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# PREPARATION, CHARACTERIZATION, AND OPTIMIZATION OF SILVER OXIDE NANOFLUIDS

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FOR
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IN
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BY

AYAN YACIN IBRAHIM

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PREPARATION, CHARACTERIZATION, AND OPTIMIZATION OF SILVER OXIDE NANOFLUIDS

By Ayan YACIN IBRAHIM

August 2022

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## ABSTRACT

# PREPARATION, CHARACTERIZATION, AND OPTIMIZATION OF SILVER OXIDE NANOFLUIDS



Ayan YACIN IBRAHIM

Master of Science in Chemical Engineering

Advisor: Asst. Prof. Dr. Semahat DORUK

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In this study, nanofluids in which the basic fluid is deionized water were prepared using silver oxide nanoparticles via two-step method, and the physical properties of these nanofluids were determined. Nanofluids were prepared in three different ratios of 0.1%, 0.5% and 1%. After the nanofluids were prepared, viscosity, thermal conductivity, zeta potential and particle diameter were measured. In the study, it was observed that the thermal conductivity, viscosity and zeta potential values increased as the ultrasonic mixing time and the amount of solids by volume increased. The thermal conductivity values were higher than the thermal conductivity values of deionized water, which is the base fluid of the nanofluid, and were in the range of 0.669 W/m.K-0.671 W/m.K. Viscosity values are in the range of 1 cp-1.07 cp and are higher than the viscosity value of deionized water. The zeta potential is in the range of 37-47.



**2022, 64 pages**

**Keywords:** Nanofluids, Silver oxide nanoparticles, Viscosity, Thermal conductivity, Zeta potential

# ÖZET

## GÜMÜŞ OKSİT NANO AKIŞKANLARIN HAZIRLANMASI, KARAKTERİZASYONU VE OPTİMİZASYONU



Ayan YACIN IBRAHIM

Kimya Mühendisliği, Yüksek Lisans

Tez Danışmanı: Dr. Öğr. Üyesi Semahat DORUK



Ağustos 2022



Bu çalışmada gümüş oksit nanoparçacıklar kullanılarak, bazik akışkanın saf su olduğu nanoakışkanlar iki aşamalı yöntemle hazırlanmış ve bu nanoakışkanların fiziksel özellikleri belirlenmiştir. Nanoakışkanlar %0,1, %0,5 ve %1 olmak üzere üç farklı oranda hazırlanmıştır. Nanoakışklanlar hazırlandıktan sonra viskozite, termal iletkenlik, zeta potansiyeli ve partikül çapı ölçülmüştür. Çalışmada ultrasonik karıştırma süresi ve hacimce katı miktarı arttıkça ısı iletim katsayısı, viskozite ve zeta potansiyeli değerlerinin arttığı görülmüştür. Isı iletim katsayıları nanoakışkanın baz sıvısı olan saf suyun ısı iletim katsayısından yüksek çıkmış ve 0,669 W/m.K-0,671 W/m.K aralığındadır. Viskozite değerleri 1 cp-1,07 cp aralığındadır ve saf suyun viskozite değerinden yüksektir. Zeta potansiyeli 37-47 aralığındadır.



**2022, 64 sayfa**

**Anahtar Kelimeler:** Nanoakışkanlar, Gümüş oksit nanopartiküller, Viskozite, Termal iletkenlik, Zeta potansiyeli

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**Ayan YACIN IBRAHIM**

**Çankırı-2022**

**CONTENTS**

## LIST OF SYMBOLS

| | |
|---|---|
| % | Percent |
| $<$ | Less than |
| $>$ | Greater than |
| ± | More or less |
| °C | Celsius degree |
| $C_p$ | Specific heat |
| $C_{p,bf}$ | Specific heat of basefluid |
| $C_{p,nf}$ | Specific heat of nanofluid |
| $F_B$ | Buoyant force |
| $F_D$ | Drag force |
| $F_G$ | Gravitational force |
| g | Gravitational acceleration |
| k | Constant of thermal conductivity |
| $k_B$ | Boltzmann's constant |
| $k_{bf}$ | Basefluid thermal conductivity |
| $k_{eff}$ | Effective thermal conductivity |
| $k_{np}$ | Nanoparticle thermal conductivity |
| mV | Millivolt |
| nf | Nanofluid |
| r | Radius |
| Re | Reynolds number |
| $U_0$ | Velocity |
| $\beta$ | Constant of hydrodynamic interaction of nanoparticles and fluid |
| $\gamma$ | Ratio of particle radius to matrix thickness |
| $\zeta$ | Zeta potential |
| $\mu_{bf}$ | Viscosity of basefluid |
| $\mu_{eff}$ | Effective viscosity |
| $\mu_{nf}$ | Viscosity of nanofluid |
| $\pi$ | Pi |
| $\rho$ | Density |
| $\rho_f$ | Density of fluid |
| $\rho_p$ | Density of particle |
| $\varphi$ | Nanoparticle volume ratio |
| $\psi$ | Sphericity coefficient |

## LIST OF ABBREVIATIONS

| | |
|---|---|
| $cm^3$ | Cubic centimeter |
| CNT | Carbon nanotube |
| CVD | Chemical vapor deposition |
| DIW | Deionized water |
| DLS | Dynamic light scattering |
| EG | Ethylene glycol |
| FTIR | Fourier-transform infrared spectroscopy |
| HeNe | Helium-neon |
| m | Meter |
| $mm^3$ | Cubic millimeter |
| MWCNTs | Multi-walled carbon nanotubes |
| NFs | Nanofluids |
| nm | Nanometer |
| pH | Potential of hydrogen |
| PVD | Physical vapor deposition |
| SEM | Scanning electron microscopy |
| SI | International system of units |
| TEM | Transmission electron microscopy |
| TGA-DSC | Thermogravimetric analysis-Differential scanning calorimetry |
| UV-VIS | Ultraviolet-visible spectroscopy |
| µm | Micrometer |

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# 1. INTRODUCTION

The human desire to preserve and improve our well-being, that of our family and that of our community is undoubtedly the main reason for the demand of energy thus the use of heat transfers. This makes them today, the pillars of the different industrial processes that we use regularly. But this mode of consumption is problematic in more than one way in the industrial world and in the society as a whole, which is becoming aware of the inevitability of the exhaustion of fossil fuels.

Presented in this way, the energy problem seems essentially technological and any answer to the problems posed by this model can only be technological. For better energy management, it would be judicious to develop new processes.

In the industrial sector, there is a real need to develop new ways for improving the thermal behavior of fluids used in heat exchangers. In these exchangers, a fluid is used to remove heat. The most common heat transfer fluids are water, sometimes mixed with ethylene glycol. However, the ability of these fluids to transfer the heat is limited with their low thermal conductivity which is one of the principal thermophysical properties to consider when evaluating the potential heat exchange (Tawfik 2017)

Since the end of the 1990s, significant advances in chemistry have facilitated the production of nano-sized particles, which, when dispersed in a base fluid, constitute nanofluids. The idea of improving the thermal properties and characteristics of a fluid is not recent. Choi and Eastman were the two first persons to introduce the term nanofluid in 1995 in the US (Choi 1995).

