies show that carbonaceous materials become prominent compared to other materials due to their advantages such as high hardness, conductivity, special strength, low density, mechanical properties at high temperatures, and non-oxidation at these temperatures (Bakış and Doğru 2016). Graphene, carbon nanotube, activated carbon, carbon fiber, carbon foam, which are advanced carbon materials produced from bio-waste, are environmentally friendly materials that can be used in many fields, and have not lost their popularities until today (Kılıç 2015).

Activated carbon cloth, one of the advanced carbon materials, is a lightweight form of carbon with high adsorption capacity and speed, high pore volume, as well as interesting mechanical properties such as flexibility or toughness. Carbonization and chemical activation processes should be applied to increase the porosity of the fabric and have a high surface area (Pastor et al. 1999; Cordero-Lanzac et al. 2018). This form called activated carbon fabric has many technological advantages over activated carbon forms such as powder, granules, pellets, etc. (Küçükgül 2004; Yörük 2019). The success of activated carbon cloths' in these new technologies is based on certain properties of activated carbon cloths, including an easily accessible pore structure, rapid pore diffusion afforded by low-dimensional fibers, and various surface functional groups that provide a reactive surface for a variety of applications (Nieto-Delgado et al. 2019).

3.1. Production

Activated carbon can be produced from the appropriate starting material to be selected in one of powder, granule, fiber, or spherical forms. Activated carbon fibers have more adsorption capacity and kinetics than granular and powdered activated carbons due to their easily accessible micro and macro pores. Activated carbon fibers can be produced not only in fiber form but also in fabric and felt form.

The production steps of activated carbon cloth and the production of activated carbon show high similarity. The main difference between the two productions is the precursor used. The production material required for the activated carbon cloth must be a fabric/textile product. The production process is divided into two as physical (gas) and chemical activation. Activated carbon cloth production generally includes the following steps.

1. Removal of water in the structure of carbon material (Dehydration),
2. Conversion of organic molecules to elemental carbon and removal of volatile substances (Carbonization),
3. Pore formation by impregnation of chemical substances and making functional groups on the surface suitable for adsorption (physical or chemical activation).

The main purpose in these stages is to reveal the carbon skeleton in the plant-based raw material and to remove other carbon-free structures, if any, from the carbon skeleton of the raw material. Another step is to increase the porosity. For this purpose, the raw material is first applied to carbonization and then to the activation process (Aygün 2002; Nieto-Delgado et al. 2019; Huidobro et al 2001; Rodriguez-Reinoso 1999; Sevimli 2017; Yörük 2019).

3.1.1. Carbonization

The carbonization of an organic precursor is one of thermal decompositions in an inert atmosphere when mainly oxygen and hydrogen are evolved. During the carbonization process, H, N, O, and S elements are separated by the pyrolytic decomposition of the carbon-containing raw material. Volatile, low molecular weights, are released first. This is followed by light aromatics, and finally, hydrogen gas. The carbonization process is usually carried out at 400-850°C using an inert atmosphere, such as nitrogen. Although this process alone is not sufficient for adsorption studies, it is suitable for increasing the pore structure and for carbon cloth-composite studies. At this stage, the pore structure and properties of carbon largely depend on the raw material used (Aygün 2002; Kılıç 2009; Pastor et al. 1999).

3.1.2. Activation

Activation is required to increase the pores in the carbonization process and the internal surface area of the structure. Activated carbon cloths' porosity is determined by using the activation process and the precursor material. The activation process can be applied in two ways as physical or chemical activation as shown in figure 3.1. (Huidobro et al 2001; Nieto-Delgado et al. 2019; Rodriguez-Reinoso 1999).

Physical activation consists of two steps: carbonization of the starting material and activation of the carbonized structure. By removing oxygen and hydrogen from the raw

material during carbonization, a porous carbon skeleton is produced. During activation, as a result of the activation of the carbonized material in an oxygenated environment, the volatile substances produced by the combustion of carbon with oxygen and then they are removed from the environment, thus increasing the pore volume and surface area to a large extent. The physical activation process is carried out by giving water vapor, air, or carbon dioxide in the temperature range of 700–1100˚C to increase the surface area of carbon-containing raw materials and to develop pores.

Activated carbons obtained by physical activation are not preferred as an adsorbent or in filters. The oxygen content of the substance used for activation affects the reactive part of the carbon skeleton, and the degradation of the carbon skeleton occurs at different rates in different parts of the surface.

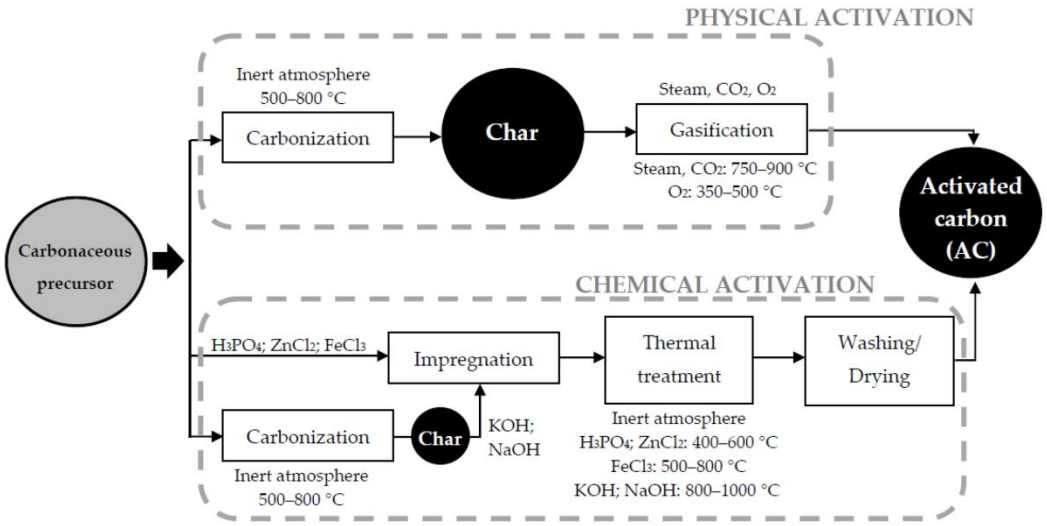

Figure 3.1. *Basic process flow chart for physical and chemical activation (Bedia et al. 2020).*

In chemical activation, the activation process is applied by impregnating with various chemicals such as $ZnCl_2$, H_3PO_4, NaOH, KOH, H_2SO_4, K_2CO_3 in order to minimize volatiles and increase porosity. H_3PO_4, $ZnCl_2$, and KOH are activation agents that are generally used in the activation phase. These chemical activation agents used usually cause dehydration of the raw material. Thus, they prevent the formation of liquid products by affecting pyrolytic decomposition and increasing the yield of solid products.

Two methods are used in the production of activated carbon by chemical activation. In the first method, the biomass is impregnated with chemical activation agents and then

heat treated. In the other method, at temperatures of 550°C and below, the biomass is first carbonized, then activated with a chemical activation agent, and finally heat-treated high temperatures in the range of 400˚C to 900˚C (Alves 2021; Bedia 2020; Kılıç 2009).

3.2. Application Fields

Activated carbon cloths have plenty of advantages over other activated carbon forms, like powder and granules, with their distinctive features such as high surface area, light and flexible structure, and high-temperature resistance.

The usability of carbon cloth in many areas has increased with the expansion of the usage areas of carbon fibers. It plays an active role in many areas from defense clothing to the automotive sector, from medical applications to composite production. Today, several reviews report that various studies have been carried out in the production of electrodes for supercapacitors and production of adsorbent materials (Cordero-Lanzac; Mendoza-Castillo 2016).

In short, the fact that carbon fabric is used in such a wide range makes it more preferred thanks to its low production cost and useful features.

3.3. Review of Related Literature

Cordero-Lanzac et al. (2018) prepared the activation carbon cloth from denim cloth waste using different impregnation ratios of H_3PO_4 as 1, 1.5, and 2 and using different carbonization temperatures as low temperature in 500°C and high temperature in 900°C. BET, SEM, XPS analysis are obtained for characterization of activated carbon cloths prepared. As a result, the highest surface area (2032 m^2/g) was obtained in activated carbon cloth produced under chemical activation conditions at 900°C, 0.5 H_3PO_4/Denim cloth impregnation ratio. This flexible activated carbon cloth was used as an electrode for the electric double-layer capacitor.

Nieto-Delgado et al. (2019) have used plant-based natural fiber (jute and henequen) as renewable precursors to produce activated carbon cloth. Chemical, impregnation with $ZnCl_2$ chemical activation agent, and physical, based on CO_2 and H_2O, activation has been applied at 600 and 800°C, respectively. Elemental analysis, pore structure, SEM, BET, and surface chemistry are carried out to the material for characterization. They evaluated the adsorption properties of the activated carbon cloth that was obtained from jute and henequen. As a result, the surface area of the samples ranged from 480 to 1200

m^2/g. Chemical activation applications develop a micro-porous range which was suitable to adsorb micro-pollutants such as phenol and cadmium, while physical activation generates porosity in the micro and low mesoporous materials which were suitable to the removal of molecules with steric restrictions such as methylene blue.

Polovina et al. (1997) produced cellulose-based activated carbon cloth by using viscous rayon cloth in a CO_2 flow at 850°C for 1 hour. After that, it has been oxidized by air, nitric acid, hydrogen peroxide, and iron nitrate cristallohydrate melt treatments. As a result of the studies carried out to observe the differences between the oxidation methods and the nature and extent of the oxygen complexes formed, the highest oxidation degree was obtained with the $Fe(NO_3)_3.9H_2O$ process, while the lowest oxidation was obtained with the H_2O_2 process from the air-oxidized activated carbon cloths.

Yörük (2019) has prepared activated carbon cloth from cellulose-based textile materials. Experimental conditions were tried at various temperatures as 500, 600, 700, 800, and 900°C by using $ZnCl_2$ and H_3PO_4 chemical activation agents as 0.5, 1 ratio agent/cloth. While H_3PO_4 impregnation contributed to the formation of large pores, narrow pore formation was observed in fabrics where $ZnCl_2$ was applied. A new structure with a controllable pore diameter was revealed by using H_3PO_4 and $ZnCl_2$ chemical activating agents together at 1 and 0.5 activation agent/fabric ratios, respectively. Under these chemical conditions, the highest BET surface area of 1506 m^2/g was obtained at 900°C.

Rodriguez-Reinoso and Marsh's group carried out a detailed study on the production of activated carbon cloth from viscose rayon fabric and published their work in a series (Huidobro, 2001; Pastor, 1999; Rodiguez-Reinoso, 2000a, Rodriguez-Reinoso 2000b). In the first stage of their study, they investigated the carbonization of viscose rayon precursor fabric and the parameters affecting carbonization.

The results of the first study show that low carbonization temperatures decrease the adsorption capacity and micropore volumes and increase them with mesopore volumes. This also means low-density microporosity (Pastor 1999).

In their second study, they examined the effects of physical activation. According to this study, the effects of carbon dioxide and water vapor on the product during activation were investigated. It provides a continuous narrow micropore formation in all

activation stages with carbon dioxide, while in water vapor, unlike carbon dioxide, it allows the expansion of micropores and leads to the formation of a low micropore volume. In addition to the pore structures, the breaking load of the cloths was found to be low in studies with physical activation (Rodríguez-Reinoso 2000a).

According to the low breaking load result of the cloths they obtained, their third study was carried out to investigate the effects of the carbonization process on CO_2 activation. All chars were activated under a flow of carbon dioxide at 825°C. In the carbon dioxide activation process of the viscose rayon, the slow heating rate made the carbonization product more homogeneous (Rodriguez-Reinoso 2000b).

In their fourth and last study, the chemical activation process was investigated. The chemical activation of viscous rayon fabric was carried out by impregnation with a different chemical activation (NH_4Cl, $AlCl_3$, $ZnCl_2$, $CuCl_2$, $FeCl_3$, H_3PO4, and Na_2HPO_4), followed by carbonization in an inert atmosphere. According to the results obtained, the kind of activating agent significantly affects the yield, elemental composition, and textural properties of the cloth obtained. The activated carbon cloth produced with the H_3PO_4 chemical agent has the highest specific surface area (Huidobro 2001).

4. COMPOSITE MATERIALS

In the 1930s, the production of modern composite started with the discovery of glass fiber in America, and glass fiber reinforced composite materials took their place in the global market. Composite materials in the broadest sense can be defined as materials consisting of two or more components that have different physical or chemical properties and are insoluble in each other.

Composite materials can be seen as relatively new and high-tech materials when evaluated in terms of materials science. The most important feature of the composite material is that it is homogeneous at the microscopic level. (Mazlumlar 2001; Ryutoku et al. 1990; Vinson and Sierakowski 2008).

Typically, composite material consists of two main elements: the matrix, and the reinforcement material. Generally composite materials are formed by reinforcing fibers in a matrix resin, as shown in Figure 4.1. The effect of the reinforcement element in the development of the properties of composite materials is quite significant.

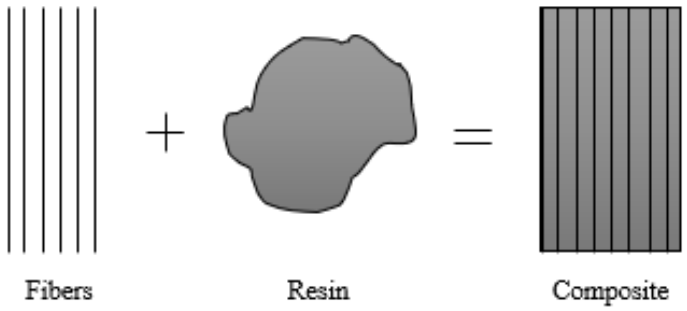

Figure 4.1. *Composition of fiber-reinforced composite material (Mazlumlar 2001).*

Reinforcement materials with high elastic modulus, high strength, and low density are required according to the usage area of the material produced. Reinforcing elements can be fibers, particles, or whiskers, while matrix materials can be metals, plastics, ceramics, or carbon-based materials in the form of continuous fibers, particles, and scraps (Gürbüz and Mutuk 2019; Mazlumlar 2001).

Composite materials can take many forms but can be divided into four categories depending on the reinforcement mechanism. These categories are whisker-reinforced, structural, particle-reinforced, and fiber-reinforced.

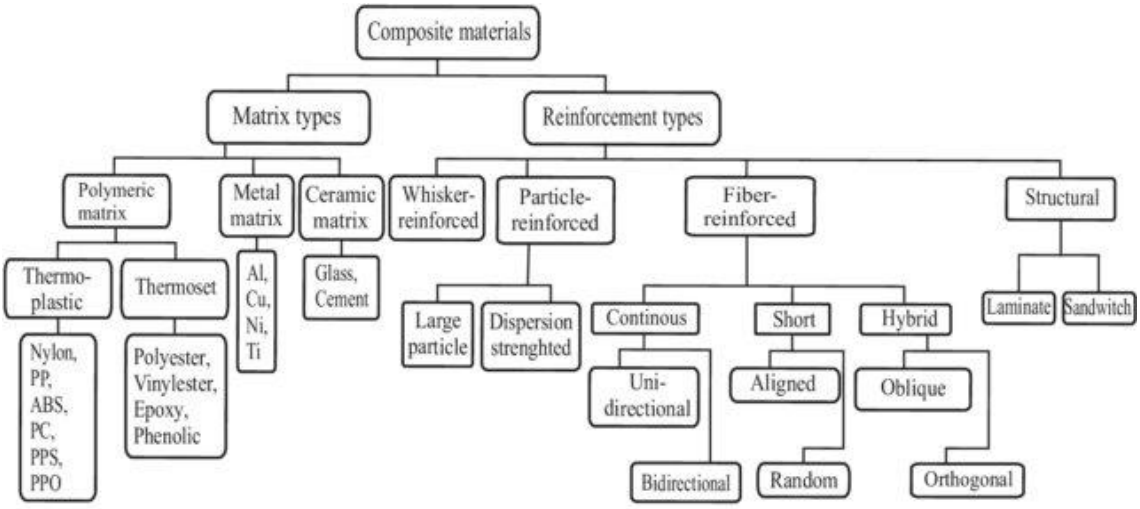

Figure 4.2. *Classification of composite materials (Priyanka et al. 2017).*

Composite materials are divided into three main groups according to the matrix basis used (Chandramohan and Marimuthu 2011; Priyanka et al. 2017).

1. Metal Matrix Composites (MMC)
2. Ceramic Matrix Composites (CMC)
3. Polymer Matrix Composites (PMC)

4.1. Properties of Composite Materials

Composite materials have outstanding properties compared to conventional materials. A composite material shows the following properties.

- Low density
- High tensile strength
- High hardness
- High tensile strength at elevated temperatures
- High corrosion resistance
- High fatigue resistance

- Low coefficient of friction
- High chemical abrasion resistance

In addition, due to their flexible and one-piece production, their aesthetic appearance, and the ability to determine the shape of the product, they are becoming more and more widespread in industrial products. Another advantage of using composites is that their strength properties of layers can be adjusted due to its anisotropic property. In addition to all of these advantages, composite materials also have some disadvantages(Gülcan 2019; Örs 2017).:

- High production costs
- Low breaking strain
- The quality of the material depends on the quality of the production methods, there is no standardized quality.
- Composites are easily damaged because they are brittle materials, and their repair can create new problems.

4.2. Types of Fibers Used in Production Composite Materials

The most common fibers used as reinforcing elements in composite materials are glass, aramid, and carbon. All of these fibers have been developed in the second half of the twentieth century. The purpose of using these fibers as reinforcement elements is to increase the strength of the composite part to be produced (Gülcan 2019).

4.2.1. Glass fiber

The most common fibers used in textile-reinforced composite materials are glass fibers. The tensile strength and specific strength of glass fibers are quite high, and the cost is lower than other fibers. Glass fibers are mostly silicate (SiO_2) based and contain calcium, sodium, aluminum, boron, and iron oxides. Glass fibers are expressed with different names such as E glass, C glass, S glass according to the chemical compositions they contain. E glass is a good electrical insulator, C glass shows more resistance to chemical corrosion than other glass types, while S glass has a high silicate content, which provides resistance to higher temperatures. E glass is generally preferred as a reinforcement element in the construction of composite materials (Gülcan 2019).

4.2.2. Aramid Fiber

Aramid is a polymer of the polyamide (nylon) class. It is also known by the trade name Kevlar and Nomex. Aramid fiber, chemical structure, amide (-CO-NH-) bonds, at least 85% of the lo synthetic polyamide directly bonded to two aromatic rings, is a synthetic fiber known for its fire in fire-resistance and strength properties, generally used in protective clothing where high temperatures are required. In addition to all these features, aramid fibers have some disadvantages. When exposed to ultraviolet light, the mechanical properties of aramid fibers weaken and disappear over time. Because aramid fibers show weak properties under pressure (Gülcan 2019).