Nanofluids are colloidal suspensions produced by dispersing solid particles with a nanometric dimension into a base liquid (Sheikholeslami and Ganji 2017). Accordingly, nanofluids have aroused curiosity and become one of the most studied subjects with numerous research and development works both in academia and industry and are increasing over the years (Zainal *et al*. 2022). They have become a support for the energy field and are used to improve energy systems (Molaei and Siavoshi 2021). The number of studies on nanofluids has risen spectacularly since the first time they were brought to the world (Malvandi 2015). For example, from 2021 to this day, 6613 types of research have been done and published on Science Direct (ScienceDirect 2022).

However, using nanofluids in considerable quantities in actual thermal installations remains a challenge (Kaggwa and Carson 2019).

The considerable amount of research that has been conducted on nanofluids has the objective of improving their thermophysical properties, excelling in their synthesis and stabilization so that it is possible to use them in industrial heat exchangers (Said *et al*. 2013). This is a fundamental goal because the thermal properties of nanofluids are affected several parameters such as pH, surfactant concentrations, nanoparticles type, nanoparticle size, temperature, or sonication time (Said *et al*. 2021). Therefore, for these suspensions to be used in industries and prepared in huge quantities, the main element is excellent thermal conductivity and avoiding any modification that can alter the dynamic viscosity. In addition, the preparation must be economically efficient, easily prepared with perfect stability, and contain non-oxidizable particles (Ali and Salam 2020).

The first part of the thesis represents the definitions, the use of nanofluids and the fundamental knowledge about nanofluids, including the different thermophysical parameters that can influence the behavior of a nanofluid. In the second part, the different materials and methods used are presented. In the third part, we will specify the different results obtained using Minitab. Finally, the last part represents the conclusion of the study in which the solid points and results will be highlighted.

# 2. BACKGROUND

## 2.1 Nanotechnology

Nanotechnology is the technology of small things that allows us to create material with atomic-level precision, as shown in (Figure 2.1) (Ismail *et al*. 2018).

The world, as we know, is changing. Accordingly, nanotechnology offers limitless potential for people and society (Schummer 2006). As a result of its ability to be applied to other disciplines, it has become a multidisciplinary collection of fields, including electronics, mechanics, chemistry, biology, and medicine. Research in this discipline is becoming increasingly integrative, similar to most modern sciences. (Porter and Youtie 2009). Nobel Prize-winning physicist Richard P. Feynman introduced the notion of nanotechnology in 1965 and is considered the inventor of nanotechnology, although the idea belongs to Maxwell in 1867 (Ali and Tariq 2014). In a famous lecture delivered in December 1959 at the American Physical Society conference, Richard P. Feynman discussed the concept of nanotechnology (Feynman 1960). In 1974, about 15 years after the term nanotechnology was coined, a scientist from Japan named Norio Taniguchi initially used the term (Bayda *et al*. 2019). He described it as the deformation, consolidation, and separation of materials caused by the manipulation an atom or a molecule (Hulla *et al*. 2015, Keiper 2003).

Nanotechnology is a term that encompasses the study of principles and properties at the nanoscale, or the level of atoms and molecules (Subedi 2014). The prefix Nano-derives from the Greek word "Nanos," meaning "dwarf," and stands for $10^{-9}$ m in the International System of Units (SI) (Boholm 2016). Nanoscience aims to create objects or materials less than 100 nanometers in diameter (Society 2004). These nanomaterials consist of intentionally shaped nanoparticles instead of fine particles from nature or combustion (Morganti 2012). Metals, ceramics, carbons, polymers, and even silicates are nanomaterials with unique properties compared to similar macroscopic-sized materials (Khan *et al*. 2019).

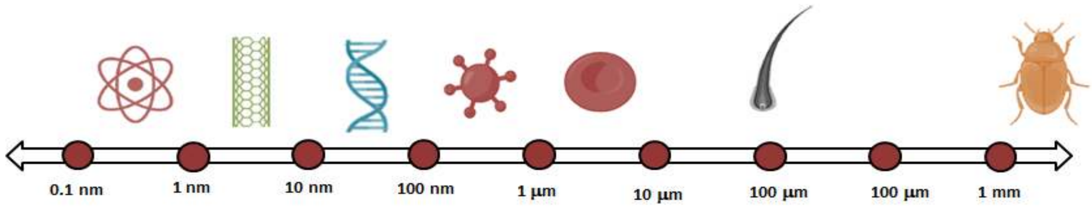


**Figure 2.1** Nanometer to millimeter scale (Serrano *et al*. 2009)

Nanotechnology offers enormous potential for discoveries and applications since the properties of matter can change dramatically at the nanoscale. Atoms are much more accessible at this scale (Roco and Bainbridge 2002). Thus, one in ten million atoms is on the surface of a cubic centimeter ($cm^3$) of matter, while over 80% of atoms are on the surface of a cubic millimeter ($mm^3$). For the same volume, materials in the form of nano-objects (e.g, nanoparticles) have a larger developed surface area (Figure 2.2). Therefore, they are more reactive as chemical reactions occur on the surface. More reactivity, on the other hand, can also mean more utility. At this scale, the surface reactivity of a material determines its properties, which can vary considerably. The boiling point, solubility, or catalytic activity could all be factors. When materials are shrunk to microscopic size, they can have unique properties that they would not have at a larger scale (Anonymous 2021).

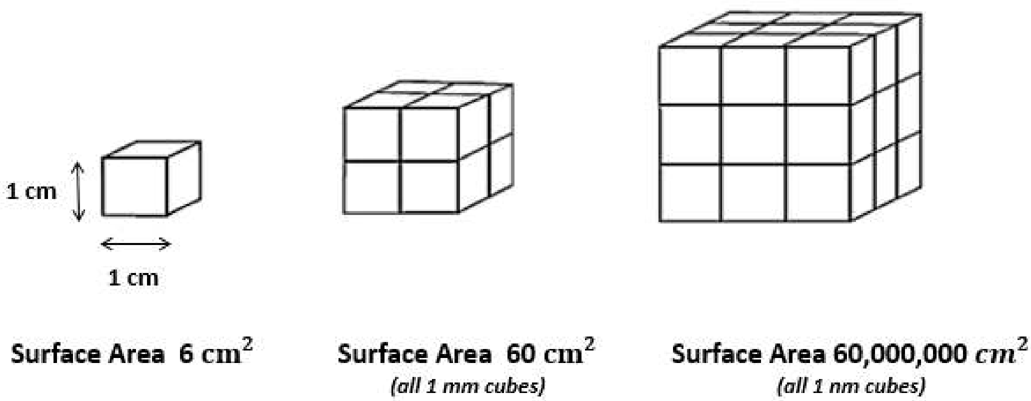


**Figure 2.2** Surface area of nanosized materials (Thaxton *et al*. 2009)

This aspect of nanomaterials is illustrated above (Figure 2.2). Suppose a cube of one centimeter on each side has a surface area of 6 $cm^2$ (providing contact with the outside). We can extend the surface area of a cube to 60 million $cm^2$ by cutting it at the nanoscale. This makes the atoms more accessible and changes the properties of the matter (Thaxton *et al*. 2009).

## 2.2 Nanoparticles

The use of nanotechnology has enabled the production of various materials at the nanoscale. Nanoparticles have always been present in the environment. They can occur naturally during volcanic activity and photosynthesis (Handy *et al*. 2008). Desert dust, for example, can be nanometric in size. Nanoparticles are also created as the by-products of a combustion reaction (Buzea *et al*. 2007). Whenever there is a fire, environmentally harmful nanoparticles are released into the atmosphere, which is exacerbated by vehicle exhaust (Jeevanantham *et al*. 2019). In addition to the various nanoparticles found in nature, some are now produced industrially to perform a specialized function (Stark *et al*. 2015).