4.2.3. Carbon Fiber

Carbon is a crystalline material with a density of 2.268 g/cm^3. Carbon fibers are a group of fibers that developed later than glass fibers and are widely used. Carbon and graphite fibers are also called organic fibers because they are produced from organic materials. Carbon fibers are fibers that contain at least 90% carbon by weight. When carbon fibers are subjected to very high heat treatment, the fiber becomes fully carbonized. Carbon fibers are an important fiber type used in the production of composites. These fibers, which have been used since the 1960s, have high tensile strength and E modulus despite their low density. The properties of carbon fibers that can resist high temperatures vary depending on the temperature of the final treatment in their production.

PAN, cellulose-based fabric (rayon), and pitch are used as raw materials. Therefore, they are named according to the materials from which they are produced. In today's world, rayon is only used for very low modulus fibers. This raw material is heated to a range of 900-3000°C in an inert atmosphere and tensile strength is applied at the same time. The process that is applied in these steps provides material strength and toughness. Polyacrylonitrile-based (PAN) fibers have a tensile strength of 2413 to 3102 MPa and their cost is low. Pitch-based fibers obtained by refining petroleum have a tensile strength of 2069 MPa. Mechanical properties are not as good as PAN-based fibers, but the production of pitch-based fiber's cost is low.

Carbon fiber generally shows high endurance and hardness properties when combined with epoxy matrices. In addition, its structure can be crystalline, amorphous, or partially crystalline. Carbon fibers are commercially available in two forms as

continuous fibers and chopped fibers. These fibers can be combined with all resins (Gülcan 2019; Örs 2017).

4.3. Manufacturing Methods

In most composite production procedures, molding is employed to shape the resin and reinforcement. A mold tool is necessary before and during the cure to shape the unformed resin/fiber combination. Typically, the manufacturing process can be categorized as open and closed molding techniques shown in Table 4.1. based on the form of molding.

Table 4.1. *Composite manufacture types of molding techniques (Akter 2021).*

Composite Molding	
Open Molding	Hand lay-up
	Spray-up
	Filament winding
Closed Molding	Vacuum bag molding
	Resin transfer molding
	Vacuum-assisted resin transfer molding
	Resin film infusion
	Compression molding
	Injection molding
	Pultrusion molding

Open molding is generally used to laminate the process easily. In general, open molding techniques are classified based on resin reinforcement applications to the mold. This method includes hand lay-up, spray-up, and filament winding applications. Open molding methods are preferred in basic composite productions due to their flexibility, easy production, simple procedures, and minimum cost.

Closed molding techniques are generally used to produce three-dimensional elements or products. In production, produced with closed molding methods, less labor and material are used but as a result superior productivity product is obtained. Vacuum bag molding, resin transfer molding, resin film infusion, pultrusion molding, etc. can be given as examples of closed molding techniques (Akter 2021).

4.4. Applications

The main application fields of fiber-reinforced composites, which have a wide range of commercial and industrial use, are aircraft, spacecraft, automotive industry, sport equipments, marine vehicles, construction infrastructure, and medical applications. However, these materials are used in the electrical-electronics industry such as printed circuit boards, circuit breakers, cable carriers, and wind turbines, and the fuel industry such as oil platforms. It also has the potential to be used in the general engineering sectors such as gears, bearings, drives, robotic arms, loom shuttles, and spring materials. Figure 4.3. shows the market shares of the major composite sectors.

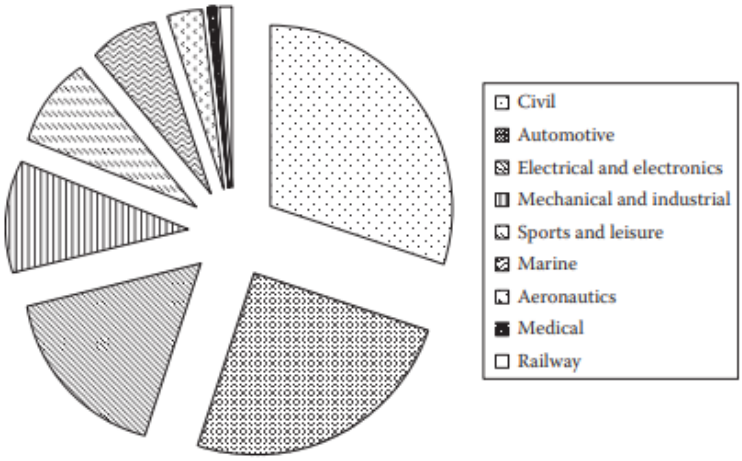

Figure 4.3. *Volume of composites used in different sectors (Balasubramanian 2014).*

Aircraft applications: Military and commercial aircraft, where weight reduction is important to achieve higher speeds and increase load carrying capacity, are the main areas of use of fiber-reinforced composites. Some of the aircraft components in which composites are used are wing leading edge panels, nose wheel doors, engine guard elements, elevators, nose cowls, tail cones, horizontal stabilizers, and vertical stabilizers.

Sport material applications: The advantages of using fiber-reinforced polymers in sports equipment; lightweight, vibration damping, and flexible design. Weight reduction has been achieved by the use of carbon fiber reinforced epoxies instead of metallic materials, providing higher speeds and faster maneuverability in racing sports such as bicycle and canoe races.

Building and construction applications: Fiber-reinforced composites have a high potential to replace reinforced concrete and steel materials in construction applications such as bridges and buildings. In addition, carbon fiber reinforced composites are used in the column reinforcement applies of the construction industry. It is aimed to strengthen the columns confinement with composite material and the columns that may be damaged during an earthquake due to the ductile behavior of carbon-based composites. The main motivation for the use of composites in these areas is their wear resistance, which allows for long-lasting and lowers maintenance and repair costs.

Medical applications: The most common usage areas of composite materials in medical applications are bone treatment (internal fixator plate) and prosthetic applications. As an alternative to traditional materials such as stainless steel and titanium alloys, materials with lower rigidity such as carbon fiber/epoxy, glass fiber/epoxy, carbon fiber/polyethylene, carbon fiber/polypropylene composites can be used. Other applications where composites are used are bone fillings, dental implants, medical equipment such as catheters, and walking aids (Var 2021).

5. INSTRUMENTAL METHODS USED IN THE CHARACTERIZATION OF THE PRODUCTS OBTAINED

In the examination of the products obtained after the applied heat treatments, Thermogravimetric Analysis (TGA), Fourier Transform Infrared Spectroscopy (FT-IR), Scanning electron microscopy (SEM), and BET surface analysis are among the instrumental and analytical methods.

5.1. Fourier Transform Infrared Spectroscopy (FT-IR)

Infrared (IR) spectroscopy is one of the spectroscopic methods developed based on the interaction between light rays and the analyzed chemical molecule (Yağmur et al. 2003).

It is an analysis method applied to determine the functional groups of products. In addition to being used for quantitative purposes, IR spectroscopy is mostly used to determine the structures of organic molecules. As can be seen in Table 5.6. the infrared region is divided into 3 regions as near-, middle-, and far-infrared regions according to the wavelengths, wavenumbers, and applications (Middle East Technical University 2021; Kılıç and Karahan 2010).

Table 5.1. *The wavelengths, wavenumbers, and usage areas of the beams used in the infrared region.*

Designation	Wavelength	Wave numbers of Spectrum	Application Areas
Near-	0.78-2.5	13000-4000	Industrials, Process control, Quantitative analysis
Middle-	2.5-50.0	4000-200	Structural determination, Qualitative organic analysis, Quantitative analysis
Far-	50-1000.0	200-10	Pure inorganic and metal-organic substrate qualitative analysis

Beams are given by wavenumbers rather than wavelengths. Generally, beams with wavenumbers between 4000 and 400 cm^{-1} are used in the infrared spectroscopy analysis (Middle East Technical University 2021; Kılıç and Karahan 2010).

There are two types of spectrophotometers as shown in Figure 5.1 single-beam spectrophotometer and double beam spectrophotometer, which can be used for measuring the transmission in low absorbing materials (Jena et al. 2015).

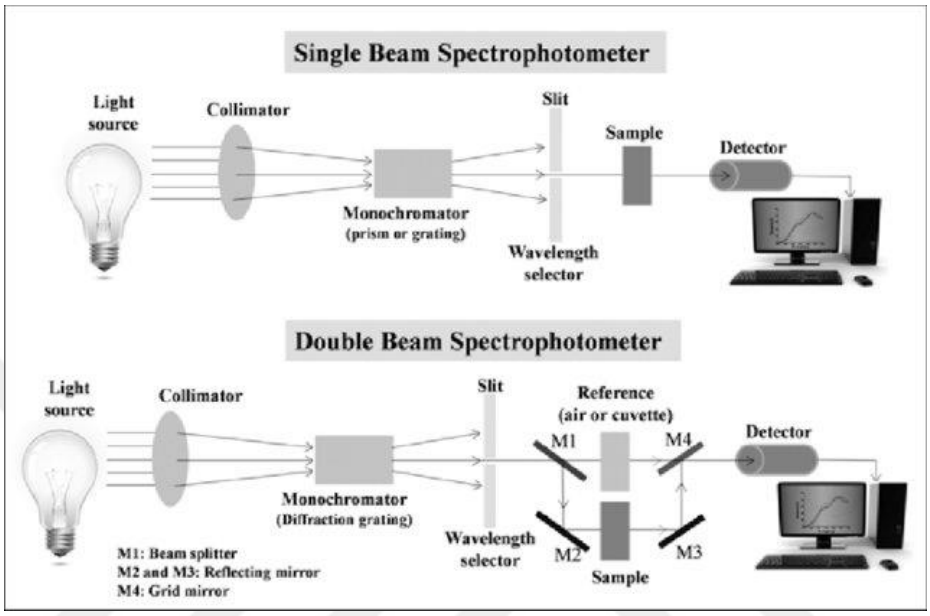

Figure 5.1. *Schematic of Single and double beam spectrophotometer (Jena et al. 2015).*

In Fourier Transform Infrared absorption spectroscopy, IR beams are absorbed by the vibrational motions of the molecule. The signal decoding process in the FTIR system is based on a mathematical transformation called the "Fourier transform" and works with the Michelson interferometer. In mathematical Fourier transform spectroscopy, the radiant intensity is taken as a function of time. Fast and high-resolution spectra can be obtained without the need to scan each wavelength separately. Infrared spectra of solid, liquid, and gas samples can be recorded with this system (Bell 2012; Skoog et al. 2007).

5.2. Thermogravimetric Analysis (TGA)

All of the methods used to examine the changes in the properties of a substance or its derivatives under a certain temperature program to measure the heat absorbed or heat released in the reaction are called thermal analysis methods (Varol 2007).

Thermogravimetric analysis is one of the thermal analysis methods in which changes in physical and chemical properties of materials. The basis of the

thermogravimetric analysis is examined of the mass change due to temperature change (Indian Institute of Technology Kanpur 2022). In this method, the decrease in the mass of the substance to be analyzed as a result of the programmatically increased or decreased temperature is examined as a function of temperature or time. The resulting temperature mass curves are called thermograms or thermal decomposition curves. Mass losses as consequences of temperature increase are generally caused by the separation of volatile compounds such as water from the structure or the decomposition of the substance (Varol 2007).

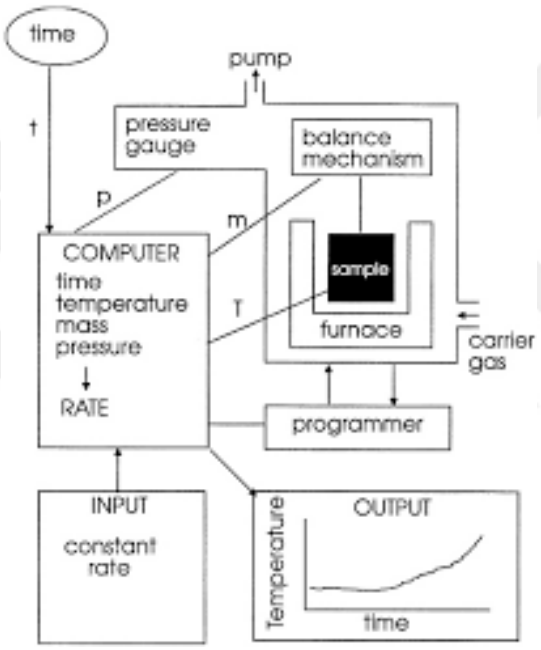

Figure 5.2. *Schematic diagram of the TGA (Indian Institute of Technology Kanpur 2022).*

Thermogravimetric analyzer comprises a closed system that includes an assay balance, a well-isolated oven, a system that automatically records the mass and temperature change, and an inert gas system as nitrogen or argon to prevent burning the sample when oxygen contact with the sample, as given in Figure 5.2. above. After the analysis, information about physical phenomena, for example second-order phase transitions, including vaporization, sublimation, adsorption, absorption, and desorption, and chemical phenomena such as chemisorption, decomposition, and solid-gas reactions are obtained from TGA (Indian Institute of Technology Kanpur 2022).

5.3. Brunauer-Emmett-Teller Surface Area Analysis (BET)

An isotherm equation applicable to multilayer adsorption was first developed by Stephan Brunauer, Paul Emmett, and Edward Teller in 1938. It is possible to determine the specific surface area of a porous solid by the BET isotherm (Hwang and Barron 2011). The starting point of the BET isotherm is based on three main assumptions. The first one is that before the adsorbent surface is covered by a monomolecular layer, several multi-molecular layers are formed. The second one is that when the adsorbent equilibrium occurs, the equilibrium condition occurs for each layer. And the last one is that subsequent layers are considered equal to the latent heat of condensation of the adsorbed if the adsorption heat of the first layer, E1, is constant and the heat of adsorption E2 of the second (Yurtay 2020).

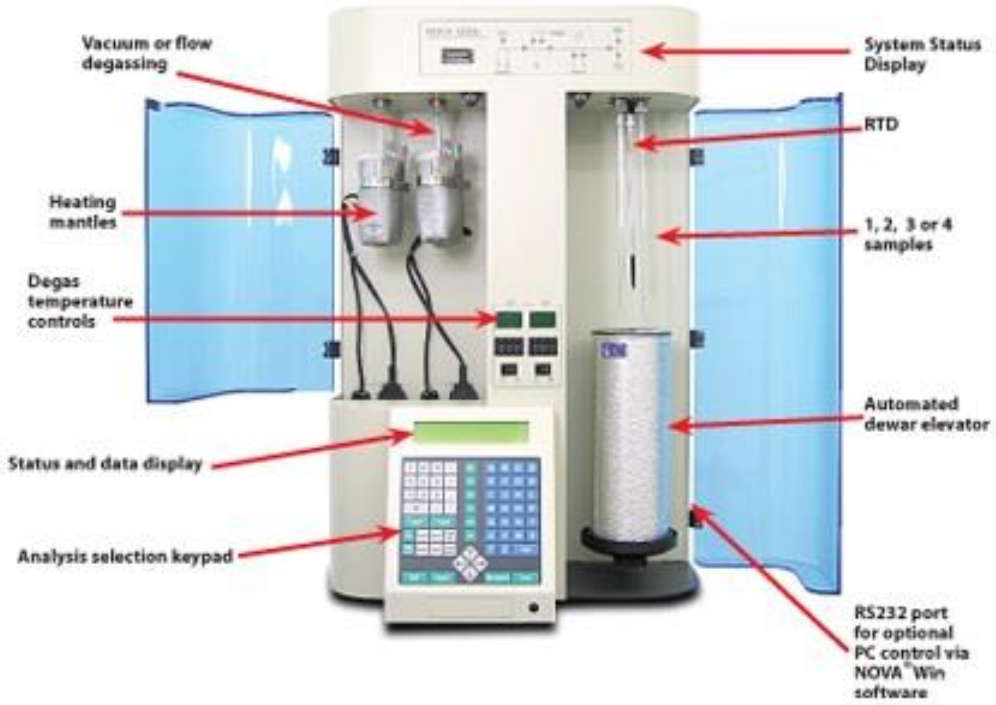

Image 5.1. *Structure of BET Analyzer (Zhuang and Lee 2015).*

The BET analyzer includes the three main parts: degas and sample chambers, system status display, and the control unit; all shown in Figure 5.3.

Adsorption means adherence with surface and atoms or molecules of gas with each other. While the amount of gas adsorbed primarily depends on the exposed surface, it also depends on temperature, gas pressure, and the strength of the interaction between gas and

solid. In BET surface area analysis, nitrogen is usually used because of its high purity, lightweight characteristic, which can easily fill the pores of the substance. The surface is cooled using liquid nitrogen to obtain a detectable amount of adsorption because the interaction between the solid phases and the gaseous is usually weak. The volume of gas adsorbed on the surface of the particles is measured at the boiling point of nitrogen (-196°C) because this temperature is below the critical temperature of nitrogen gas and above at this degree, the gas condenses on the particles' surface.

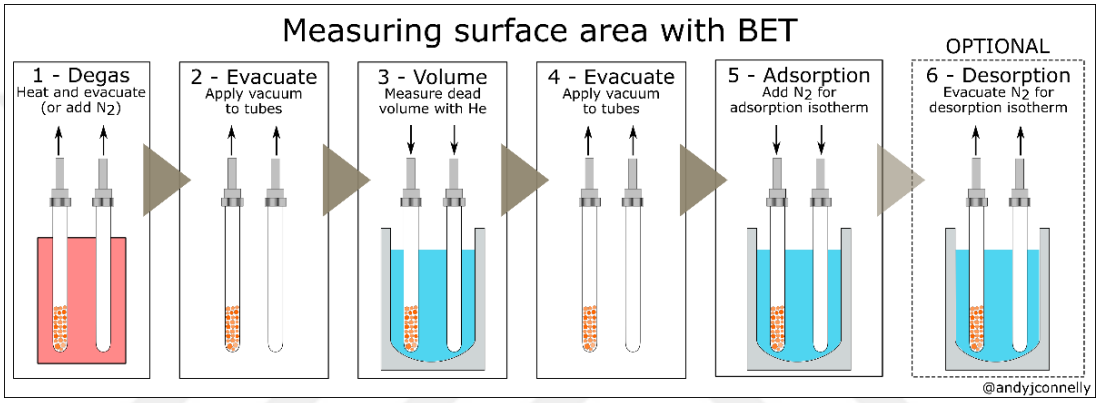

Figure 5.3. *Measuring surface area with BET (Connelly 2017).*

The measuring surface area of substrate consists of six steps, as shown in Figure 5.4 and detailed as follows:

1. In the first step, the sample must be degassed. This step consists of the sample under a vacuum or gas-washing (e.g., nitrogen) at a high temperature. The temperature used in the degas step depends on the stability of the sample, and a temperature of 110°C is preferred for the stability of the sample against this temperature. After the process is finished and the sample is cooled, the sample must be weighted again to calculate mass loss during degassing.
2. The sample and reference tubes are evacuated for measurement.
3. The third step is about for volume of the sample. To correct the amount of adsorbate absorbed, the dead volume measurement uses a gas such as helium. The sample and the reference tube's dead volume should be similar.
4. The dead volume is removed from the tubes by vacuum.

5. Later, adsorbent gas starts to fill the tube, and the gas is adsorbed on the sample. The pressure in the confined volume continues to decrease until the system reaches equilibrium. Thereby, there is a difference between the initial and the equilibrium adsorption amount. By calculating the adsorption difference, a chosen P/P0 adsorption isotherm is obtained.
6. Desorption step can be also applied in BET. This step is the opposite of Step 5. The vacuum is applied to the sample to obtain the desorption isotherm (Connelly 2017).

5.4. Scanning Electron Microscopy Analysis (SEM)

A scanning electron microscope, also abbreviated as SEM, is an instrument that produces a widely magnified image by using electrons instead of light to form an image. Scanning Electron Microscope is widely used in research and development studies in many branches such as quality control, biological sciences, material science, and failure analysis (Perdue University 2022; Kılıç 2009).

In the scanning electron microscope, the image is formed by receiving the signals produced from the interaction of the electron beam and the surface of the sample being examined. These interactions are divided into elastic and inelastic interactions. Various signals produced by electron beam sample interaction within the scanning electron microscope and the regions where the signals can be detected are shown in Figure 5.5-b.

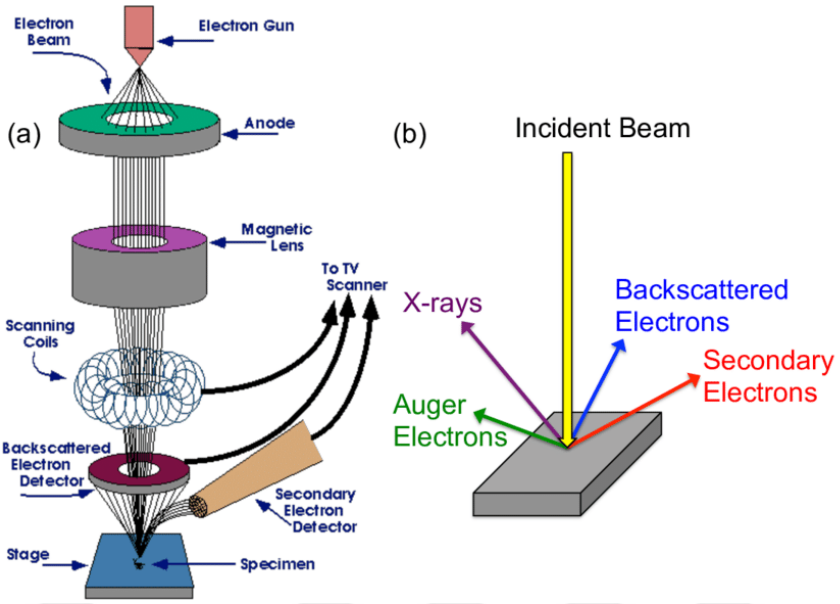

Figure 5.4. *Schematic drawing of (a) the Scanning Electron Microscope (SEM), and (b) sample-beam interactions within an SEM (Walock 2012).*

For example, the electrons that emerge as a result of the inelastic collision of the electrons in the incident beam with the atoms in the material are secondary electrons. Secondary electrons are collected using the photomultiplier tube. Another electron group that emerges during the interaction between the electron beam and the material on the sample surface is called backscatter electrons. Backscattering electrons are higher-energy electrons from deep regions of the surface (Perdue University 2022; Yurtay 2019).

Internal parts of the electron microscope include an electron gun, anodes, a condenser lens, an objective lens, an x-ray, and secondary defectors, as shown in Figure 5.5-a. The working principle of the SEM is realized by following the steps given below.

- Firstly, the electron sources generate electrons from the top of the microscope column.
- Then, an anode plate attracts the electrons and which are sent to the magnetic lens. The magnetic lens includes the condenser and the objective lenses.
- The condenser lens controlles the size of the beam and determines the electron amount in the beam. The size of the beam is defined by the resolution of the image.

- The other lens is the objective lens. It is the last lens in the sequence of lenses. As the lens gets closeser to the sample, its focus on the beam converges to a very small spot on the sample.
- The obtained current is passed through the coil, and as a result,magnetic field occurs. Electrons are highly sensitive to these magnetic fields, thus allowing the lenses in the microscope to control them.
- The electrons interact with atoms on the surface of the sample. This interaction generates signals in the form of secondary electrons, backscattered electrons, and rays.
- Finally, detectors at the bottom of the microscope pick up these signals and occur at high-resolution images that are displayed on a computer screen (Scimed 2022; Kılıç 2009; Walock 2012).

6. EXPERIMENTAL TECHNIQUES

This section gives information about the experimental processes applied for both the raw material used and the product obtained in this thesis study. In this section, the characterization studies of the product with the optimum parameter obtained by the determination of the properties of the raw material and the carefully selected parametric experimental studies based on these properties are explained.

6.1. Properties of the Raw Material

In this study, cotton denim waste was used as the raw material belonging to different brands which are now on sale in Turkey. The products were obtained from recycled denim cloths.

6.1.1. Bulk density determination

To determine the bulk density, the raw material is encapsulated in a closed cup whose volume and weight are known, without being compressed. Then, the closed cup is weighted with the sample. Bulk density is calculated by the equation given below (ASTM E 873-83).

$$\text{Bulk density (g/cm}^3) = \frac{g_2 - g_2}{V} \quad (6.1)$$

where

g_1 = The empty weight of the closed cup, g,

g_2 = The total weight of the sample and closed cup, g, and

V = The volume of the closed cup, cm^3.

6.1.2. Moisture content determination

An amount of the sample prepared before for analysis is stored in a watch glass with a sensitivity of 0.2% and kept in an oven set to 103±2°C. It is kept at this temperature for 2 more hours until the difference between the two weights are equalized, and this process is repeated. The moisture content is calculated from the following equation, as a percentage of weight (ASTM D 2016-74)

$$\text{Moisture Content (\%)} = \frac{g_1 - g_2}{g_2} \times 100 \quad (6.2)$$

where

g_1 = Initial weight of the sample, g, and

g_2 = Weight of the sample after drying in the oven, g.

6.1.3. Volatile matter determination

1.0 g of air-dried sample is placed into the crucible that has constant weight with an accuracy of 0.1 mg, which is placed in a furnace at 950±20 °C for 7 mins after covering the crucible. Caution should be taken not to burn the sample. The crucible removed from the oven is cooled in a desiccator and weighed. The amount of volatile matter in the sample is calculated from the following equation (ASTM E 872-82).

$$\text{Volatile Matter Content (\%)} \left(\frac{g_1 - g_2}{g_2} - M\right) \times 100 \qquad (6.3)$$

where

g_1 = Weight of the sample, g,

g_2 = Weight of the sample after drying in the oven, g, and

M = Moisture content of the sample.

6.1.4. Ash content determination

From the raw material, ~2 g is weighed and placed in a crucible with a constant weight after being covered. The sample is dried in an oven set to 100–105 °C. After one hour, the crucible is taken out of the oven, covered with its lid, cooled in a desiccator, and weighed. This process is continued until the difference between the two weights reaches 0.1 mg and the dry sample weight in the oven is found. The raw material in the crucible is burned with the lid of the crucible open, in an oven at a temperature of approximately 600°C until all carbon is removed. The heating process should be slow, and the burned sample should not catch fire. After the combustion process, the crucible, which is removed from the furnace, is cooled in the desiccator with the lid covered. This process is repeated once in every half an hour until the difference between the two weighing reaches 0.2 mg. The ash content is calculated as a percentage of the weight using the following equation (ASTM D 1102-84)

$$\text{Ash Content (\%)} = \frac{g_1}{g_2} \times 100 \qquad (6.4)$$

where

g_1 = Weight of the ash, g, and

g_2 = Weight of the dry sample in the furnace, g.

6.1.5. Fixed carbon content

The fixed carbon content is a calculated value. This value is determined by subtracting the total of percent moisture, ash, and volatile matter from one hundred. The percentage value is calculated as apercentage of the weight using the equation given below (ASTM E 870-82).

Fixed Carbon Content (%) = 100 – (Moisture % + Ash % + Volatile Matter %) (6.5)

6.1.6. Elemental analysis

Elemental analysis, which is applied to determine the carbon, hydrogen, nitrogen, and oxygen contents of the raw material, was analyzed in the Carlo Erba EA 1108 device at Eskişehir Technical University's Engineering Faculty Chemical Engineering Department Research Laboratory according to the ASTM D5373.

The higher heating value (HHV) of the substances whose carbon, hydrogen, oxygen, and sulfur contents were determined, were calculated using the Dulong Equation as given below.

$$\text{HHV (kJ/kg)} = 338.2 + 1442.8\left(H - \frac{O}{8}\right) + 94.2S \quad (6.6)$$

In the equation above, C, H, O, and S are expressed as the weight percentages of carbon, hydrogen, oxygen, and sulfur, respectively, on a dry and ashless basis.

6.1.7. Thermogravimetric analysis of the raw material

In order to examine the thermal degradation of raw materials, the STERAM LABSYS Evo device, which is in the Chemical Engineering Research Laboratory at Eskişehir Technical University, was used for thermogravimetric analysis. Samples weighing between 5-15 mg are placed in the crucible and heated at a heating rate of 10°C/min from room temperature to 1000°C in a nitrogen flow of 100 cm^3/min. The thermal behaviors of the raw materials were investigated with the mass loss (TG), heat flow (DSC), and mass loss derivative (DTG) curves were obtained as a result of the analysis. The mass loss is calculated by the equation given below.

$$\frac{dW}{dt} = -\frac{1}{W_0}\left(\frac{dW_t}{dt}\right) \quad (6.7)$$

where

t = time, min,

W_0 = Initial weight of the sample used, g, and

W_t = Weight of the sample at time t, g.

6.1.8. Fourier transform infrared spectroscopic analysis of the raw material

Thermo Scientific-Smart OMNI Transmission FT-IR device in Eskişehir Technical University Chemical Engineering Research Laboratories was used to determine the functional groups contained in the raw material. For the analysis of each sample, pre-dried 99% potassium bromide and 1% sample are mixed homogeneously and ground in the mortar until they turned into a good powder. A lower concentration should be required in the obtained sample (Beer's Law). Too high concentration often causes difficulties in obtaining clear pellets. The IR beam is completely absorbed or scattered from the sample, resulting in very noisy spectra. Finally, the prepared pellets are placed in the chamber of the device, and FTIR spectrum is obtained at 400-4000 cm^{-1} wavelengths.

6.1.9. Scanning electron microscopy analysis of the raw material

SEM (scanning electron microscope) images were taken with the ZEISS Supra 40 VP model device in Bilecik Şeyh Edebali University Central Laboratory to observe the raw material's fiber structure. For SEM images, samples were placed on holders with carbon tape and coated with gold-palladium with the coating device operating under vacuum.

6.2. Physical and Chemical Activation of the Cotton Denim Cloths

Activated carbon fabric production consists of two different application steps as carbonization and chemical activation processes. Carbon cloth experiments were carried out in a fixed bed reactor shown in Figure 6.1. in Eskişehir Technical University Chemical Engineering Carbon Materials Research Laboratory. The reactor is designed from stainless steel and around the reactor exists a furnace that has asbestos insulated with 2000 W heating.

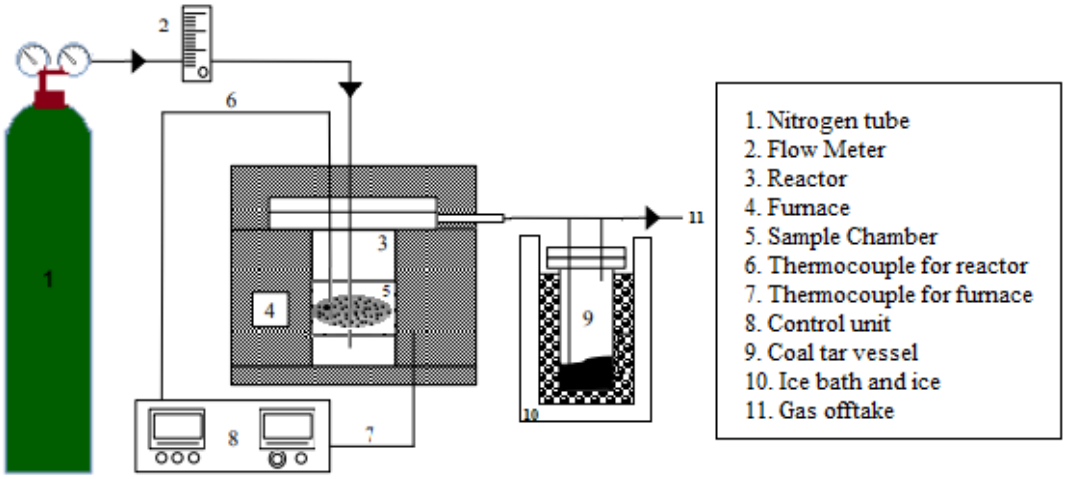

Figure 6.1. *Fixed Bed, Nitrogen Flow Experiment Equipment Schematic Design*.

In Physical activation, namely the carbonization process, cotton cloth non-impregnated with chemical activation agent were carbonized at 600 °C under nitrogen (N_2) flow of 100 cm^3/min and at a heating rate of 10 °C/min in a stainless-steel fixed bed reactor. Firstly, denim fabrics that have been pre-sized and cleaned are placed in the reactor. The process is started with the selected operating temperature depending on the thermal gravimetric analysis of the raw material. only 600 °C temperature applied for carbonization. The sample is heated up to 600 °C at a heating rate of 10 °C/min for 60 minutes and kept at this temperature for 30 minutes. When the process is finished, the device automatically shuts itself down and waits for it to reach room temperature to open the reactor. Temperature control is monitored from the controller on the device. As a result, carbon fabric is obtained.

For the chemical activation of carbon fabric, three different parameters were used in production. The first parameter is the chemical activation agent. The samples are impregnated with six different chemical activation agents such as potassium hydroxide (KOH), potassium carbonate (K_2CO_3), zinc chloride ($ZnCl_2$), ammonium chloride (NH_4Cl), sodium chloride (NaCl), and phosphoric acid (H_3PO_4). The second parameter is the impregnation ratio. The ratio of chemical activating agent to carbon fabric was determined as 2:1, 3:2, 1:1, and 1:2 by mass. Reactor application temperature, which is the third and last parameter, was determined based on the TG curve of carbonized carbon fabric as 600, 700, and 800 °C.

Carbon cloth that was impregnated with different ratios has been awaited overnight in a dark place. Then it was dried by turning it inside out every half hour in a 75°C drying oven. When the samples were dried, they were placed in the reactor under pre-determined conditions. The stainless-steel fixed bed reactor operates under a nitrogen (N2) flow of 100 cm3/min and a heating rate of 10 °C/min. The sample is heated up to the determined temperatures at a heating rate of 10 °C/min and kept at these temperatures for 30 minutes. When the reactor temperature decreases to room temperature, it is opened and the activated carbon cloth is obtained. Lastly, the activated carbon cloths obtained were washed with distilled hot water until the pH level was neutralized. Afterwards, they were dried at 105°C in the drying oven.

The efficiency of the activated carbon cloth is calculated with the equation given below:

$$\text{Efficiency } \% = \frac{W_i - W_{ACC}}{Wi} \times 100 \tag{6.8}$$

where

W_i = Weight of carbon fabric fed into the reactor, g, and

W_{ACC} = Weight of the activated carbon cloth after being washed, g.

6.3. Characterization of the Activated Carbon Cloths

The characterization applications of activated carbon fabrics obtained from a fixed bed reactor under nitrogen during the production phase are described in this section.

6.3.1. Moisture content determination

Each product contains a specific amount of humidity. Precisa XM 60 Moisture analyzer in Eskişehir Technical University Chemical Engineering Research Laboratories was used to identify the humidity inside the activated carbon cloth. The analyzer has a UV lamp which balances function on the gravimetric or loss on drying (LOD) principle. A halogen moisture analyzer balance uses the heat from the halogen lamp to dry the sample. Moisture content is calculated based on the weight difference before and after the drying procedure. 150°C heat treatment is applied according to ASTM D2867 standard for moisture content determination of activated carbon samples.

6.3.2. True density determination

The true (skeletal) density values of the activated carbon cloths were determined by using Quantachrome brand UltraFoam 1200e model automatic Helium pycnometer.

A Helium pycnometer aims to find the volume and true density based on Archimedes' principle. Helium gas is the preferred displacement fluid because of its small dimensions and ideal gas behavior. True densities are obtained when the helium penetrates through open pores and there are no closed or inaccessible pores left in the material. For the analysis, a few small pieces of activated carbon cloth are taken to the chamber of the device and then covered with its cover. True density is determined according to the amount of Helium gas consumed per unit mass. Consequently, the real density is calculated with the mass to volume ratio automatically by the device.

6.3.3. Elemental analysis

The amounts of carbon, hydrogen, nitrogen and oxygen contained in carbon cloths were determined with the LECO CHN628 elemental analyzer that measures according to the ASTM D5373 standard.

6.3.4. TG analysis

The thermal gravimetric curves of activated carbon cloths with both physical and chemical activation applied by impregnating with different chemical activation agents were obtained with the SETERAM LABSYS Evo device in Eskişehir Technical University Chemical Engineering Carbon Materials Research Laboratory.First, 10 mg of small pieces cut from activated carbon fabric are taken into a crucible, and later, the crucible is placed in the device. It is heated from 25°C to 1000°C under operating conditions at a nitrogen flow of 100 cm^3/min and a heating rate of 10°C /min. The thermal behavior of carbon fabrics is determined by the mass loss curve (TG). The dTG curve is then obtained for thermal phase transitions by taking the first derivatives of the TG curve.

6.3.5. FTIR analysis

To determine the functional groups of the activated carbon cloths produced from cotton denim, a Thermo Scientific-Smart OMNI Transmission FT-IR device in Eskişehir Technical University Chemical Engineering Research Laboratories was used.For each analysis, 99% KBr (dehumidified) and 1% samples were mixed homogeneously and ground in a mortar until they became a fine powder. The prepared powder mixture was

then turned into pellets under pressure, which were placed in the sample chamber of the device and the spectrum was taken in the wavelength range of 400-4000 cm^{-1}.

6.3.6. Brunauer-Emmett-Teller surface area analysis

The structural characterizations of the produced carbon cloths, their macro, meso, and micro pore distributions they contain, and surface area were determined by nitrogen adsorption-desorption isotherms at 77 K using BET Analyzer. Analyzes were carried out using the Quantachrome brand Autosorb 1-A device in the Research Laboratory of the Department of Chemical Engineering of Eskisehir Technical University. In the first stage, nightlong degas were applied by outgassing the samples weighing approximately 50 mg. After the degas process for analyzing nitrogen adsorption-desorption, the isotherms of the samples were retrieved at 77 K. The surface areas of the samples were calculated with the BET equation. When P/P_o, the relative pressure value was 0.995, the total pore volume was found, and information about micropore areas was obtained by using V-t curves.