Nanoparticles or ultra-fine particles are particles with a dimension less than 100 nanometers (nm) in size, where most atoms are located on the surface. Their total dimension can exceed 100 nm, but their constituent components are of a smaller size or equal to 100 nm (Mathiazhagan and Joseph 2011). They are generally particles bonded by relatively weak forces, such as Van der Waals, electrostatic, or surface tension (He and Alexandridis 2015). Nanoparticles, owing to their minimal size, have a large exchange surface area (specific surface area) per gram. Due to this wide exchange surface area, they exhibit particular physical, chemical, and optical properties when contacting living organisms (Kango *et al*. 2013).

Moreover, they have higher surface reactivity with increasing size and are predicted to have more significant biological activity than larger particles of the same mass. They are known as nano-objects with three external dimensions (Lanone and Boczkowski 2010).

The ability of nanomaterials to be explicitly generated to meet a particular purpose makes them useful in many industries, ranging from healthcare and cosmetics to environmental preservation and air purification (Klaine *et al*. 2008).

### 2.2.1 Types of nanoparticles

Diverse nanoparticles have been used to produce nanofluids and are classified into different types according to their size, morphology (e.g, Figure 2.3), and physical and chemical properties. They can be divided into four categories (Ahmed 2019):

- Metal nanoparticles such as aluminum, copper, iron, and silver.
- Nanoparticles of ceramic oxides include $Al_2O_3$, $CuO$, $TiO_2$, $ZnO$, and $SiO_2$.
- Nanoparticles of metal carbides such as silicon carbide metal.
- Non-metallic nanoparticles such as graphite and carbon nanotube CNT.

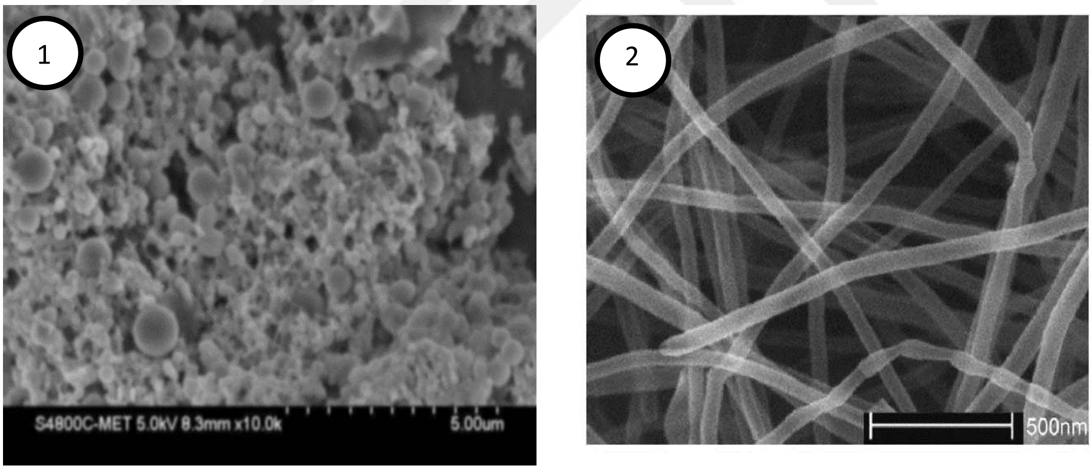


**Figure 2.3** (1) spherical nanoparticles (2) carbon nanotubes (Pande *et al*. 2014, Yuan and Chen 2016)

Furthermore, the most widely utilized base fluids are (Suneetha *et al*. 2022):

- Water,
- Ethylene and propylene glycol,

— Oils and other lubricants,
— Toluene,
— And bio-fluids.

The most available nanoparticles are shaped in:

— Spherical,
— Cylindrical,
— Agglomerate, forms.

The range of volume fractions studied in general is:

— 1%
— 1 - 10%
— >10%

### 2.3 Production of Nanoparticles

Various methods can manufacture nanoparticles, including physical, chemical, and biological approaches (Figure 2.4). In general, it is believed that physical and chemical processes are best suited to produce nanoparticles of uniform size and with long-term stability (Augustine and Hasan 2020).

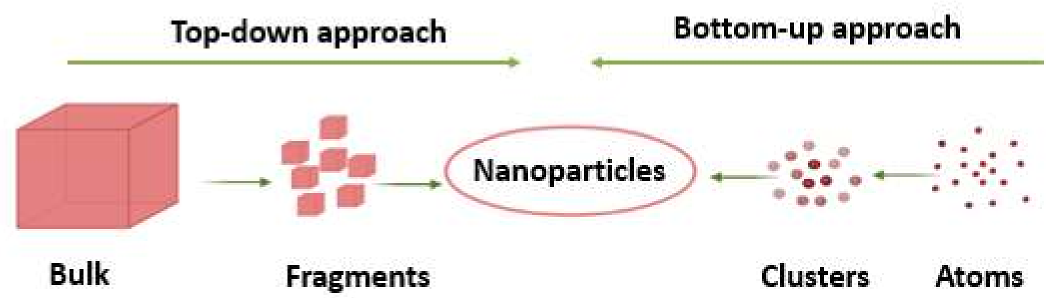


**Figure 2.4** Surface area provided by nanostructured materials (Sakhare *et al*. 2022)

The bottom-up method builds nanoparticles atom by atom or molecule by molecule. There are several ways to make such materials, including chemical synthesis, self-assembly, and assembly by individual positioning. Chemical synthesis of nanoparticles is well known and can produce a significant number of particles, while group by unique positioning of atoms requires insufficient productivity methods of microscopy. Self-assembly is a technique in which atoms form ordered structures at the nanoscale by arranging themselves. Crystal formation from a supersaturated solution is an example of self-assembly that enables mass production (Arnall 2003, Siegel *et al*. 1999).

A prominent structure is gradually reduced to nanoscale dimensions in the top-down technique. Standard methods include etching, precision engineering, lithography, and milling. Many of these techniques are used in clean rooms in the electronics sector, where production and health and safety conditions are controlled (Feigenbaum *et al*. 2004, Society 2004).

In the Top-Down approach, there are physical processing techniques which are:

— Mechanical techniques: etching, cutting, ball milling, grinding

— Lithographic techniques: Photolithography, Electron Beam Lithography

Whereas, in the Bottom-Up approach, there are physical as well as chemical processing techniques which are:

- Physical techniques: Sputtering, Laser ablation, Physical Vapor Deposition (PVD), Plasma Arcing.
- Chemical techniques: chemical vapor deposition (CVD), Self-assembled Monolayer (Sol-gel method, pyrolysis…) (Arole *et al*. 2014).

## 2.4 Nanofluids

Nanofluids are colloidal solutions consisting of particles in nanometer dimension and size suspended in a carrier fluid. Since their unique thermal properties were discovered, this solution has attracted much attention. However, the thermal conductivities of the standard base fluids commonly used for cooling or heating are low (Table 2.1), which limits their heat transfer capabilities. Consequently, the objective is to introduce nanoparticles into basic fluids to increase the heat conductivity of the mixture and improve their thermal performance. The base fluids used to prepare nanofluids include those commonly used for heat transfer, such as DIW, EG, or motor oil. The nanoparticles can be based on oxides, nitrides, metals,carbon or carbides (Saidur *et al*. 2011).

**Table 2.1** Thermal conductivity of base fluids and materials at 20°C (Vajjha *et al*. 2009)

| ELEMENT | THERMAL CONDUCTIVITY λ (W/m. K) |
|---|---|
| Water | 0,599 |
| 50% water + 50% ethylene glycol | 0,41 |
| Ethylene glycol | 0,25 |
| Motor oil | 0,14 |
| Carbon nanotubes | 3000-6000 |
| Diamond | 1000-2600 |
| Copper | 401 |
| Nickel (Metal) | 237 |
| Silicone (non-metallic) | 148 |
| Alumina ($Al_2O_3$) | 40 |

### 2.4.1 Types of nanofluids

Suspensions of nanoparticles in a fluid, known as nanofluids, offer intriguing thermal properties compared to normal base fluids and undeniably improved heat transfer properties. The thermal conductivity of nanofluids can be significantly higher than that of pure liquids. A critical factor in preparing a nanofluid is the choice of appropriate base fluids, as they ensure stability over time and prevent aggregation. The type of nanofluids depends on the materials used, such as nanoparticles and base fluids. However, they are generally classified into Mono nanofluids and Hybrid nanofluids (Bumataria *et al*. 2019).

### 2.4.2 Mono nanofluids

Mono-nanofluids, also known as simple nanofluids, are colloidal suspensions formed by combining a single type of nanoparticle with any base fluid in a one- or two-step method. They were the first nanofluids to improve a suspension's thermal properties. The type of base fluid, size, shape, kind of nanoparticles, and concentration influence their high thermal conductivity. However, mono-nanofluids lack overall hydrothermal features, including increased stability and thermal conductivity. On the other hand, metal nanofluids (e.g, copper nanofluids) have a high thermal conductivity but low stability. Moreover, clogging, fouling, sedimentation and system failures are all conceivable because of their standard strength. These factors make such nanofluids inadequate for applications involving heat transfer (Chandravadhana *et al*. 2020).

### 2.4.3 Hybrid nanofluids

Hybrid nanofluids are a new type of nanofluids developed to solve the problems of stability and low thermal conductivity. They are fluids created by colliding multiple types of nanosized solid particles of two or more different materials into a base fluid. They can optimize heat transfer rates in many applications by providing a higher heat transfer rate than standard nanofluids and reportedly exhibit higher thermal conductivity. Due to its synergistically improved thermal properties and enhanced overall hydrothermal characteristics, it is now a prospective candidate for the management of heat transfer applications (Sarkar *et al*. 2015).

### 2.5 Production Methods of Nanofluids

Nanofluids are most of the time prepared by the one-step method and the two-step method. The first step of the two-step method is the synthesis of nanoparticles, and the second step is the dispersion of nanoparticles in a base fluid. Therefore, the two-step method is advantageous when mass production of nanofluids is considered (Mukherjee and Paria 2013).

#### 2.5.1 One-step method

In the one-step process, the nanoparticles are dispersed directly in a base fluid, and oxidation of the pure metal particles is prevented (Figure 2.5). These nanoparticles are prepared in a single step from powdered nanofibers or nanomaterials such as nanotubes (Babita *et al*. 2016). It is less industrial and can only be applied to specific nanofluids, preventing nanoparticles aggregation and oxidation. One of the environmentally friendly methods for metal nanoparticle production is the laser ablation approach, a unique tool for nanoparticle production. Another one-step process is condensing vapor made of metal in a reactor to produce nanoparticles on a liquid film at a very low pressure of vapor. Its disadvantage is that it is compatible with low vapor pressure liquids and is made in the base liquid, so it can only be produced in small quantities. Another disadvantage of the method is being an incomplete reaction; a portion of the reagent remains in the nanosuspension, causing the purity and properties of the nanofluids to be compromised. On the other hand, the advantage is that the nanoparticles are pure and have a more homogeneous size (Babar *et al*. 2019).

### 2.5.2 Two-step method

The two-step method is commonly used to produce nanofluids. The first step of this method is to generate the nanoparticles, while the second step is to disperse the nanoparticles into a base fluid (Figure 2.5). Rotary stirrers or ultrasounds are often required to break up the agglomerates to ensure good dispersion. Additionally, electrostatic repulsive forces are utilized by charging the surface of the particles by altering the pH to prevent aggregation caused by attractive forces between particles. Using molecules adsorbed or grafted on the surface, we can also use steric repulsive forces. Due to the general industrial production of nanoparticles, this method offers cost advantages and enables the production of nanofluids on a large scale (Babar *et al*. 2019).

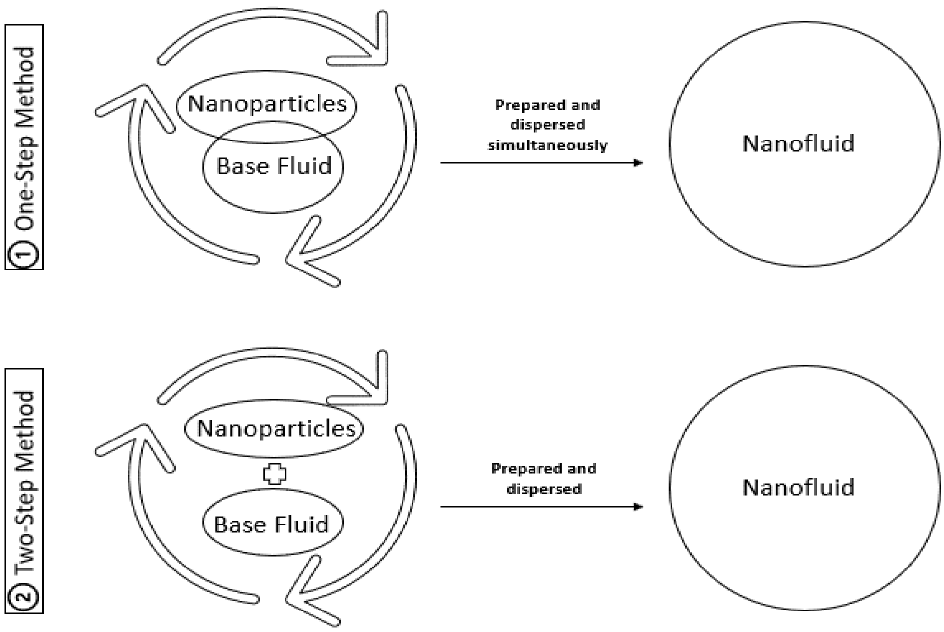


**Figure 2.5** Production methods (1) One-step method (2) Two-step method (Babar *et al*. 2019)

### 2.5.3 Mechanisms of nanofluids stability

The principal factors influencing the stability of the suspensions are particle aggregation and sedimentation. In contrast, the instability of a nanofluid causes mixture inhomogeneity and non-constant thermophysical properties. As a result, reducing the influence of these two factors is required. Furthermore, particle fall velocity can be influenced by various factors regarding sedimentation. Therefore, considering the process of particle sedimentation as one of the variables that destabilize nanofluids is essential for the establishment of a suitable technique of stabilization. A particle is susceptible to three forces when in a fluid (Figure 2.6) (Wei *et al*. 2012).