6.3.7. SEM analysis

ZEISS Supra 40 VP model device, located in the central laboratory of Bilecik Şeyh Edebali University, was used to examine the fiber structures of activated carbon fabrics, and to explain the structure and the porous dense texture formed. Before the images were taken, the samples were placed on the double side carbon tape and coated with gold and palladium under pressure. Afterwards, images taken in 100, 500, 1000, 2000 and 4000X sizes.

6.4. Reinforced Activated Carbon Cloth Obtained Using Two-Phase Resin-Reinforcement Studies

In this thesis, studies to strengthen the tensile properties of activated carbon cloths by adding resin were investigated. Reinforced activated carbon cloth obtained by two-phase resin reinforcement studies were carried out in Eskişehir Technical University Chemical Engineering Research Laboratory, with 2-phase resins obtained from Civil Engineering Research Laboratory.

Master Brace SAT4500 resin with two-component, solvent-free, high-strength epoxy resin (Component A) and an epoxy hardener (Component B) containing a special epoxy-based adhesive was used to manufacture the composite material. Before starting to mix Component A (3.73 kg) and Component B (1.27) to form the resin, Component A

weighed into a flask and should be mixed for 30 seconds and then Component B should be added at a ratio of 3:1 g/g by mass until a homogeneous mixture has been obtained.

The resulting resin-based mixture was confined without waiting for it to freeze, by absorbing it into the fabric so that a thickness of 0.8-1 mm was obtained between the silicone pad, such that there was no gap on the surface.

Confined samples were kept in a dark environment at room temperature to ensure the product is frozen. Tensile strength analysis was applied to the obtained high strength, lightweight, and flexible resin reinforced composite materials at a test speed of 2 mm/min in accordance with ISO 527-2/X/Y and ASTM 638 standards.

6.4.1. Tensile strength analysis

The tensile test of the obtained carbon cloth composites was carried out with the INSTRON brand tensile, fatigue and compression device in the Eskişehir Technical University Materials Science and Engineering department research laboratory.

A test composite is prepared according to the dimensions given in Figure 6.2. For analysis, the composite was attached to the measuring chamber of the instrument. A tensile strength was applied on composite material at a test speed of 2 mm/min according to the ASTM D3039/D3039M standard.

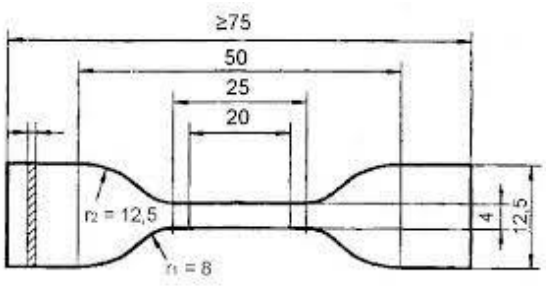

Figure 6.2. *The sample size for the tensile test.*

6.5. Strengthening-Repair of Structural Members Studies

The carbon cloths produced are used in structural engineering, which is a sub-field of Civil Engineering, in the strengthening-repair processes for structurally damaged or inadequate elements against earthquakes. This process can be summarized as follows:

First of all, concrete molds with different strength ratios were poured to carry out the experiments. After the casting molds are set up and ready for the experiment, the parts to be wrapped with the produced carbon cloth are ground and the pointed corners are rounded to a certain radius. This is an important step to prevent sharp corners from damaging the cloth under dynamic loads. Reference material is used pure concrete that includes different ratios of cement, water, and different size of aggregate. The determination of the ratio of reference material is calculated with the ratio of water to cement. Optimum concrete strength is will be used in experimental studies as determined result of the strength test. It is very important that all test elements show the same characteristics in order to better examine the results of the reinforcements. In order to prevent possible differences such as environmental conditions changes, concrete mix, and workmanship defects that may occur during the production of the test elements, all test elements were poured at once.

Then, lining materials are applied like a roll with a brush on the surfaces of the optimum concrete to be confined with carbon cloth. Epoxy-based repair mortar is used to increase the adhesion between the concrete surface and the cloth. Afterwards, the fabric strips cut in specified sizes and numbers are applied together with the epoxy material that ensures the hardening and strength of the strips before the repair mortar hardens. Reference material is used pure concrete that includes different ratios of cement, water, and different size of aggregate. The determination of the ratio of reference material is calculated with the ratio of water to cement .

All the repairing and strengthening experiments, which were applied to test the reinforcement of concrete structures, were carried out in Eskişehir Technical University Civil Engineering Research Laboratories. Strength tests were carried out using FORE A.Ş. PROFI XS Concrete Pressure Test Press.

7. EXPERIMENTAL RESULTS

In this study, activated carbon cloth has been obtained by using cotton denim wastes. Three different parameters were used in production as chemical activation agent (KOH, K_2CO_3, $ZnCl_2$, NH_4Cl, NaCl, H_3PO_4), temperature (600, 700, 800˚C), and chemical agent ratio (2:1; 3:2; 1:1; 1:2 w/w). The activated carbon, and carbon cloths obtained from the activation, and carbonization process were characterized and studies were carried out on the strengthening of construction structures by confining with the cloths and resin.

In this section, the properties and characterizations of the raw material, the properties and optimum conditions of active carbon cloths produced at different impregnation ratios and different temperatures, the production of composite materials, and the application studies of this material on structural strengthening are given respectively.

7.1. Experimental Results for Precursor as Cotton Denim Wastes

Precursor analyses are very important steps to determine the process algorithm for activated carbon and carbon cloth productions. The pre-washed and dried denim clothes were sized as 6x3 and 3x3 cm^2 for pre-production, each production consists of 3 pieces. All the analyzes were repeated at least twice. The results given are calculated as the average of the experiments performed.

7.1.1. Properties

Table 7.1. and Table 7.2. give the information of proximate and ultimate analyses of the 100% cotton denim waste as a precursor.

Table 7.1. *Proximate analysis (wt.%) of cotton denim (as received).*

Analysis	wt. % (as received)
Moisture	5.03
Ash	0.98
Volatiles	90.1
Fixed Carbon*	3.89

* Calculated by the difference

The ash content of precursor is a central factor for elements or compounds in organic and inorganic matter. Ash chemical composition is an important parameter to consider during thermochemical conversion. It gives information about slag formation at higher temperatures in the production. Volatile matter content gives information about the efficiency and carbon content of the produced activated carbon cloths. It is expected that the production of activated carbon cloth, which will be made with cotton precursor with a high volatile substance content of 90%, will contain a high percentage of carbon elements and the product will be formed with a high yield.

Table 7.2. *Ultimate analysis (wt.%) of cotton denim (as received).*

Analysis	**wt. % (as received)**
C	41.63
H	5.35
N	1.98
O*	51.04
Atomic Ratio	
H/C	1.15
O/C	0.92
N/C	0.04
Molar expression	$CH_{1.53}N_{0.04}O_{0,92}$
Theoretical HHV (MJ/kg)	12.59

* Calculated by the difference

When Table 7.1. and Table 7.2. are examined, we can say that a high carbon ratio and low ash content of cotton denim indicate that the precursor is suitable for carbon materials production. The oxygen and fixed carbon contents in these tables are calculated by the difference.

7.1.2. FTIR analysis

In the FT-IR spectrum, the absorption regions are divided into two groups the group frequencies region (4000-2500 cm^{-1}), and the fingerprint region (1400-4000 cm^{-1}). FT-IR spectrum of cotton denim fabric is given in Figure 7.1.

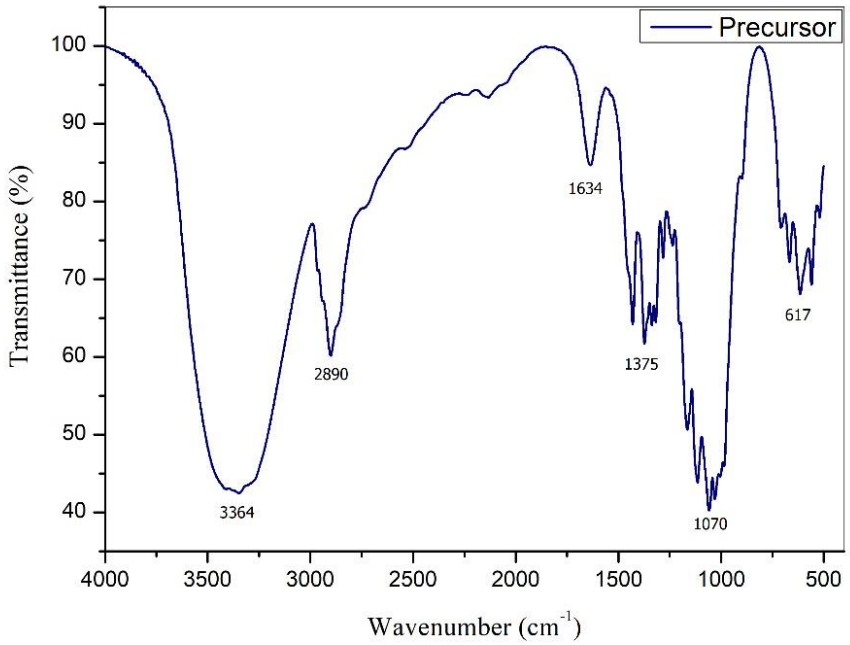

Figure 7.1. *FTIR spectra of cotton denim fabric.*

When the FT-IR spectra are examined, cellulose-based precursor showed characteristic absorption peaks due to -OH, C-H (sp^3), and -C-O stretching vibrations in cellulose at 3364, 2850, and 1070 cm^{-1}, respectively.

Table 7.3. *Spectrum datas for cotton denim fabric.*

	Nr of signal	Experimental frequency (cm^{-1})	Literature frequency (cm^{1})	Bond	Band
1.	**Stretching**	3364	3200-2500	C-H from CHO	Broad
2.	**Stretching**	2890	1684-1674	C-H from CHC	Sharp and intense
3.	**Stretching**	1634	1775-1600	C=O from aromatic	Sharp and intense
4.	**Stretching**	1375	1325-1215	C-H from C-H-C	Short and intense
5.	**Stretching**	1070	1225-1175	C-O from C-O-C	Sharp and intense
6.	**Stretching**	617	Printfinger region	-	-

On the other hand, the IR spectrum shows the absorption peaks occurring in 1400-1300 cm-1 expressing the -CH vibration in cellulosic structure. The broad -OH band observed in the range of 3700-3000 cm^{-1} indicates the presence of water content and α-cellulose in the raw material. It has been determined that the intense peaks seen around 1775-1600 cm-1 are caused by C=O vibrations found in aromatic structures and belonging to hemicellulose.

7.1.3. Thermal gravimetric analysis

Thermal decomposition of the precursor was examined by applying thermogravimetric analysis. The thermal decomposition of raw materials depends on their chemical composition and structure. It is a differential feature of every precursor. When the thermal degradation is examined, the curve can generally be categorized into 3 main sections. In the first part, mass loss occurs range at 50-150 ˚C due to moisture content in the precursor removes. Then following the second part of the curves, the sharpest peak occurs in this section at different temperature ranges for each material depending on the precursor. This means that it is the part with the highest mass loss. Generally, the second part starts at 200 ˚C and, ends about the range at 400-500˚C. After these temperatures, the mass loss decreases by time and reaches stabilize in the last part.

In other words, the thermal degradation curve gives information about the decomposition of hemicellulose, cellulose, and lignin contained in the precursor. Hemicellulose decomposes range at 250-350 ˚C, cellulose degrades range of 325-400 ˚C, and lignin decomposes at higher temperatures (Yang et al. 2007; Varol 2007).

Figure 7.2. shows thermal gravimetric and derivative thermal gravimetric curves of precursor. The peak that appears at about 80 ˚C refers to the moisture loss in the raw material. When the highest peak formed at about 350 ˚C, hemicellulose in the precursor decomposed with the highest mass loss and following with cellulose degrades with lower mass loss at 390 ˚C. There is no peak at higher temperatures because of the fact that the precursor is plant-based raw materials. In other words, the precursor does not contain a stable amount of lignin. As a result of thermogravimetric analysis, the operating range was determined at between 600-800 ˚C.

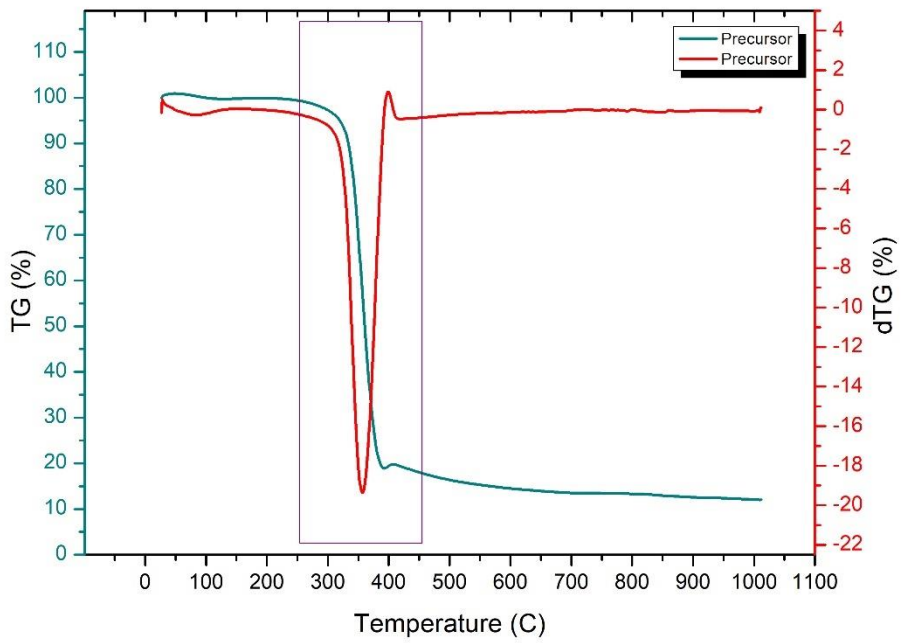

Figure 7.2. *Thermal decomposition curves of precursor.*

7.1.4. SEM analysis

The scanning electron microscopy technique was used to observe the surface physical morphology of cotton denim fabric. As shown in Figure 7.3.a and b are the magnification of 100 and 2000 SEM images of the cotton denim fabric, respectively.

It is seen that cotton denim fabric has a close texture and knitting model when considering the SEM image with 100X. The fabric surface is almost smooth and clean. The smooth surface provided a favourable condition for carbon cloth production. When the fiber is examined the fiber closer, it can be seen that the structure of the fibers that make up the cotton fabric.

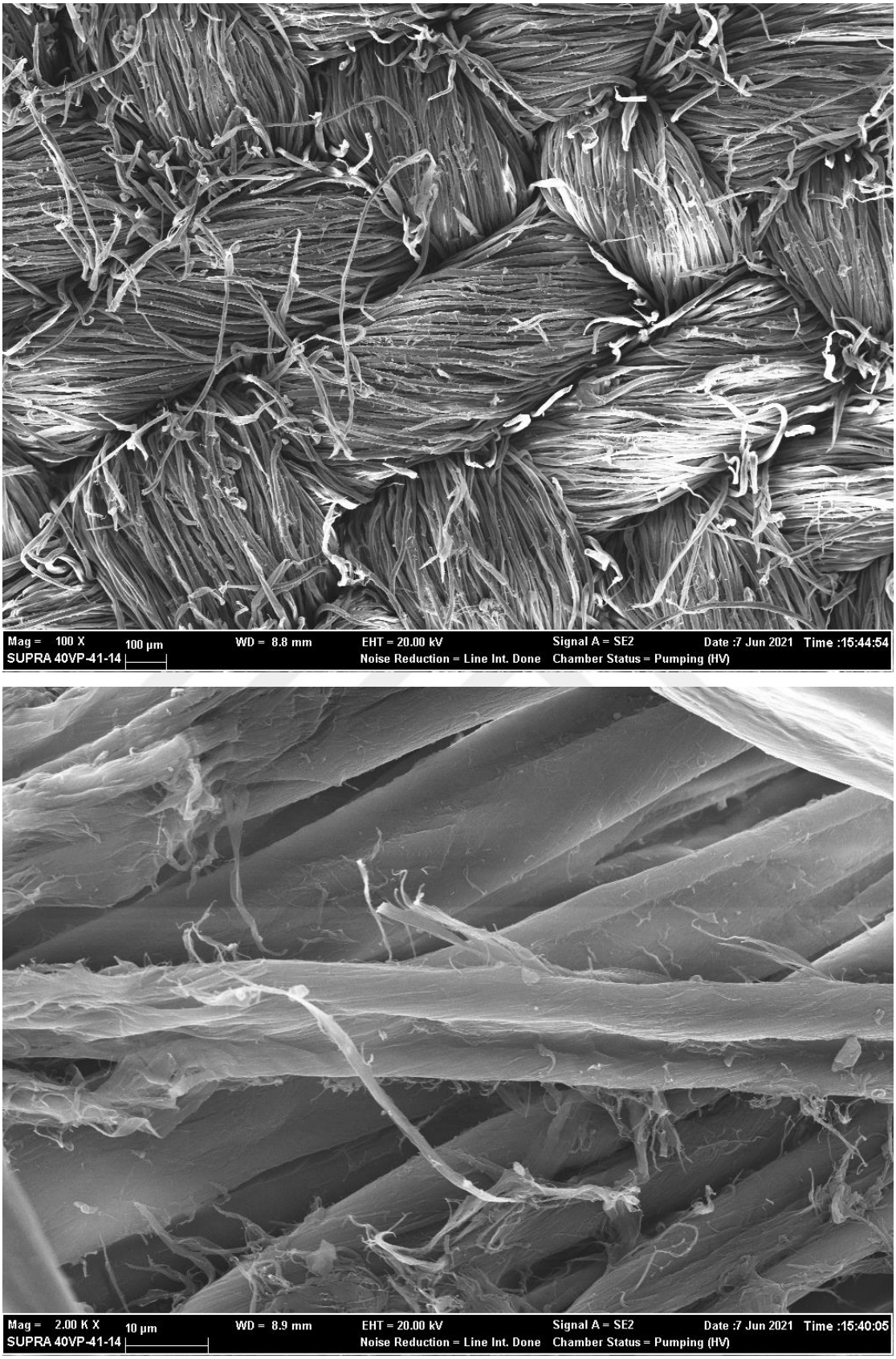

Figure 7.3. *SEM images of cotton fabric with a) 100X and b) 2KX.*

7.2. Experimental Results for Carbon Cloth

The carbonization process has been applied to form a carbon skeleton structure at a lower temperature than the activation process by removing elements such as oxygen, hydrogen. The carbonization process was applied by using nitrogen flow at 100 cm^3/min in a fixed bed reactor, at 600 ˚C, determined as a result of the thermal analysis applied to the precursor. The reaction time was 120 minutes where 60 minutes for heating and 60 minutes for carbonization at constant temperature.