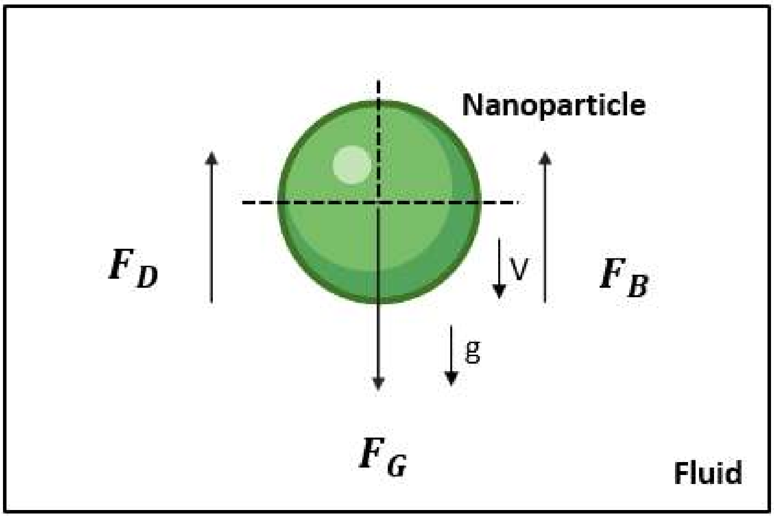


**Figure 2.6** Different forces acting on a nanoparticle immersed in fluid (Wcíslik 2020)

In a suspension, there is three forces that the fluid exerts on each particle: the buoyant force $F_B$ oriented upwards, gravitational force $F_G$ oriented downwards, and drag force $F_D$ oriented upwards, resulting from the fluid's frictional forces on each particle. Newton's second law states that when these forces approach equilibrium:

$$F_D + F_B - F_G = 0 \qquad (1.1)$$

According to Stokes's law the expression for the drag force of a spherical particle of radius r moving at a deficient Reynolds number (Re >>1) and with speed

$U_0$ through a viscous fluid of density ρ and viscosity coefficient μ takes as an expression (Gregersen 2021):

$$F_D = 6\pi\mu\, r U_0 \tag{1.2}$$

Moreover, the apparent weight or apparent gravitational force is the weight of a body as affected by the buoyancy of a fluid in which it is immersed, being the accurate weight minus the weight of the particle and is written:

$$F_G - F_B = (\rho_p - \rho_f) g \frac{4}{3} \pi r^3 \tag{1.3}$$

with $\rho_f$ and $\rho_p$ the mass densities of the fluid and the particle, respectively, and g the acceleration. Finally, the expression of the velocity of the fall of a particle is written according to the previous relations:

$$V = \frac{2}{9} \frac{(\rho_p - \rho_f)}{\mu} g r^2 \tag{1.4}$$

The diameter or radius of a particle, according to Equation 1.4, is the most crucial parameter for sedimentation rate. A nanometric particle settles 1 million times slower than a particle with a diameter on a micrometer, explaining the excellent theoretical stability of nanofluids. Besides the three forces described in equation 1.1, there are other forces such as electrostatic forces, atomic forces (Brownian motion), and thermophoretic forces. These forces enhance the probability of the nanofluid collapsing and the chance of numerous particles aggregating together, thus raising the danger of aggregation. Once agglomerated, the particles have a wider diameter than individual particles, which aids in sedimentation. Therefore, to generate stable suspensions, it is necessary to consider these two factors by employing minuscule particles and reducing the probability of aggregation (Wcíslik 2020).

### 2.5.4 Stability evaluation methods

The stability of nanofluids can be evaluated and determined by using a variety of methods. Observing the suspension with the eyes is one of the most straightforward techniques to assess the stability of a nanofluid. Particle sedimentation produces a visible concentration gradient, especially in opaque suspensions. Furthermore, absorbance measurements can be taken, which are related to the concentration of nanoparticles over time by the Beer-Lambert law. A reduction in absorbance over time implies particle sedimentation. However, extended observation times are required, and no information concerning particle aggregation can be collected (Arora *et al*. 2021).

Centrifugation and sedimentation method

Sedimentation is one of the simplest and fundamental methods to determine the stability of nanofluids. In the presence of external forces, the volume or weight of nanoparticles directly relates to the level of stability for nanofluids. In this case, the nanofluid is stable if the concentration, proportion, and size of the particles moving through it are stable. In another common technique, a specialized camera would monitor the sedimentation in a base fluid at an interval of time. However, using this method for an extended period would be unwise since long observation times would degrade the accuracy. For this reason, centrifugation was developed. This method is perfect for evaluating the nanofluids' stability after centrifugation at a different rpm (Rathodiya *et al*. 2017).

Measurement of zeta potential

The zeta potential (or potential-ζ), also known as the electrokinetic potential, is a fundamental quantitative parameter and a faster technique for evaluating the stability of a suspension. Its unit is commonly represented by the Greek letter (ζ). It represents the potential difference between the stationary base fluid layer affixed to the dispersed particles and the dispersion environment. The model most generally used to describe the electric double-layer structure is the Gouy-Chapman-Stern-Grahame model (Figure 2.7). Moreover, zeta potential reveals the degree of electrostatic repulsions between particles of similar charges. Particles having charges of the same sign reject each other when the zeta potential increases, preventing agglomeration. Values greater than 30 mV or below -30 mV indicate good suspension stability (Figure 2.7) (Table 2.2). The potential difference between these two values, especially at the isoelectric point, is small. It becomes negligible to balance the forces of attraction, resulting in the risk of particle flocculation. Generally, nanofluids with high zeta potential value are considered to have higher stability, whereas nanofluids with lower zeta potential have lower stability (Rathodiya *et al*. 2017).

**Table 2.2** Stability behavior of nanofluids (Kumar *et al*. 2017)

| **Zeta potential (mV)** | **Stability behavior**[1] |
|---|---|
| 0 to ±5 | Fast flocculation or coagulation |
| ±10 to ±30 | Emerging instability |
| ±30 to ±40 | Moderated stability |
| ±40 to ±60 | Good stability |
| >61 | Excellent stability |

There are two ways to increase the zeta potential: adding surfactants or adjusting the pH. A surfactant is a surface-active agent which modifies the surface tension between two surfaces (Cacua *et al*. 2019). Surfactants are amphiphilic[2] molecules, which means they contain two sections with distinct polarities: apolar (dissolves in lipids), and the other is polar (dissolves in water or other hydrophilic solvents), which can be mainly cationic or anionic. Surfactants modify the surface potential of

[1] Rheological behavior of a fluid, i.e. the relationship between ther deformations of the fluid and the applied constraints.
[2] Chemical compounds which have have both nonpolar and polar regions, making them hydrophilic and lipophilic.

the particles, surface tension and wettability of the mixture by interacting at the particle-fluid interface. Since the coagulate molecules form a thermal contact resistance, thermal conductivity can be reduced. On the other hand, Nonionic surfactants aid in dispersion while leaving the zeta potential unaffected.