The activation process has been applied to carbon cloth after the carbonization. Before the activation, carbon cloth was impregnation with different chemical activation agents (KOH, K_2CO_4, ZnCl2, NH_4Cl, NaCl, and H_3PO_4). The activation process was applied by using nitrogen flow at 100 cm^3/min in a fixed bed reactor, at 800 ˚C, determined as a result of the thermal analysis applied to the precursor. The reaction time is 140 minutes where 80 minutes for heating and 60 minutes for activation at constant temperature. Ultimate, FTIR, and surface area analysis were applied for all the obtained product.

As a result, the best chemical-activated agent was determined as zinc chloride for production. After that, parametric studies for temperature and impregnation ratio of the determined chemical activating agents were carried out on the carbon cloth.

TGA, FTIR, SEM, BET surface area, and elemental analysis have been applied to obtain optimum production conditions: temperature, chemical activating agent, and its ratio. Supplied parametric conditions table as given in Table 7.4.

In the table given in below, the produced activated carbon cloths explain in three various studies: The temperature (T), the chemical activating agent (A), and the chemical activating agent’s ratio by weight (R). In addition, carbon cloth is stated with CC, ACC is the abbreviation of activated carbon cloth.

Table 7.4. *Information about parametric studies of production.*

Name of Sample	Parametric Studies	Chemical Activation Agent	The ratio of Chemical Activation Agent (w/w)	Temperature (˚C)
CC	×	**Carbon Cloth**	×	600
ACC/KOH	*A*	**KOH**	1:1	800
ACC/K_2CO_3	*A*	**K_2CO_3**	1:1	800
ACC/NH_4Cl	*A*	**NH_4Cl**	1:1	800
ACC/NaCl	*A*	**NaCl**	1:1	800
ACC/H_3PO_4	*A*	**H_3PO_4**	1:1	800
ACC/Z800-1	*A T, R*	**$ZnCl_2$**	1:1	800
ACC/Z700	*T*	**$ZnCl_2$**	1:1	700
ACC/Z600	*T*	**$ZnCl_2$**	1:1	600
ACC/Z800-2	*R*	**$ZnCl_2$**	2:1	800
ACC/Z800-1.5	*R*	**$ZnCl_2$**	3:2	800
ACC/Z800-0.5	*R*	**$ZnCl_2$**	1:2	800

7.2.1. Impregnation with different chemical activating agents

In order to examine the effect of the chemical activating agent on the carbonized cloth's surface areas and their structure, fabrics sized for production were soaked overnight in the aqueous solutions containing different chemical activated agents with a 1:1 ratio by weight of potassium hydroxide, potassium carbonate, zinc chloride, ammonium chloride, sodium chloride, and phosphoric acid. After overnight, the soaked cloths were dried under of air at 80 ˚C in the drying oven. The impregnated cloths were reversed every half an hour until all the cloths are dry for about 4 hours. After all, steps were activated in a vertical fixed bed reactor under a flow of 100 cm^3/min of nitrogen, using a heating rate of 10 ˚C/min up to 800 ˚C. All activated samples were thoroughly washed within distillated water. Flexible, lightweight pore samples were obtained from activated carbon cloths produced using $ZnCl_2$, NH_4Cl, NaCl, and H_3PO_4. The other samples, produced by using K_2CO_3, KOH, were destroyed during activation.

The carbon cloth produced from denim fabric was destroyed when a potassium hydroxide activating agent was used. Potassium hydroxide is a prototypical strong base. The strong base has damaged fiber structure from the beginning process because of the denim fabric comprising cotton fibers. As a result, the production with KOH activating agent has already melted when it reaches a higher temperature. The chemical activating

agent, potassium carbonate, strongly influenced the structure of the carbon products. The mesopore volume of carbon denim has increased in the product instead of micropore volume, which is produced by impregnating carbon fabric with potassium carbonate. Both the void volumes of the fibers and the large pore volumes created by the K2CO3 chemical caused most of the fabric to be destroyed.

The obtained samples were characterized by using characterization tests like FTIR, SEM, TGA, and BET surface area. Hence, the enforced characterization tests have been decisive for optimum production conditions.

7.2.2. Properties

The elements that compose the materials often used as ACC precursors are carbon, hydrogen, oxygen, inorganics, and minor components such as nitrogen and sulfur. Table 7.5. shows the ultimate analysis results of impregnated samples with various chemical activating agents. As seen from Table 7.5., carbon cloth consists of high-value carbon contents as %82.88 When the other samples are examined.

Table 7.5. *Ultimate analysis of ACC produced by using various chemical activated agents with wt. % (as received).*

Applied Analysis	Carbon Cloth	ACC w/ $ZnCl_2$	ACC w/NH_4Cl	ACC w/H_3PO_4	ACC w/ NaCl
Ultimate Analysis					
C	82.88	82.98	80.31	72.24	83.96
H	2.699	1.912	1.849	3.004	3.095
N	0.417	1.584	1.852	2.128	2.165
O*	14.00	13.52	9.989	22.63	10.78

*Calculated by difference

7.2.3. Surface area analysis

The surface area of carbonized and activated cloth was calculated from N_2 adsorption isotherms by the BET (Brunauer-Emmett-Teller) method. The samples were outgassed at 300 ˚C for 2 hours. Table 7.6 indicates the surface areas of the studies carried out.

As a consequence of the data acquired, the activated carbon cloths manufactured with KOH and K_2CO_3 chemical activating agents show that the fabrics melt, and no data

can be obtained as a result of the production. Furthermore, the fabric produced by using an NH_4Cl chemical agent has a BET surface area of 883.34 m^2/g, but it does not provide flexibility and integrity. Due to this reason found to be unsuitable to produce. When all the activated carbons obtained are compared considering the surface properties of the fabrics, it is seen that the activated carbon fabric at 800 ˚C with $ZnCl_2$ at a ratio of 1:1 by mass gives the best results. In other words, $ZnCl_2$ is acknowledged as the most suitable chemical activating agent with a surface area of 625.58 m^2/g. Besides carbon cloth has a surface area of 355.70 m^2/g. This process can be applied quickly also, the energy consumption during the production and manufacturing cost are lower than the other processes. Accordingly, it is stand out owning to have high surface area when compared to the other carbon materials

Table 7.6. *Parametric studies and BET surface areas of obtained activated carbons.*

Name of Sample	Chemical Activating Agent	Impregnation Ratio (g/g)	Temperature (˚C)	Surface Area (m^2/g)
CC	x	×	600	355.70
ACC/KOH	KOH	1:1	800	x
ACC/K_2CO_3	K_2CO_3	1:1	800	x
ACC/NH_4Cl	NH_4Cl	1:1	800	883.84
ACC/NaCl	NaCl	1:1	800	549.26
ACC/H_3PO_4	H_3PO_4	1:1	800	525.22
ACC/Z600	$ZnCl_2$	1:1	600	269.31
ACC/Z700	$ZnCl_2$	1:1	700	338.61
ACC/Z800-0.5	$ZnCl_2$	1:2	800	516.97
ACC/Z800-1	$ZnCl_2$	1:1	800	625.28
ACC/Z800-1.5	$ZnCl_2$	3:2	800	471.65
ACC/Z800-2	$ZnCl_2$	2:1	800	541.29

7.2.4. FTIR analysis

In order to detect the changes built by the chemicals used in the structure of the produced material, the FT-IR spectra of the carbon cloths saturated with disparate chemicals and produced at different temperatures were appropriated. The FT-IR spectrum of carbon cloths saturated with phosphoric acid, sodium chloride, ammonium chloride, and zinc chloride, respectively, are given in Figure 7.4.

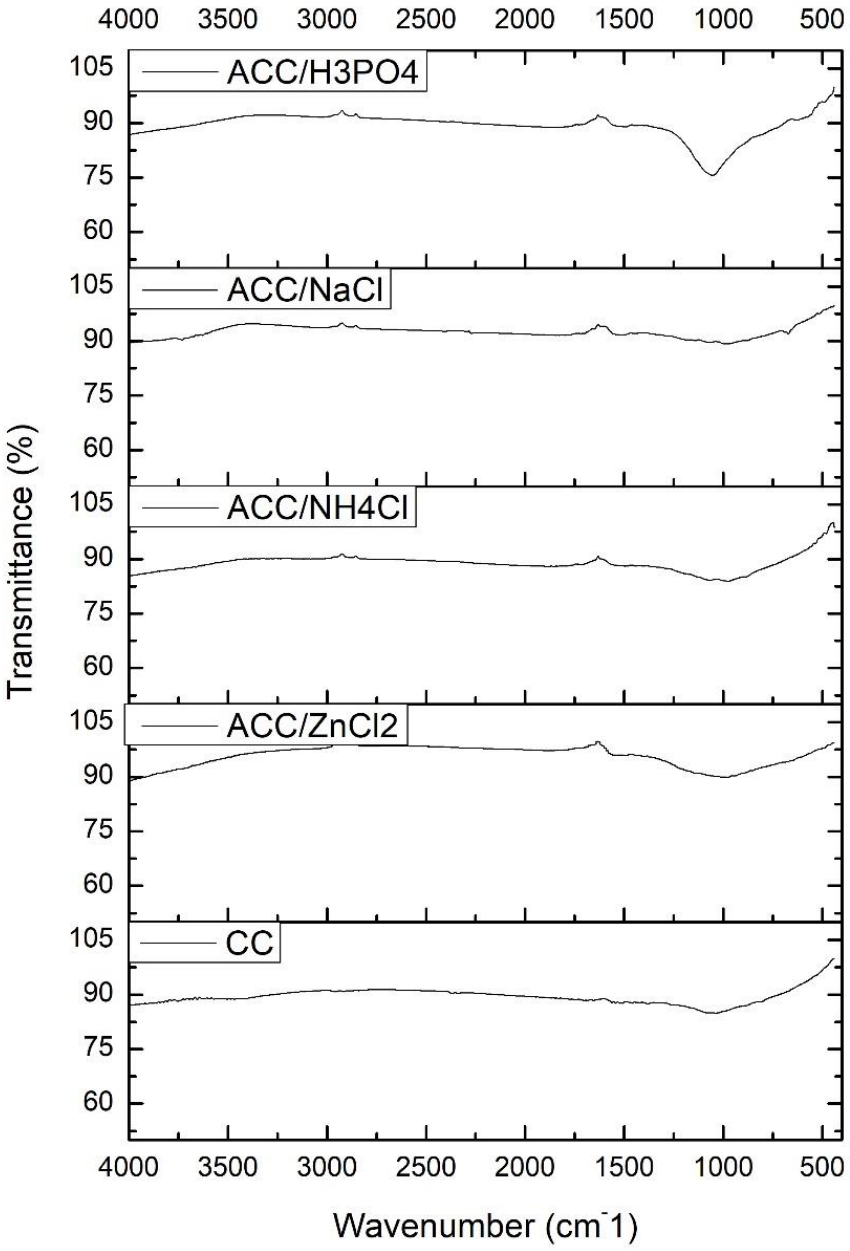

Figure 7.4. *FTIR spectra of carbon cloths impregnated with the chemical activating agent a) H_3PO_4, b) NaCl, c) NH_4Cl, d) $ZnCl_2$, and e) without impregnation.*

When the FT-IR spectra of the activated carbons were compared with the FT-IR spectra of the raw materials, it was determined that the peak of the -OH band, which was seen around 3600 cm^{-1} and caused by the moisture in the structure of the carbon raw material, disappeared with the effect of temperature during carbonization as well as activation as indicated in Figure 7.4-7.6.

The absence of any specific peak in the FTIR analysis means the presence of carbonaceous material. We can say that the activated carbon fabrics, which are saturated with different chemicals activated agents, as shown in Figure 7.4, consist of a high percentage of carbon elements, just as desired. In the activated carbon cloth impregnated with phosphoric acid given in 7.4-a, a peak was formed at approximately 1050 cm^{-1}. According to the information given in the literature, the peak occurring in this range is due to C-O vibrations in the structure. It can be said that carbon formation is less in activated carbon cloths produced with phosphoric acid impregnation compared to other productions. The elemental analysis results given in Table 7.5, confirm the accuracy of the results which obtained from the FTIR spectra.

Flexible and all activated carbon cloths with the highest surface area and suitable pore formation among the impregnated chemical cloths were produced with zinc chloride saturated production. In order to consider the effects of the impregnation proportion and temperature needed for production, parametric studies were achieved. FTIR spectra of adjusted impregnation rates and production temperatures are given in Figure 7.5 and Figure 7.6, respectively.

When $ZnCl_2$ is used, it is seen that there is no significant alteration in the functional groups of the carbon cloth with increasing the impregnation ratio as shown in Figure 7.5. It can be said that the aforementioned chemical does not disrupt the carbon structure formed in production and can increase the formation of pores by fitting to the carbon cloth.

Figure 7.6 shows the effect of temperature on ACCs. FTIR peaks of ACCs obtained taken away carbonization process utilized to cotton fabric (a) and addition activation of carbon cloth obtained which they impregnated with 1:1 zinc chloride by weight at 600 (b), 700 (c), and 800˚C (c) are given. In ACC production at 600°C, the temperature may have been insufficient for the formation of a carbonaceous structure and the removal of zinc chloride. Specific peaks were occurred within 1600 cm^{-1}, 1250 cm^{-1}, and the range of 850-700 cm^{-1}. The peak observed at about 1600 cm^{-1} is thought to originate from esters and aromatic groups. The peak seen at 1250 cm^{-1} is due to -CH vibrations from C-H-C. Furthermore, the weaker bands between 850 and 600 cm^{-1} are ascribed to C-Cl vibrations. When Figure 7.6 is considered, it could be stated that as the temperature increases, the carbonaceous structure formed is more uniform.

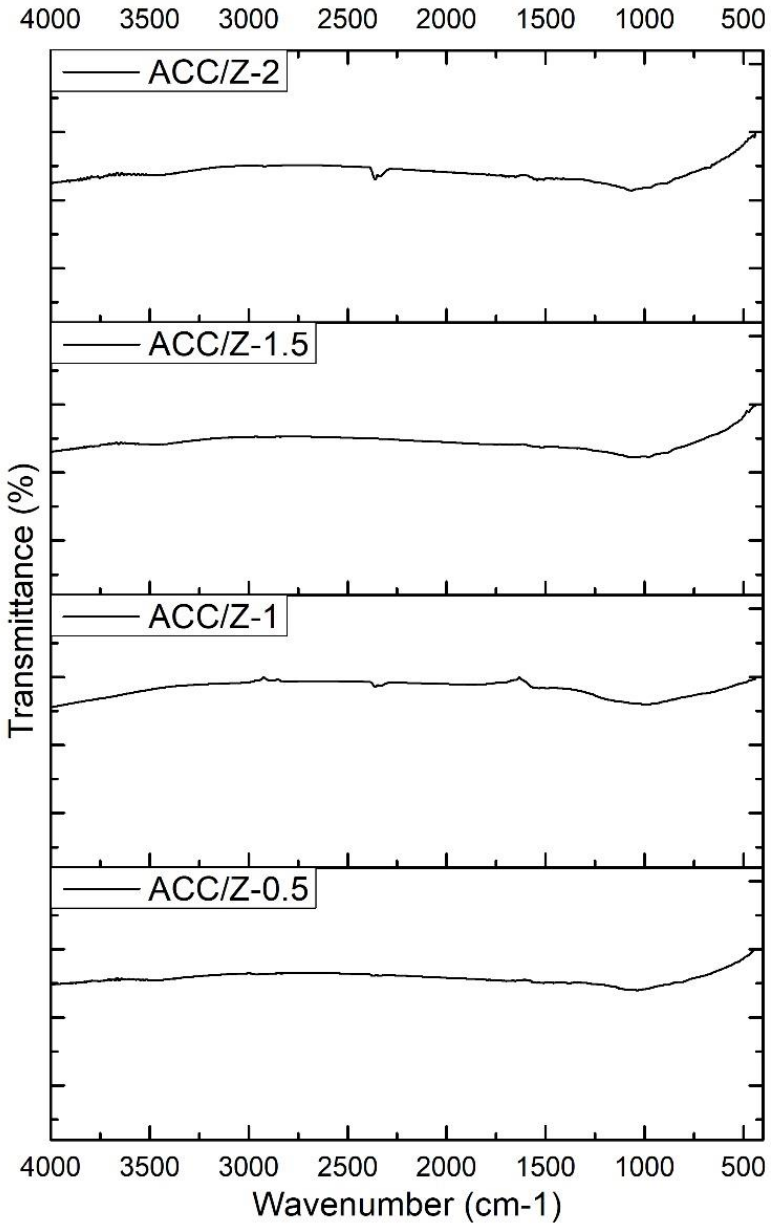

Figure 7.5. *FTIR spectra of carbon cloths impregnated with different chemical activator ratios as a) 2:1, b) 3:2, c) 1:1, and d) 1:2 by weight.*

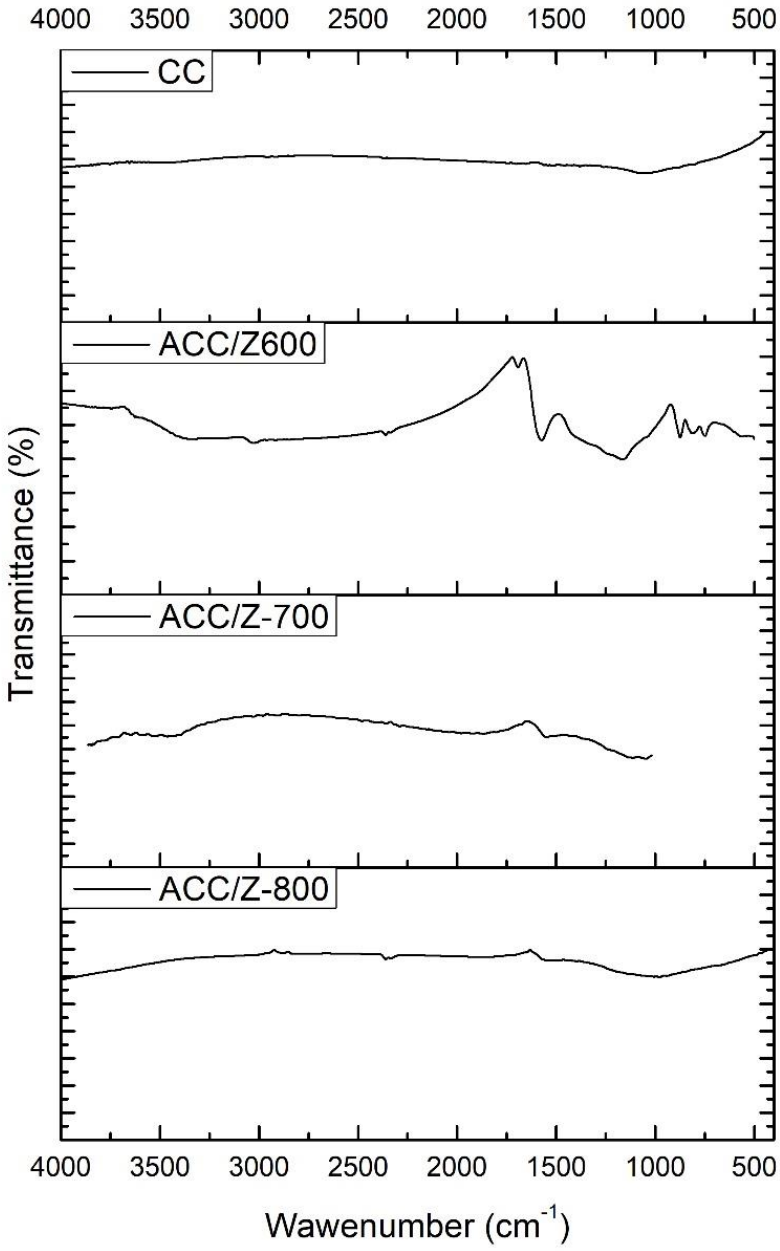

Figure 7.6. *FTIR spectrum of activated carbon produced at a) 600 ˚C without activation, b) 600 ˚C, c) 700 ˚C, and d) 800 ˚C temperature.*

7.2.5. Thermal gravimetric analysis

In Figure 7.7 and 7.8, thermograms of pure cotton denim, carbon cloth, activated carbon cloth produced up to 800°C and impregnated by using a 1:1 molar ratio ZnCl2 by weight, and pure ZnCl2 are given, respectively. The thermal decomposition of zinc chloride in the nitrogen atmosphere starts from about 500°C up to 600°C.

Therefore, the production of activated carbon cloth above 600°C is important in terms of the purity of the cloths impregnated with $ZnCl_2$. It appears that the cellulose decomposes completely at 356.2 ˚C, which corresponds to the 2nd region of the raw material, and thermal stability is achieved when it was working at an average of 600 ˚C and above (3rd region). Adhering to this curve, we determined our operating range as 600-800 ˚C. Besides, the stability of carbon fabric and activated carbon fabric in thermal degradation indicates the suitability of applied temperature.

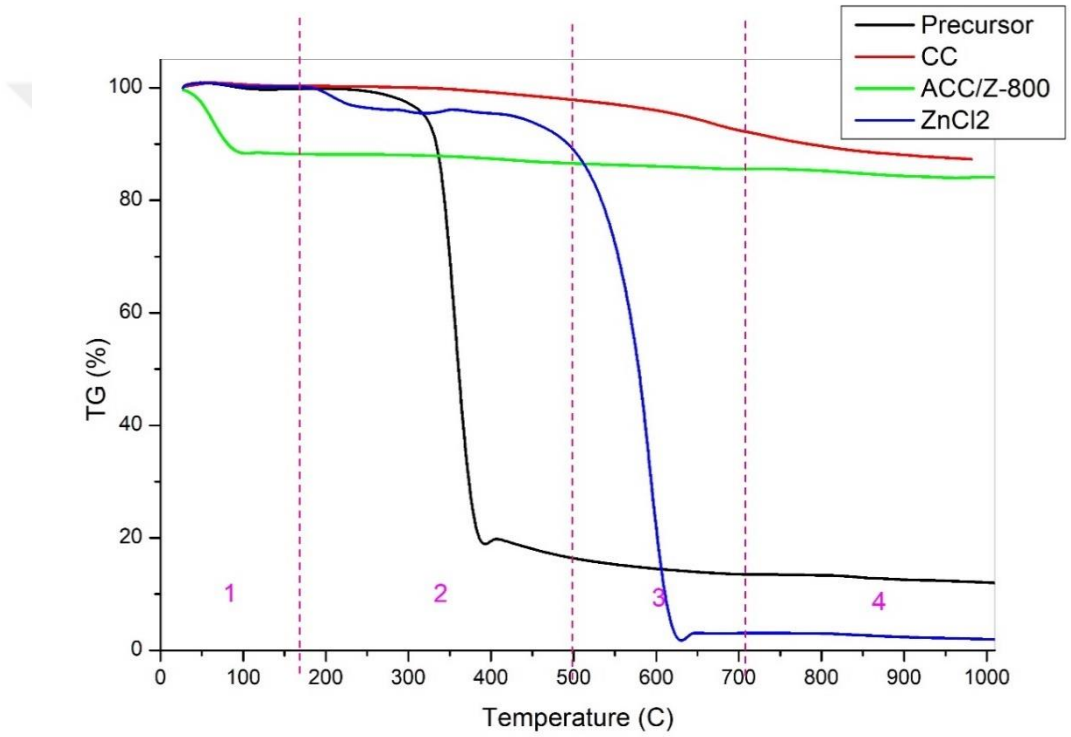

Figure 7.7. *TGA spectra of pure cotton denim (black), carbon cloth (red), activated carbon cloth (green), and zinc chloride (blue).*

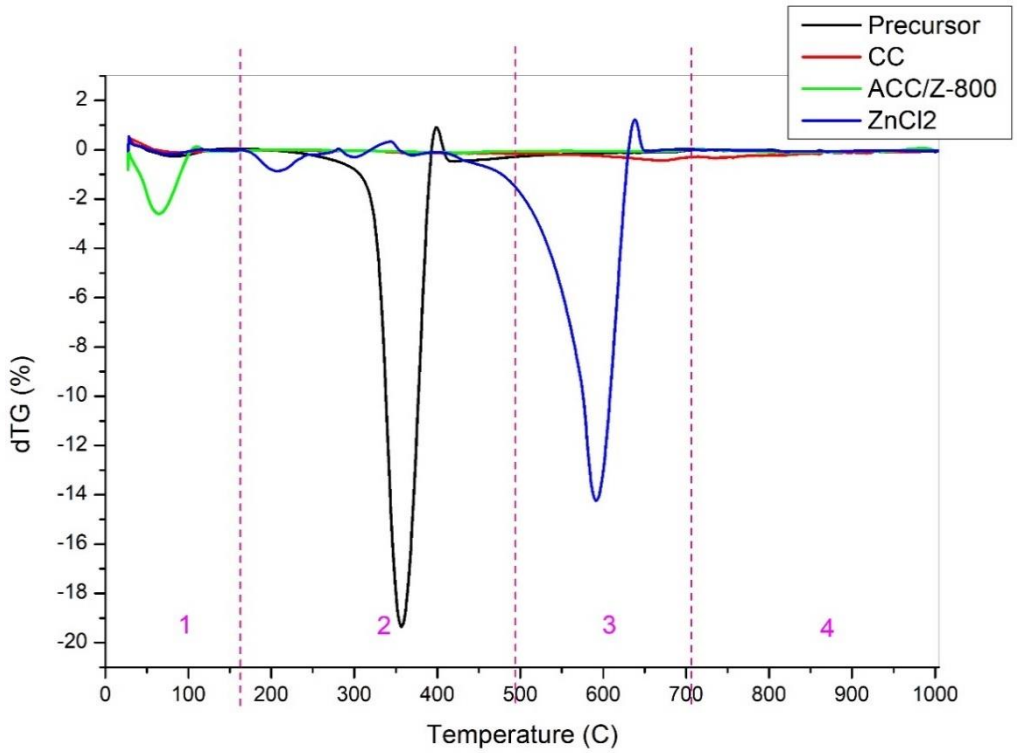

Figure 7.8. *Derivative TG thermographs for pure cotton denim (black), carbon cloth (red), activated carbon cloth (green), and zinc chloride (blue).*

7.2.6. SEM analysis

SEM images provide significant details about the physical morphology of the surface. The changes in the surface after the chemical activation applied to the raw material at different temperatures are seen in the SEM images given below. Each image is given in 100X and its magnified in 2KX dimensions. The changes in the surface of the heating process by carbonizing and activating the raw material given in Figure 7.3 at different temperatures can be seen in the SEM images below. Figure 7.9 shows the SEM image of the carbonized material. Likewise, Figure 7.10-11-12 shows the fibers' morphology of the activated carbon cloths acquired by activation of carbonized material impregnated with zinc chloride by a weight ratio of 1:1 at 600, 700, and 800°C, respectively. From these images, it is evidence that the fabrics are made up of knit denim patterns with 4 tracks.

Comparing Figures 7.9-12 respectively, it is seen that the fiber structure of the carbonized cloth is not only flexible and whole but also pattern texture is preserved. Chemical activated agent ($ZnCl_2$) has occurred on the fiber bunch in carbon material

associated with the activation process at 600°C. The heat treatment is not adequate to remove the chemical from the fabric. In addition, fractures occurred in the fiber structure, and damage to the structural integrity occurred. Nevertheless, the surface morphology of activated carbon cloths is smooth through higher temperature applied. While the fiber structures shrink with the increase in temperature, it is seen that the whole structure is preserved, and pores are formed on the fiber.

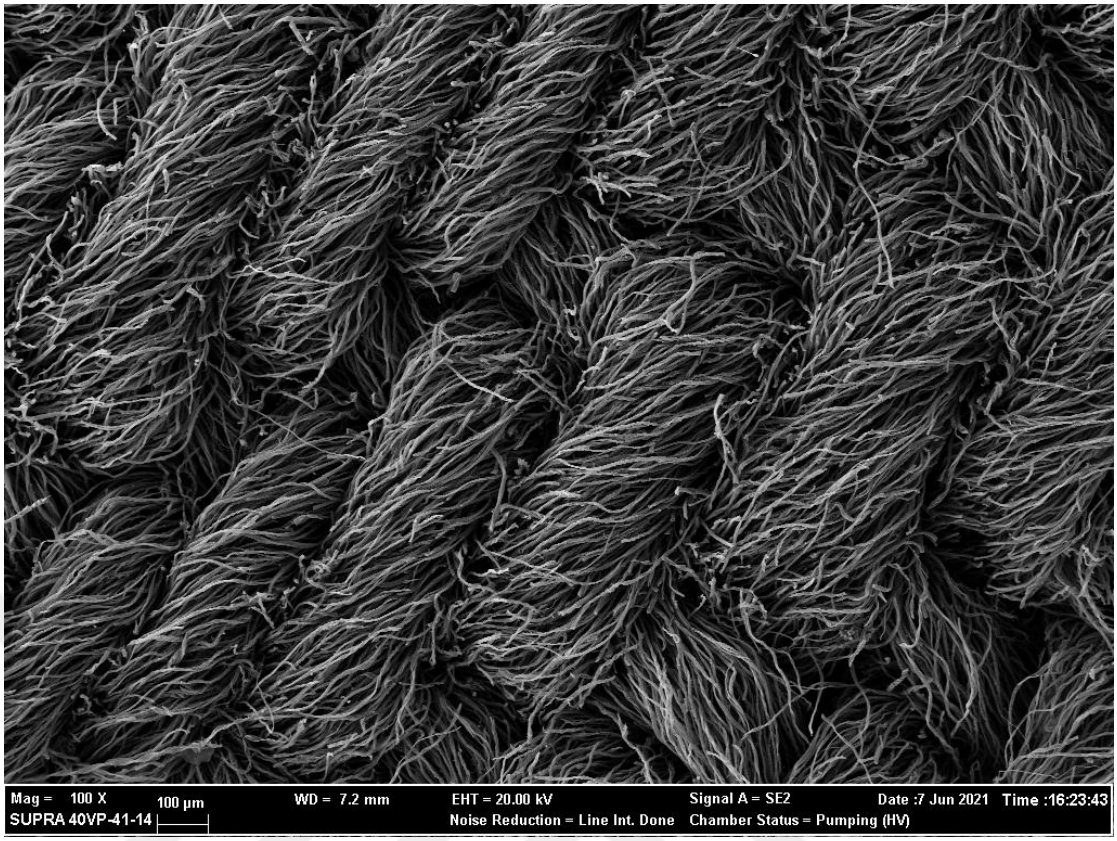

Figure 7.9. *SEM image of carbon cloth obtained from cotton denim waste.*

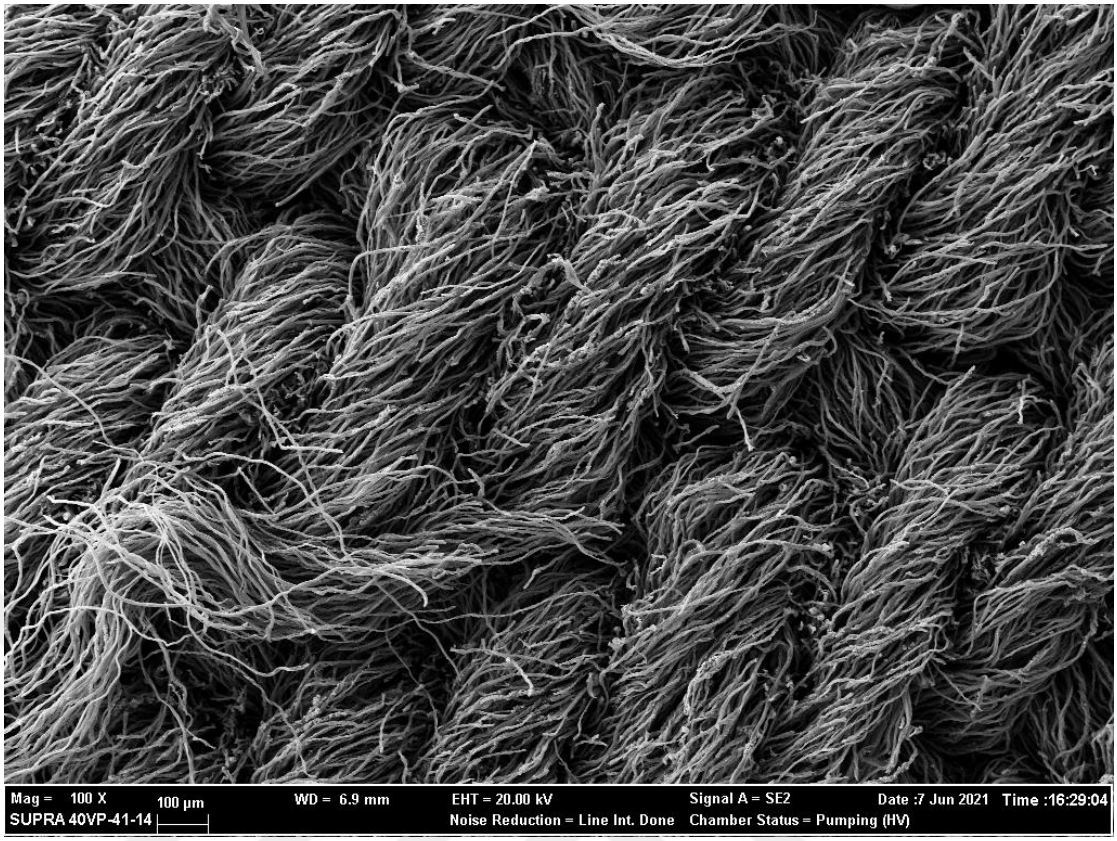

Figure 7.10. *SEM image of activated carbon cloth at 600 ˚C (by using 1:1 $ZnCl_2$).*

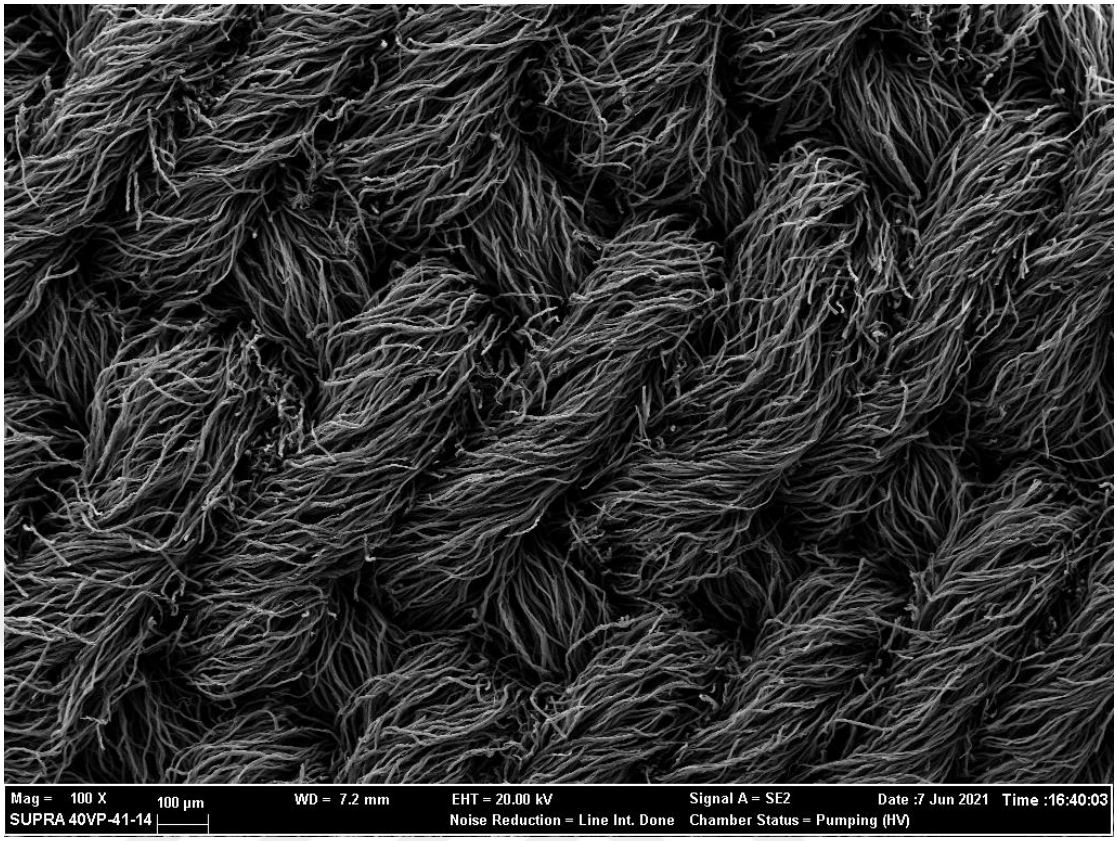

Figure 7.11. *SEM image of activated carbon cloth at 700 ˚C (by using 1:1 ZnCl2).*

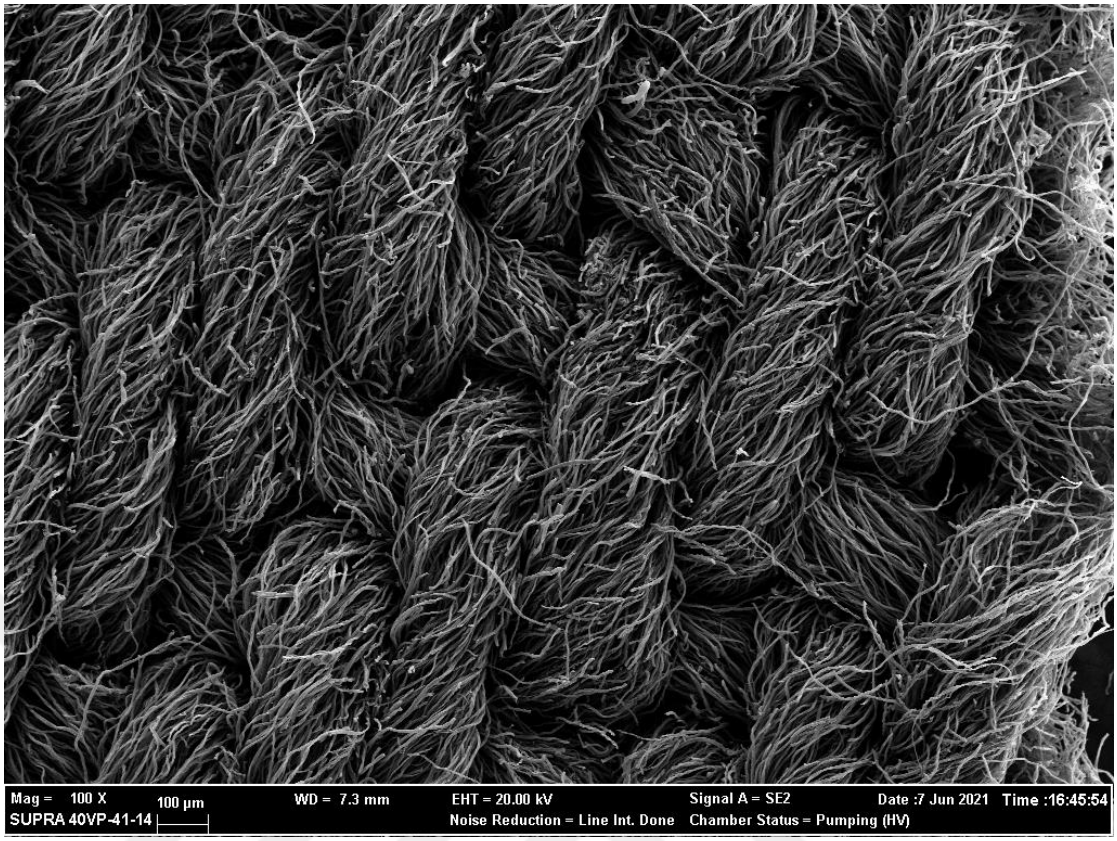

Figure 7.12. *SEM image of activated carbon cloth at 800 ˚C (by using 1:1 ZnCl2).*

7.3. Experimental Results for Composite Material

The strength tests of the composite materials produced were carried out on the concrete samples that consisting of water, cement, and aggregates. Image 7.1 and Figure 7.13 is shown the samples, their contents, and their size details.