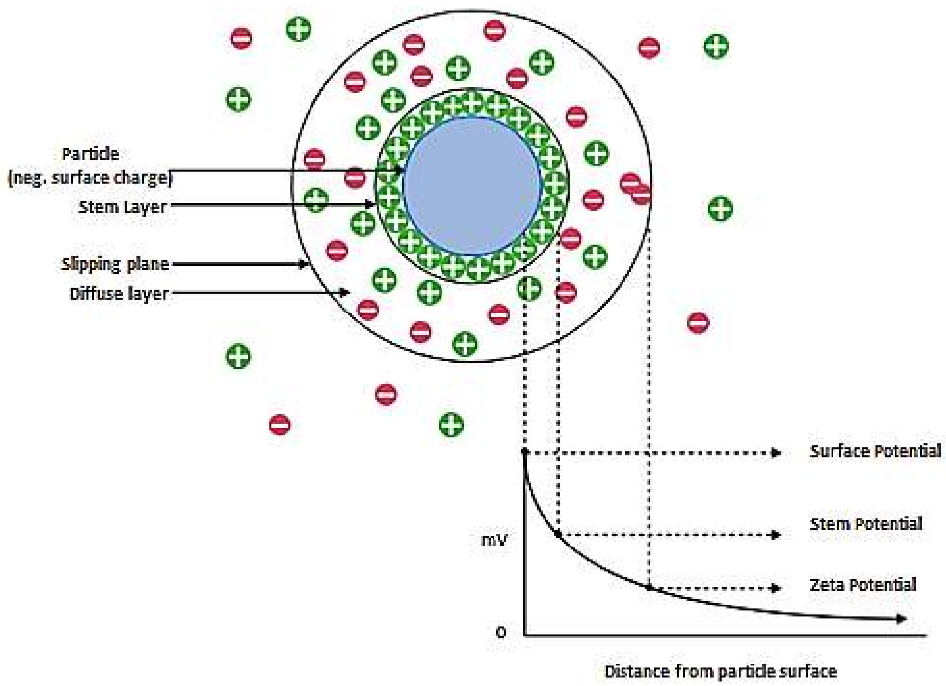


**Figure 2.7** Representation of zeta potential (ξ) (Matusiak and Grządka 2017)

Adjusting the pH increases the concentration of charges present on the particles's surfaces, causing the zeta potential to vary. Figure 2.8 illustrates the zeta potential evolution as a function of pH. Typically, a nanofluid can be stabilized by either adding an acid (reducing the pH to 0) or adding a base (increasing the pH to 14). The isoelectric point is determined by the particle/base fluid pair and is not always at neutral pH. The addition of pH-modifying substances or surfactants, on the other hand, might change thermophysical properties, particularly thermal conductivity.

Consequently, it is crucial to establish the optimal balance between nanofluid stability and property modification for each application. In addition to zeta potential, techniques such as DLS (dynamic light scattering) are commonly utilized to determine the size of nanoparticles in suspension. The hydrodynamic diameter is the actual diameter of the particle plus the layers of solvent, surfactant, or other chemical species that move with it and is represented by this dimension. Thus, it differs from the particle size before suspension. Nonetheless, comparative measurements can be used to analyze the dynamics of particle aggregation and so complete the zeta potential measurements (Huang *et al*. 2009).

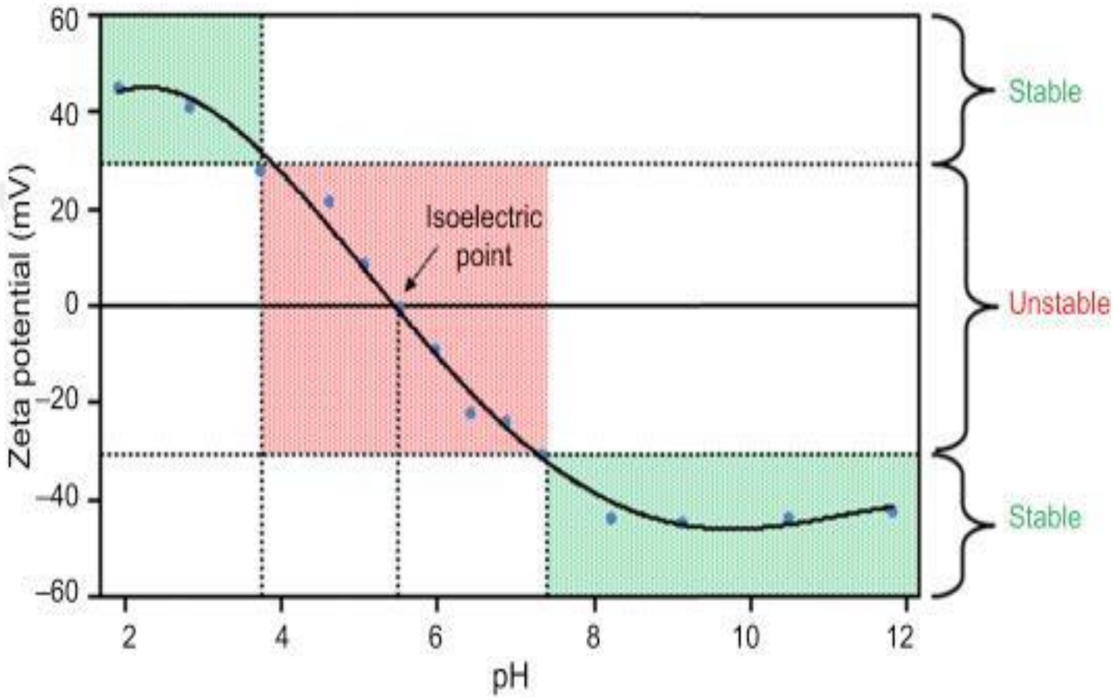


**Figure 2.8** Zeta potential in function of pH (Pate *et al*. 2016)

Measurement of spectral absorbency

Another quantitative and qualitative analysis technique for determining the stability of nanofluids is absorbance spectrometry. This method is founded on a single element used to reconstitute metal analysis in various materials. It is based on the idea that free-moving atoms can absorb light of a specific wavelength for a specific amount of time. Each element uniquely absorbs its wavelength; no other element absorbs it. In the limit of the Beer-Lambert law's proportionality between concentration and absorbance, the intensity of the absorbed radiation is essential when the solution is concentrated. The spectrophotometry UV-visible is particularly interesting in the case of nanoparticles distributed in a basic fluid to show the state of dispersion, the presence or absence of agglomerates of particles depending on the intensity of the absorbed or transmitted light. Indeed, the absorbance of the suspensions is used to identify changes in the concentration and size of the supernatant particles over time (Sharif *et al*. 2017).

Measurement of scanning electron microscopy

SEM (Scanning Electron Microscopy) allows you to see elements at the atomic level, including their sizes, shapes, and distributions. Since the tests are often carried out on samples that have been dried. These approaches do not allow for real-time measurement of nanoparticles in suspension in a basic liquid. Nonetheless, these techniques provide valuable information about nanoparticle structure and the networks they might form. They enable the creation and size of aggregates inside nanofluids to be controlled (Erdman *et al*. 2019).

### 2.5.5 Thermophysical properties of nanofluids

Although nanofluids are considered homogeneous media in many studies, they are two-phase fluids. Therefore, they share some properties with solid-liquid mixtures. The amount of models created to calculate these two values shows that dynamic viscosity and thermal conductivity are the two thermophysical properties of nanofluids that have been investigated the most. In contrast, the density of nanofluids and specific heat has received little attention, especially at the practical level (Rudyak *et al*. 2018).

Thermal conductivity

Thermal conductivity is the capacity of a material to conduct or transfer heat and is the most significant property for improving the performance of a heat transfer fluid.

Many researches have been devoted theoretically and experimentally to estimating the thermal conductivity value. This specific property depends on different parameters, such: temperature, thermal conductivity of base fluids, thermophysical properties of nanoparticles, size and shape of the particles, Brownian motion of particles, and the volume fraction of the particles in suspension. The studies carried out on these parameters are numerous and varied (Gandhi 2007).

Maxwell (1873) studied the thermal conductivity of a nanofluid containing nanoparticles with a form of a sphere. He assumed that the particles have the same diameter and are separated by a sufficient distance to neglect their mutual interactions. In his model, the thermal conductivity value depends only on the thermal conductivities of the fluid and the nanoparticles and the volume fraction of the suspended nanoparticles (Maxwell 1873).

$$k_{eff} = \left[\frac{k_{np}+2k_{bf}+2(k_{np}-k_{bf})\varphi}{k_{np}+2k_{bf}-(k_{np}-k_{bf})\varphi}\right] k_{bf} \tag{1.5}$$

Bruggeman (1935) theorized the heat transfer coefficient of a nanofluid containing spherical nanoparticles with high concentration. He proposed a relationship based on the fluid and nanoparticle thermal conductivities, as well as the volume fraction. For the case of spherical nanoparticles, this model appears to better approach some experimental results (Bruggeman 1935, Wang *et al*. 2008).

$$\varphi\left(\frac{k_{np}-k_{eff}}{k_{np}+2k_{eff}}\right) + \left(1-\varphi\left(\frac{k_{bf}-k_{eff}}{k_{bf}+2k_{eff}}\right)\right) = 0 \tag{1.6}$$

Hamilton and Crosser (1962) modified the formula proposed by Maxwell by introducing the form factor of nanoparticles. For this purpose, the authors used the sphericity coefficient ($\psi$), which is the ratio of the surface area of a sphere with the same volume as nanoparticles to the surface area of a nanoparticle. This relation is valid when ratio of thermal conductivity of nanoparticles to liquid is greater than 100 (Hamilton and Crosser 1962).

$$k_{eff} = \left[\frac{k_{np}+(n-1)k_{bf}-(n-1)\varphi(k_{bf}-k_{np})}{k_{np}+2(n-1)k_{bf}+\varphi(k_{bf}-k_{np})}\right]k_{bf} \quad (1.7)$$

n represent form factor and its expression is written as follows $\boldsymbol{n} = \frac{\mathbf{3}}{\Psi}$

For cylindrical particles n = 6 and for spherical particles n = 3. For spherical particles, Hamilton's model coincides with Maxwell's. As conclusion, the model of Hamilton coincides with the model of Maxwell.

**Table 2.3** Thermal conductivity models of nanofluids (Aybar *et al*. 2015)

| **Researchers** | **Expressions** |
|---|---|
| (Maxwell 1873) | $k_{eff} = \left[\frac{k_{np}+2k_{bf}+2(k_{np}-k_{bf})\varphi}{k_{np}+2k_{bf}-(k_{np}-k_{bf})\varphi}\right]k_{bf}$ |
| (Bruggeman 1935) | $\varphi\left(\frac{k_{np}-k_{eff}}{k_{np}+2k_{eff}}\right)+\left(1-\varphi\left(\frac{k_{bf}-k_{eff}}{k_{bf}+2k_{eff}}\right)\right)=0$ |
| (Hamilton *et al*. 1962) | $k_{eff} = \left[\frac{k_{np}+(n-1)k_{bf}-(n-1)\varphi(k_{bf}-k_{np})}{k_{np}+2(n-1)k_{bf}+\varphi(k_{bf}-k_{np})}\right]k_{bf}$ |
| (Wasp *et al*. 1977) | $k_{eff} = k_{bf}\left[\frac{k_{np}+2k_{bf}-2\varphi(k_{bf}-k_{np})}{k_{np}+2k_{bf}+\varphi(k_{bf}-k_{np})}\right]$ |
| (Xie *et al*. 2002) | $k_{eff} = 1+3\Theta\,\varphi_T+\frac{3\Theta^2{\varphi^2}_T}{1-\Theta\,\varphi_T}$ |
| (Xuan *et al*. 2003) | $\mathrm{k}_{eff} = \frac{k_{np}+2k_{bf}-2(k_{bf}-k_{np})\varphi}{k_{np}+2k_{bf}+(k_{bf}-k_{np})\varphi}k_{bf}+\frac{\rho_p\varphi c_p}{2}\sqrt{\frac{k_BT}{3\pi r_c\mu}}$ |
| (Yu *et al*. 2003) | $k_{nf} = k_{bf}\left[\frac{[2(1-\gamma)+(1+\beta)^3(1+2\gamma)]\gamma}{-(1-\gamma)+(1+\beta)^3(1+2\gamma)}\right]$ |
| (Pak *et al*. 2007) | $k_{eff} = 1+7.47\varphi$ |
| (Avsec *et al*. 2007) | $k_{eff} = k_{bf}\left[\frac{k_{np}+(n-1)k_{bf}-(n-1)\varphi_{nf}(k_{np}-k_{bf})}{k_{np}+(n-1)k_{bf}+\varphi_{nf}(k_{np}-k_{bf})}\right]$ |

Viscosity

The ability of a fluid to flow is defined by its viscosity. Due to its capability of affecting the pressure drop during forced convection, it is known as an essential parameter for practical parameters. The addition of solid particles to a liquid causes a greater or lesser increase in the mixture's viscosity. Again, many factors may be the cause of the observed differences. For identical nanoparticles (type, size) and the same base fluid, the quality of the dispersion and the temperature can significantly affect the viscosity. Therefore, to enable nanofluids in potential applications, the degree of viscosity increases of nanofluids compared to conventional fluids must be carefully studied. A number of theoretical models have been created to anticipate how the dynamic viscosity of suspensions may vary under specific circumstances (Mahbubul *et al*. 2012).

In general, the existing formulas go back to the work of Einstein (1906). Starting from the assumption that energy depends linearly on viscosity, Einstein determined the energy lost around a single particle in a fluid by relating its energy to the work done to move it relative to the fluid. This result determined the expression describing a linear dependence of the viscosity of suspensions in a liquid on concentration (Einstein 1906).

$$\mu_{nf} = \mu_{bf}(1 + 2{,}5\varphi) \qquad (1.8)$$

Einstein (1906)' s formula has been studied experimentally since Einstein development and is regarded as suitable for diluted suspensions composed of spherical particles, specifically for volume concentrations below 1%. It should be mentioned that this model does not consider the impact of particle size and inter-particle interactions (Einstein 1906).

Brinkman (2004) extended Einstein's formula to cover a wide range of volume concentrations. His relation leads to the Einstein relation for low volume fractions.

$$\mu_{nf} = \mu_{bf}(1 - \varphi)^{-2,5} \quad (1.9)$$

These first two relationships do not consider the impacts of Brownian motion or the size of the nanoparticles in suspension (Brinkman 2004).