Image 7.1. *The samples and their contents.*

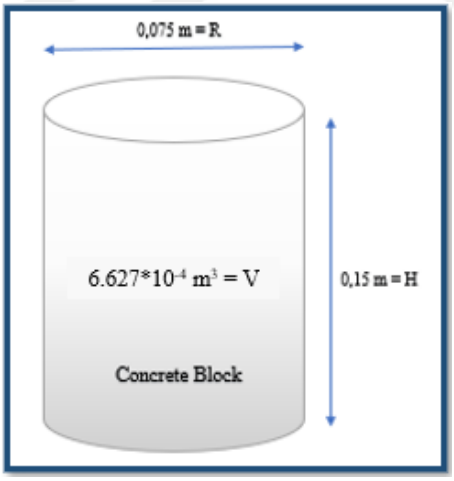

Figure 7.13. *Details of the concrete sample.*

Firstly, the reference concrete was determined as a result of the strength test. In the first step, three types of concrete shown in Table 7.1 were used in order to determine optimum resistance for the strength test based on TS 802 standards. Each experiment was repeated with three samples in order to accept the accuracy of the result. As a conclusion

of the tensile load that given in Table 7.2 as maximum load with MPa, the sample 3 was chosen in order to use in the concretes to be confined for reinforcement of cylinder concrete.

Table 7.7. *The ratios of the consisting concrete (for 1m^3) conducive to determining optimum resistance (TS 802).*

Ingredients	**Sample 1**	**Sample 2**	**Sample 3**
Cement, *kg*	282	352	234
Water, *kg*	282	282	282
Aggregates			
0-5 mm, *kg*	950	950	950
5-15 mm, *kg*	624	624	624
15-22 mm, *kg*	518	518	518

Table 7.8. *The compressive strength results of the samples.*

Experimental / **Sample**		**1**	**2**	**3**
1	Maximum Compressive strength, MPa	17.75	17.49	18.20
2	Maximum Compressive strength, MPa	25.72	24.69	22.64
3	Maximum Compressive strength, MPa	11.32	12.34	12.34

7.3.1. Properties of the resin

Master Brace 4500 SAT was used in order to provide bonding where reinforcement is required in the experimental studies. Master Brace is an innovative repair and strengthening system based on fiber-reinforced composites that provide stability to structural members. Features and benefits of the resin product are given below as items:

- Easy to apply
- Low viscosity
- High strength
- Solvent free

The Master Brace includes two parts in pails as Part A (Resin) and Part B (Hardener). The resin product is obtained by mixing by weight of Part A and Part B homogeneously. Mixing ratio should be 3 to 1, respectively.

Table 7.3 shows technical data sheet of the MasterBrace (Master Builders Solution, 2020).

Table 7.9. *Technical data of the resin product (Master Builders Solution, 2020).*

Product Chemistry	
MasterBrace® SAT 4500 Part A	**Epoxy Resin**
MasterBrace® SAT 4500 Part B	**Epoxy Hardener**
Color	Blue
Mixed Density	1.02 kg/litre
Viscosity	1500-2500 mPa.s
Stiffness TS EN 196 (7 days)	>60 N/mm2
Flexural Strength TS EN 196 (7days)	>50 N/mm2
Bonding Strength to concrete (7 days)	>3,0 N/mm2
Application Temperature	+5°C + 30°C
Pot life	30 min
Fully Cured at 20°C	7 days

7.3.2. Results of the confined cylinder concretes

In Image 7.2, cylinder concrete reinforcement studies show pure concrete (reference sample without confined), carbon denim, glass, aramid, and carbon fiber reinforced resin-based concretes from the right. All the samples are confined with 1.5 layers so that the cylinder concrete can be covered without space. Details of the confinement effect are given in Table 7.3.

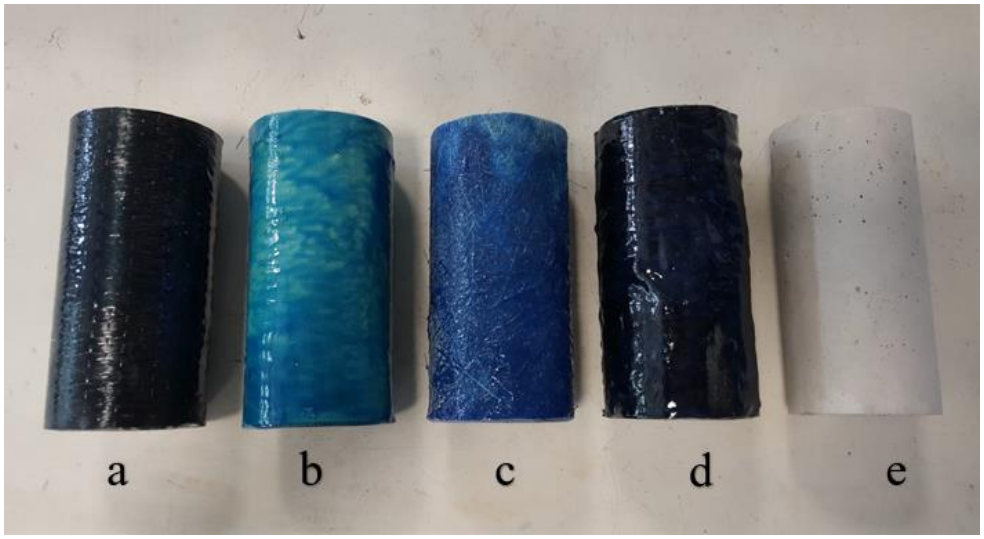

Image 7.2. *Fiber a) carbon fiber (CF), b) aramid (AF), c) glass (GF), d) carbon cloth (CC), and e) without (ref.) reinforced resin-based confined cylinder concrete.*

Table 7.10. *Properties of the confined samples*

Member	Size of sample (cm x cm)	Size of Concrete (diameter x height) (cm x cm)	Number of Layers	Surface Bonded to Concrete
Ref	-	*7.5 x 15*	-	-
CC	*35.35 x 15*	*7.5 x 15*	*1.5*	*Carbon Cloth*
GF	*35.35 x 15*	*7.5 x 15*	*1.5*	*Glass Fiber*
AF	*35.35 x 15*	*7.5 x 15*	*1.5*	*Aramid Fiber*
CF	*35.35 x 15*	*7.5 x 15*	*1.5*	*Carbon Fiber*

Every sample was measured with five repetitions in order to determine how to change the strength of the concrete correctly. The stiffness of the samples against unit deformation under 28 days of axial loading was determined for the samples. As a result of 5 repetition tests, the average strain was calculated as % and the average concrete stress was calculated in MPa. The graphic representation and results of each experiment (for every surface bonded of concrete) are given in Figure 7.14-7.18 and Table 7.4-7.9. In these graphs, the stress - strain behavior under axial pressure is completely related to the tensile strength of the confined material.

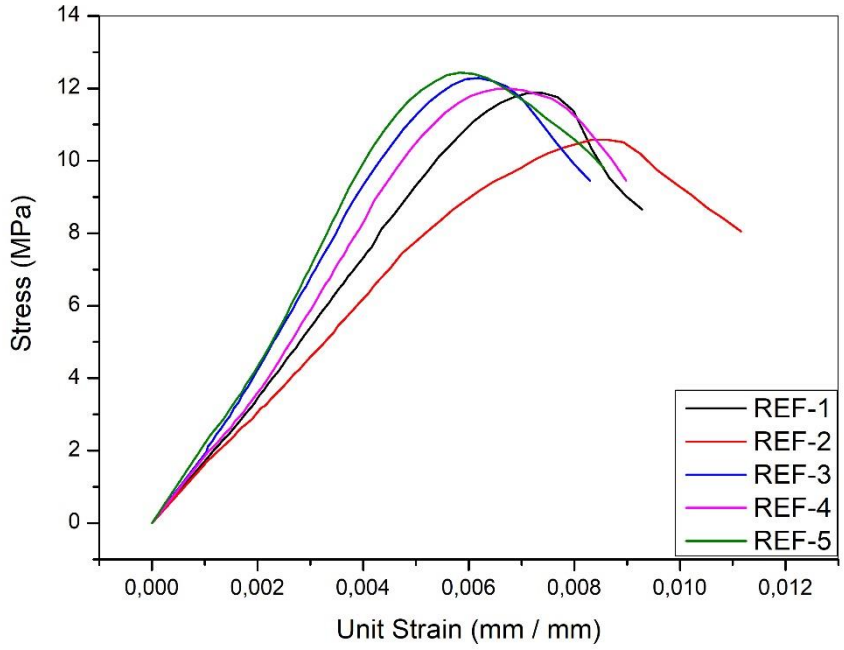

Figure 7.14. *Reference samples' stiffness stress-strain curve on cylinder concrete.*

Table 7.11. *Stiffness results of the reference samples.*

Sample	Stress (MPa)	Strain (mm/mm)
REF1	11.87	0.007
REF2	10.58	0.009
REF3	12.29	0.006
REF4	11.98	0.007
REF5	12.44	0.006
REF_{ave}	11.83	0.700

* % change by reference

Image 7.3. *Creaking of reference concrete.*

When the Figure 7.14 and Table 7.4 are examined, the reference material, which has without confined, shows how can its strength increasing for the other samples. Average value of the REF sample is 11.83 for the stress and also unit stain is as a percentage of 0.7. We'll interpret to the results of the other samples based on these values.

Figure 7.15-7.17 shows the stress against strain of the glass, aramid, and carbon fiber under load, respectively. In aramid and glass fiber, a significant improvement was achieved in the strain values with an increase in strength of 2.12 and 1.86 times, respectively. At this point, although glass fiber is a brittle material compared with other fiber materials, the significant improvement in the strain rate is an important point. With the resin reinforcement applied to the glass fiber, this brittle behavior decreased, and the structure improved. It is apparent from Figure 7.17 that best material which is carbon fiber because it is the confining product that provides the best interaction with the resin. It is the most widely used material in the construction industry due to its properties. The main reason for searching for an alternative material to this material is the cost of the product.

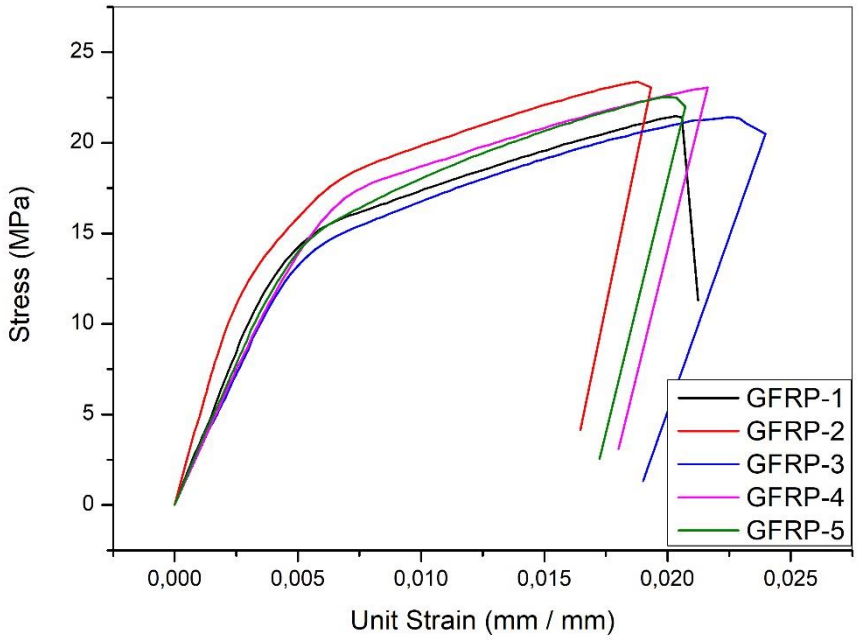

Figure 7.15. *Glass Fiber samples' stiffness stress-strain curve on cylinder concrete.*

Table 7.12. *Stiffness results of the glass fiber sample.*

Sample	Stress (MPa)	Strain (mm/mm)
GFRP1	21.47	0.020
GFRP2	23.36	0.019
GFRP3	21.40	0.023
GFRP4	23.06	0.022
GFRP5	22.53	0.020
$GFRP_{ave}$	22.36	2.07*

* % change by reference

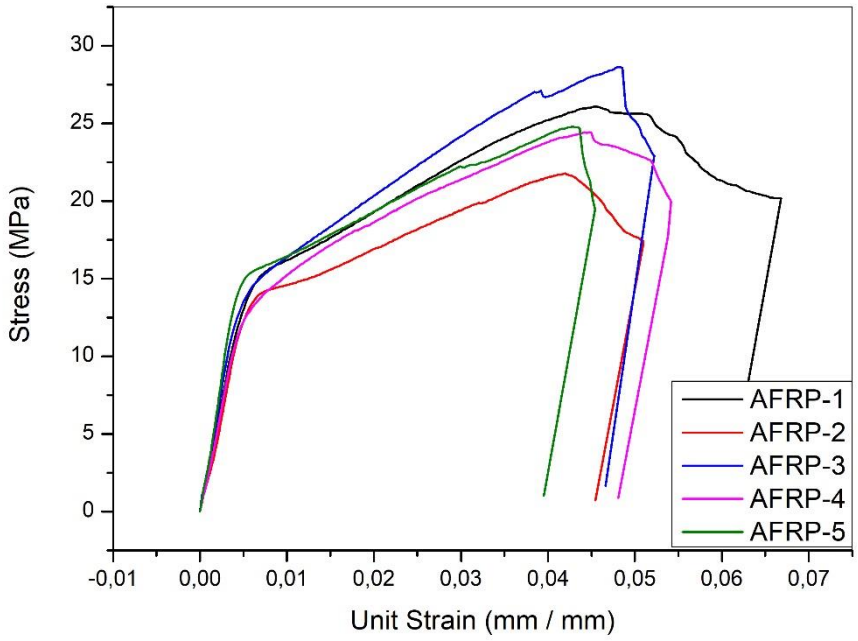

Figure 7.16. *Aramid Fiber samples' stiffness stress-strain curve on cylinder concrete.*

Table 7.13. *Stiffness results of the aramid fiber sample.*

Sample	Stress (MPa)	Strain (mm/mm)
AFRP1	26.08	0.046
AFRP2	21.77	0.042
AFRP3	28.62	0.048
AFRP4	24.42	0.045
AFRP5	24.80	0.043
$AFRP_{ave}$	25.14	4.47*

* % change by reference

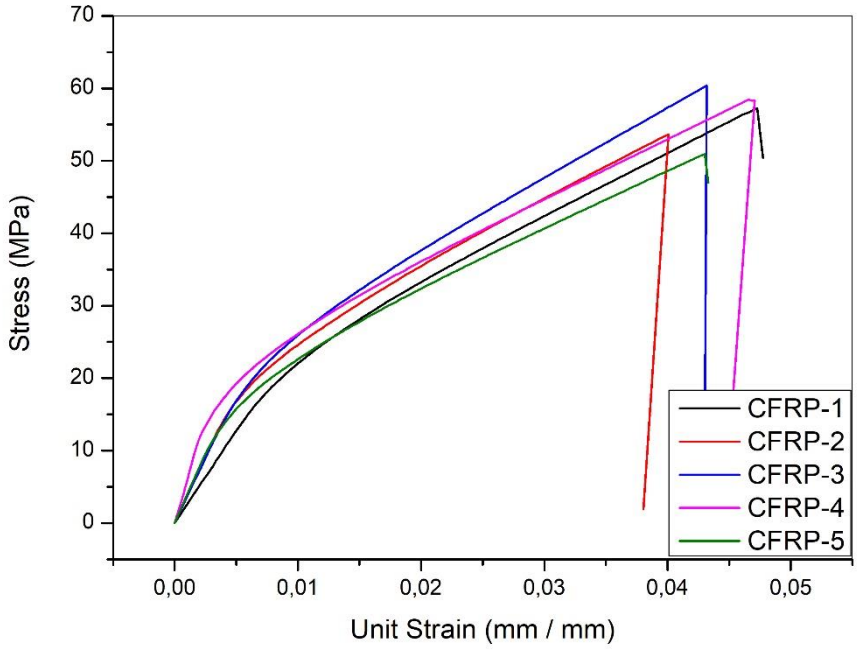

Figure 7.17. *Carbon Fiber samples' stiffness stress-strain curve on cylinder concrete.*

Table 7.14. *Stiffness results of the carbon fiber sample.*

Sample	Stress (MPa)	Strain (mm/mm)
CFRP1	57.27	0.047
CFRP2	53.68	0.040
CFRP3	60.41	0.043
CFRP4	58.44	0.047
CFRP5	50.92	0.043
$CFRP_{ave}$	56.14	4.40

* % change by reference

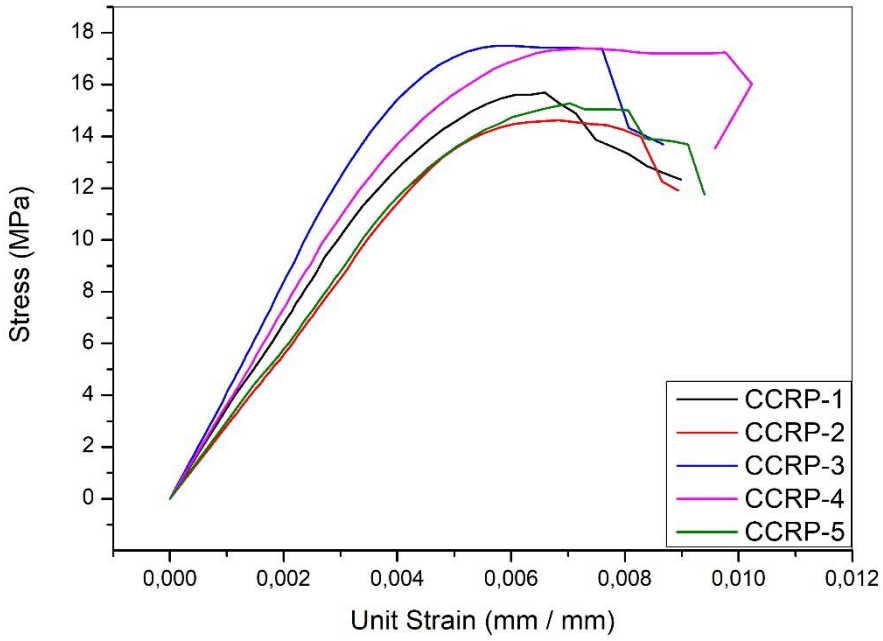

Figure 7.18. *Carbon Cloth samples' stiffness stress-strain curve on cylinder concrete.*

Table 7.15. *Stiffness results of the carbon cloth sample.*

Sample	Stress (MPa)	Strain (mm/mm)
CCRP1	15.69	0.007
CCRP2	14.63	0.007
CCRP3	17.50	0.006
CCRP4	17.39	0.007
CCRP5	15.27	0.007
$CCRP_{ave}$	16.10	0.68*

* % change by reference

Carbon cloth was produced from denim waste. This material showed the high heat resistance, cheap production cost, and lightweight properties. The 28-days axial stiffness is 11.83 MPa on average. In other words, an increase of approximately 36.0% occurred when the low-strength cylindrical concrete samples were confined with the carbon cloth in 1.5 layers. There was no change in the strain compare with the reference sample. Strain values at ultimate stiffness are the order of 0.70% in both cases.

Therefore, even if the confinement material, which is produced in this thesis study, is confined on the cylinder concrete surface in a single layer, it increases the stiffness by 36.00% and keeps the ductility levels constant.

When compared with other products, it's not adequate for the strength of the concrete. But this experiment was done in only 1.5 layers with the carbon cloth. When considering the cost of the production, there is a significant difference from the other materials. Furthermore, production time and less labor are highlights to produce carbon cloth and use.

The product is 5-10 times cheaper than carbon and glass fibers. Although higher strength capacities are achieved with carbon fiber, glass fiber and aramid fiber, this problem will can be solved with more layers confined of the cylinder concrete with carbon cloth.

Image 7.4. *Cracking of concrete which confined with a) glass fiber, b) aramid fiber, c) carbon fiber and d) carbon cloth fiber.*

8. CONCLUSION AND RECOMMENDATION

In this study, denim waste was evaluated as an alternative raw material for the production of flexible carbon composite materials.

Based on the experiments conducted within the scope of this thesis, following results were obtained,

- Carbon cloths were obtained by carbonization and activation studies of used denim wastes. The characterization studies of the used carbon cloth and its application studies, which repair of the structural elements in the building and construction, were carried out.
- Denim cloth was converted into carbon cloth with using oven at 600˚C. It's also converted into activated carbon cloth with using chemical activated agent (NH_4Cl, $AlCl_3$, $ZnCl_2$, $CuCl_2$, $FeCl_3$, H_3PO_4, and Na_2HPO_4) at 600, 700 and 800˚C.
- Optimum production conditions: The porous texture of the samples was determined by evaluating their strength, chemical composition, and effects on fiber morphology. According to the obtained data, the optimum carbonization process for carbon cloth, cotton denim wastes were carbonized at 600 °C under nitrogen (N_2) flow of 100 cm3/min and at a heating rate of 10 °C/min in a stainless-steel fixed bed reactor.
- Optimum chemical activation process for activated carbon cloth were obtained with pure cotton denim wastes were impregnated with 1:1 $ZnCl_2$ chemical activating agent by weight at 800 °C under nitrogen (N_2) flow of 100 cm^3/min and at a heating rate of 10 °C/min in a stainless-steel fixed bed reactor
- The obtained products' surface areas were 355.70 m^2/g and 625.28 m^2/g for carbon and activated carbon cloth, respectively. The surface area was increased approximately two times higher with the used $ZnCl_2$ chemical activation agent Product were impregnated with $ZnCl_2$. These values are adequate in order to applying adsorption processes.
- The most important feature of the products produced from waste is the flexible material. The material has also a whole structure without tearing.

These features provide convenience while using them in the application areas.

- The produced carbon product has been used in Structural Engineering for strengthening-repairing structural elements damaged or inadequate against earthquakes after earthquakes. Activated carbon fabrics were used for volatile organic component (VOC) removal (This thesis not included VOC experimental part).
- Since it is produced from waste denim cloths, the carbonization process preserves the original texture form of the denim. Therefore, there is no need for an additional weaving process, as in other confining fibers. Moreover, the 45° weaving form in the denim enables the fibers to work more effectively against both shear forces and bending moments.
- Compression tests with resin-based carbon cloths confining low-strength cylindrical concrete samples were investigated. It has increased the axial stiffness to an average of 16.10 MPa with one and a half layers of confining. Thus, an increase of 36% in the strength of the cylindrical sample was observed.
- On the other hand, the strain values observed at the ultimate stiffness levels are in the order of 0.70% in both cases. Consequently, the produced fiber increases the stiffness by 36.00% with a single layer confining on the concrete surface, while keeping the ductility levels constant.
- When the application results were evaluated, a high strength and suitable ductility or rigidity were not obtained compared to other fiber products. However, it provides advantages with its low cost, ease of application, and less labor compared to its equivalents. Therefore, the same performance levels can be achieved by confining the element surface in more layers and opposite the fiber tissue.
- In brief, it is important to emphasize that carbon fabric has great potential as a low-cost, alternative material that can be used in many different areas such as building-construction applications, automotive sector, energy sector, and adsorption processes.

It was concluded that denim wastes can be effectively used as an efficient, low-cost, environmentally friendly, and alternative starting material in the production of flexible carbon cloth, taking into consideration that the economic, technological, and ecological balance of resin reinforced carbon composite materials.

REFERENCES

Akter, M. (2021). *Development of epoxy/GNPs and hybrid carbon fiber/epoxy/GNPs nanocomposites and their mechanical/thermal/electrical properties* (Doctoral dissertation, Erciyes University).

Altun, Ş. (2012). Prediction of textile waste profile and recycling opportunities in Turkey. *Fibres and Textiles in Eastern Europe*, *94*(5), 16-20.

Altun, Ş. (2016). *Tekstil üretim ve kullanım atıklarının, geri kazanımı, çevresel ve ekonomik etkileri.* Uşak: Uşak Ticaret ve Sanayi Odası

Alves, A. T., Lasmar, D. J., de Andrade Miranda, I. P., da Silva Chaar, J., & dos Santos Reis, J. (2021). The Potential of Activated Carbon in the Treatment of Water for Human Consumption, a Study of the State of the Art and Its Techniques Used for Its Development. *Advances in Bioscience and Biotechnology*, *12*(6), 143-153.

Aygün, A. (2002). *Yerli doğal hammaddelerden aktif karbon üretimi ve adsorpsiyon özelliklerinin incelenmesi* (Doctoral dissertation, Fen Bilimleri Enstitüsü).

Bakış-Doğru, I., (2016) , Fabrication and characterization of carbon nanotube based supercapacitor electrodes master thesis, Ankara: ODTÜ Doğal ve Uygulamalı Bilimler Enstitüsü

Balasubramanian, M. (2014). *Composite materials and processing* (pp. 335-343). Boca Raton: CRC press.

Bedia, J., Peñas-Garzón, M., Gómez-Avilés, A., Rodriguez, J. J., & Belver, C. (2020). Review on activated carbons by chemical activation with FeCl3. *C*, *6*(2), 21.

Blackwell, W. (2014). Hoboken. *NJ, USA.*

Can, Ö. (2008). Endüstride Kullanılan Teknik Tekstiller Üzerine Bir Araştırma. *Tekstil Teknolojileri Elektronik Dergisi*, *3*, 31-43.

Chandramohan, D., & Marimuthu, K. (2011). A review on natural fibers. *International Journal of Research and Reviews in Applied Sciences*, *8*(2), 194-206.

Connelly, A. (2017). BET surface area. *Andy Connelly*.

Cordero-Lanzac, T., Garcia-Mateos, F. J., Rosas, J. M., Rodriguez-Mirasol, J., & Cordero, T. (2018). Flexible binderless capacitors based on P-and N-containing fibrous activated carbons from denim cloth waste. *Carbon*, *139*, 599-608.

Ekundayo, G., & Adejuyigbe, S. (2019). Reviewing the development of natural fiber polymer composite: a case study of sisal and jute. *American Journal of Mechanical and Materials Engineering*, *3*(1), 1.

Erkan, M. (2013). Pamuk lifi ile uyumlu sentetik lif karışımlarından iplik ve kumaş üretilip özelliklerinin karışılaştırılması Yayımlanmamış Yüksek Lisans Tezi. Isparta: Süleyman Demirel Üniversitesi, Fen Bilimleri Enstitüsü

European Commission's Institute for Prospective Technological Studies, Date Accessed: 01.03.2022

https://labfresh.eu/pages/fashion-waste-index?lang=en&locale=en

Gülcan, E. (2019). *Karbon dokuma kumaş takviyeli kompozit malzemelerin fiziksel özelliklerinin incelenmesi* (Doctoral dissertation, Bursa Uludag University (Turkey)).

Gürbüz, M., & Mutuk, T. (2019). Karbon Esaslı Malzeme Takviyeli Titanyum Kompozitler ve Grafen Üzerine Yeni Eğilimler. *Mühendis ve Makina*, *60*(695), 101-118.

Huidobro, A., Pastor, A. C., & Rodrıguez-Reinoso, F. (2001). Preparation of activated carbon cloth from viscous rayon: Part IV. Chemical activation. *Carbon*, *39*(3), 389-398.

Hwang, N., & Barron, A. R. (2011). BET surface area analysis of nanoparticles. *The Connexions project*, 1-11.

Indian Institute of Technology Kanpur, Accessed Date: 09.03.2022

https://www.iitk.ac.in/che/pdf/resources/TGA-DSC-reading-material.pdf

İlter, M. (2015). Tekstil üretimi ve yardımcı kimyasallar. *TMMOB Kimya Mühendisleri Odası, İzmir*.

Jena, S., Tokas, R., Thakur, S., & Sahoo, N. K. (2015). Characterization of optical thin films by spectrophotometry and atomic force microscopy. *SMC Bulletin*, *6*(1), 1-9.

Jiang, X., Bai, Y., Chen, X., & Liu, W. (2020). A review on raw materials, commercial production, and properties of lyocell fiber. *Journal of Bioresources and Bioproducts*, *5*(1), 16-25.

Kılıç, G. B., & Karahan, A. G. (2010). Fourier dönüşümlü kızılötesi (FTIR) spektroskopisi ve laktik asit bakterilerinin tanısında kullanılması. *Gıda*, *35*(6), 445-452.

Kılıç, M. (2015), Biyokütle esaslı karbon köpük üretimi ve karakterizasyonu Doktora Tezi, Eskişehir: Anadolu Üniversitesi Fen Bilimleri Enstitüsü.

Kılıç, M. (2009). *Euphorbia rigida'dan elde edilen aktif karbonun sulu çözeltilerdeki adsorpsiyon özelliklerinin ve kinetiğinin incelenmesi* (Master's thesis, Anadolu Üniversitesi).

Koszewska, M. (2018). Challenges for the textile and clothing industry, AUTEX Research Journal, 18 (4), 337-347

Kozak, M. (2010). Tekstil atıkların yapı malzemesi olarak kullanım alanlarının araştırılması. *Yapı Teknolojileri Elektronik Dergisi*, *6*(1), 62-70.

Küçükgül, E. Y. (2004). Ticari aktif karbon üretimi ve özelliklerinin belirlenmesi. *Dokuz Eylül Üniversitesi Mühendislik Fakültesi Fen ve Mühendislik Dergisi*, *6*(3), 41-56.

Master Builders Solution (2020). MasterBrace SAT 4500 epoxy based adhesive for MasterBrace FIB system. *User Manual, 1-3.* Accessed Date: 13.05.2022

https://www.master-builders-solutions.com/en-tr/products/masterbrace/masterbrace-sat-4500-

Mazumdar, S. (2001). *Composites manufacturing: materials, product, and process engineering*. CrC press.

Mendoza-Castillo, D. I., Reynel-Avila, H. E., Bonilla-Petriciolet, A., & Silvestre-Albero, J. (2016). Synthesis of denim waste-based adsorbents and their application in water defluoridation. *Journal of Molecular Liquids*, *221*, 469-478.

Middle East Technical University, Accessed Date: 10.12.2021

https://merlab.metu.edu.tr/tr/fourier-donusumlu-kizil-otesi-spektrometresi

Nieto-Delgado, C., Partida-Gutierrez, D., & Rangel-Mendez, J. R. (2019). Preparation of activated carbon cloths from renewable natural fabrics and their performance during the adsorption of model organic and inorganic pollutants in water. *Journal of Cleaner Production*, *213*, 650-658.

Örs, S. (2017). *Farklı Karbon Tekstilleriyle Ve Reçinelerle Oluşturulan Karbon Kompozitlerin Karakterizasyonu* (Doctoral dissertation, Fen Bilimleri Enstitüsü).

Pastor, A. C., Rodrıguez-Reinoso, F., Marsh, H., & Martınez, M. A. (1999). Preparation of activated carbon cloths from viscous rayon. Part I. Carbonization procedures. *Carbon*, *37*(8), 1275-1283.

Perdue University, Accessed Date: 12.03.2022

https://www.purdue.edu/ehps/rem/laboratory/equipment%20safety/Research%20Equipment/sem.html

Polovina, M., Babić, B., Kaluderović, B., & Dekanski, A. (1997). Surface characterization of oxidized activated carbon cloth. *Carbon*, *35*(8), 1047-1052.

Popescu, C. M. (2017). Wood as a bio-based building material. *Performance of Bio-Based Building Materials*, 21-96.

Priyanka, P., Dixit, A., & Mali, H. S. (2017). High-Strength Hybrid Textile Composites with Carbon, Kevlar, and E-Glass Fibers for Impact-Resistant Structures. A Review. *Mechanics of Composite Materials*, *53*(5), 685-704.

Saake, B., & Lehnen, R. (2000). Lignin. *Ullmann's Encyclopedia of Industrial Chemistry*.

Scimed, The Scientific Equipment Company, Accessed Date: 07.03.2022

https://www.scimed.co.uk/education/sem-scanning-electron-microscopy/

Sevimli, K. (2017). *Kimyasal aktivasyon yöntemi kullanılarak atık çay çalısından aktif karbon üretimi* (Master's thesis, Fen Bilimleri Enstitüsü).

Skoog, D. A., Holler, F. J., & Crouch, S. R. (2007). *Instrumental analysis* (Vol. 47). Belmont: Brooks/Cole, Cengage Learning.

Qin, Y. (2016). A brief description of textile fibers. *Medical textile materials*, *3*, 23-42.

Reise, S. P., & Waller, N. G. (2009). Item response theory and clinical measurement. *Annual review of clinical psychology*, *5*, 27-48.

Rodríguez-Reinoso, F., Pastor, A. C., Marsh, H., & Martínez, M. A. (2000). Preparation of activated carbon cloths from viscous rayon. Part II: physical activation processes. *Carbon*, *38*(3), 379-395.

Rodriguez-Reinoso F., Pastor A. C., Marsh H., Huidobro A. (2000). Preparation of activated carbon cloths from viscous rayon Part III. Effect of carbonization on CO activation. Carbon, 38, 397–406.

Üçgül, İ. ve Elibüyük, U. (2014). Tekstil atıklarının piroliz ile değerlendirilmesi, SDU Teknik Bilimler Dergisi, 4 (2), 40-46.

Üçgül, İ. Ve Turak, B. (2015). Tekstil katı atıklarının geri dönüşümü ve yalıtım malzemesi olarak değerlendirilmesi, Akademik Platform Mühendislik ve Fen Bilimleri Dergisi, (3), 39-48.

Var, C. (2021). *Karbon kumaş takviyeli hibrit kompozit yapıların mekanik özelliklerinin araştırılması* (Master's thesis, Pamukkale Üniversitesi Fen Bilimleri Enstitüsü).

Varol, E. A. (2007). *Farklı biyokütlelere değişik ısıl işlemler uygulanması ve elde edilen ürün özelliklerinin belirlenmesi* (Doctoral dissertation, Anadolu University (Turkey)).

Walock, M. J. (2012). *Nanocomposite coatings based on quaternary metal-nitrogen and nanocarbon systems*. The University of Alabama at Birmingham.

Yağmur, F., Bozbıyık, A., & Hancı, İ. H. (2003). Elektromanyetik dalgaların insan biyokimyası üzerine etkileri. *Sted*, *12*(8), 296-7.

Yang, H., Yan, R., Chen, H., Lee, D. H., & Zheng, C. (2007). Characteristics of hemicellulose, cellulose, and lignin pyrolysis. *Fuel*, *86*(12-13), 1781-1788.

Yimer, T., & Ababa, A. (2013). Development of Natural Fiber composites for Automotive Applications.

Yörük, C. M. (2019). Selüloz bazlı kumaştan üstün özellikli aktif karbon kumaş üretimi ve karakterizasyonu.

Yurtay, A. (2020). *Ani (flaş) ısıtma yöntemi ile aktif karbon üretimi, karakterizasyonu ve antibiyotik gideriminde kullanımı* (Master's thesis, Lisansüstü Eğitim Enstitüsü).

Zhuang, S., Shi, X., & Lee, E. S. (2015, June). A Review on Non-PGM Cathode Catalysts for Polymer Electrolyte Membrane (PEM) Fuel Cell. In *International Conference on Fuel Cell Science, Engineering and Technology* (Vol. 56611, p. V001T04A005). American Society of Mechanical Engineers.

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