Batchelor (1977) considered the effect of hydrodynamic contact between two spherical nanoparticles when theoretically studying a nanofluid's dynamic viscosity. He demonstrated that, unlike the "Einstein and Brinkman" relationships, the volume fraction has no linear relationship with the dynamic viscosity of a nanofluid (Batchelor 1977).

$$\mu_{nf} = \mu_{bf}(1 + 2.5\varphi + 6.2\varphi^2) \quad (1.10)$$

**Table 2.4** Viscosity models of nanofluids (Mukesh *et al*. 2012)

| Researchers | Expressions |
|---|---|
| (Einstein 1906) | $\mu_{nf} = \mu_{bf}(1 + 2,5\varphi)$ |
| (Hatschek 1913) | $\mu_{nf} = \mu_{bf}(1 + 4,5\varphi)$ |
| (De Bruijn 1942) | $\mu_{nf} = \mu_{bf}\left(\frac{1}{1 - 2.5\varphi + 1.552\varphi^2}\right)$ |
| (Mooney 1951) | $\mu_{nf} = \mu_{bf}e^{\left(\frac{2,5\varphi}{1-\frac{\varphi}{\varphi_m}}\right)}$ |
| (Batchelor 1977) | $\mu_{nf} = \mu_{bf}(1 + 2.5\varphi + 6.2\varphi^2)$ |
| (Krieger *et al*. 2000) | $\frac{\mu_{nf}}{\mu_{bf}} = \left[1 - \frac{\varphi}{\varphi_m}\right]^{-[\mu]\varphi_m}$ |
| (Brinkman 2004) | $\mu_{nf} = \mu_{bf}(1 - \varphi)^{-2,5}$ |
| (Wang *et al*. 2007) | $\mu_{nf} = \mu_{bf}(1 + 7,3\varphi + 123\varphi^2)$ |

Specific Heat

According to the literature, viscosity and thermal conductivity have been extensively studied and described. However, it is not the case for the specific heat, which is rarely investigated when it comes to experiments. The specific heat capacity, often known as mass heat capacity, is measured in J/Kg. K and indicates the specific heat capacity unit mass of a material or homogeneous system. It is the amount of heat necessary to increase the temperature of the unit of material by one Kelvin during the transition under consideration. This specific quantity is linked to the energy equation and must be determined carefully. Two well-known models have been most frequently used in all studies performed on the specific heat of nanofluids (Rudyak *et al*. 2018).

Model (1) of Pak *et al*. (2007) connects the specific heat of nanofluids collectively to all the components combined based on the law of mixtures (Pak *et al*. 2007):

$$C_{p,nf} = \varphi C_p + (1-\varphi) C_{p,bf} \tag{1.11}$$

Model (2) of Xuan and Roetzel (2000), based on the theory that the nanoparticles and liquid phases are in thermal equilibrium, the specific heat capacity may be calculated using the following formula (Xuan and Roetzel 2000).

$$C_{p,nf} = \varphi(\rho C_p)_p + (1-\varphi)\left(\rho C_p\right)f \mid \varphi\rho_p + (1-\varphi)\rho_f \tag{1.12}$$

with, $C_p$: specific heat, φ: particle volume concentration, and *ρ:* density.

**2.5.6 Usage areas of nanofluids**

Nanofluids are an attractive solution in many applications with their exceptional thermal properties. Many thermal systems can benefit from nanofluids to increase heat transfer and efficiency. Depending on their intended use, nanofluids can be tribological nanofluids, heat transfer nanofluids, chemical nanofluids, surfactant nanofluids, environmental nanofluids, pharmaceutical nanofluids, and medical nanofluids. This section provides some examples of all the applications (Figure 2.9).


**Figure 2.9** Usage areas of nanofluids

Nanofluids have had many applications in industry, which has prompted several experimental investigations. Dispersions of nanoparticles already find many fields of application for their magnetic properties (magnetic bearings with Ferrofluids, contrast agents in medical imaging), their electrical properties (electro-wetting conductive deposition for liquid lenses), and cell marking or counterfeiting with functionalized nanoparticles. In thermal applications, nanofluids can be used wherever large heat flows must be evacuated by closed liquid loops: cooling of electrical and electronic components, radars, or automobiles. Nanofluids could provide an interesting solution for cooling mirrors, targets, and filters of high-power lasers and X-rays. The very small size of nanoparticles and the lubricating properties of some of them would allow their use as fluids for transmissions, gearboxes, and thermal engine oils. In dysphasics, by managing to improve their performance, we can envisage improved air conditioning and refrigeration systems, as well as the use of more efficient heat pipes for space applications and electronic cooling. It is also envisaged to use the increase of the critical flow for the emergency injection circuits of pressurized water nuclear plants (Wei *et al*. 2012).

## 3. LITERATURE REVIEW

Many studies have focused on developing the different properties of nanofluids to enhance heat transfer. In comparison to typical heat transfer fluids, metals like silver, copper, and iron, as well as nonmetals like aluminum oxide ($Al_2O_3$), copper oxide (CuO), titanium dioxide ($TiO_2$), zinc oxide (ZnO), SiC, and carbon nanotubes (graphene), have higher thermal conductivities. Considering the importance of thermal conductivity and viscosity for heat transport, many studies in the literature focus on these two properties. These studies can be separated into three categories depending on the type of nanoparticles used:

### 3.1 Metallic Nanoparticles Nanofluids

The term "nanofluid" was first used by Choi (1995) in 1995 and defined as colloidal solutions obtained by dispersing solid particles of nanometric size ($10^{-9}$ m) in a base fluid (Choi 1995). They proposed using metallic nanoparticles as part of heat transfer fluids to increase their thermal conductivity and improve heat transfer. Their idea stems from the observation that fluids commonly used for heat transfer have very low thermal conductivities, 10 to 1000 times lower than solids and especially metals, as shown in Table 2.1.

Li *et al*. (2002) presented a study showing that thermal conductivity increases with volume fraction. Thermal conductivity ratio of Cu/water nanofluid to base fluid increases 75% and 45% for Cu/oil nanofluid when the values of volume fraction of nanoparticles varies between 2.5% to 7.5%.

Theoretically, Xuan *et al*. (2003) used the model Hamilton and Crosser to study the thermal conductivity of the previous nanofluids and obtained results close to the experimental results.

Eastman *et al*. (1997) in 1997, observed a 45% improvement in thermal conductivity by adding less than 0.055% by volume of 35nm copper nanoparticles in oil (Eastman 1997). In 2001 they found a 40% improvement in thermal conductivity for the Cu/ethylene glycol nanofluid containing less than 0.3% by volume of nanoparticles smaller than 10nm.

Patel *et al*. (2010) studied the nanofluid Au-thionate/toluene at a temperature of 60°C and obtained an 8.8% improvement in thermal conductivity for a nanoparticle volume fraction $\varphi$=0.011%. For a solution of Au-citrate/water, they obtained an 8.3% improvement in thermal conductivity for $\varphi$= 0.00026; on the other hand, for a solution of (Ag-citrate/water) they obtained an improvement of 4.5% for $\varphi$=0.001%. For the Ag/water nanofluid, the obtained thermal conductivity is lower than that of the gold/water nanofluid, although silver is a better thermal conductor than gold. This apparent improvement in thermal conductivity when using gold nanoparticles compared to silver particles could be attributed to the size of the gold nanoparticles, which are 3 to 8 times smaller than silver.

P. Kumar *et al*. (2019) found an increase in thermal conductivity of 20% for $\varphi$= 0.00013% gold in water.

### 3.2 Metal Oxide Nanoparticles Nanofluids

In 1993, Masuda *et al*. published the first research on aluminum oxide and thermal conductivity enhancement. Nanoparticles $Al_2O_3$ were dispersed in water with a nanoparticle size of 13nm in diameter. They found a 30% increase in heat conductivity for a fraction $\varphi$ = 4.3 %. Another research published by Eastman *et al*. (2001) discovered a 30% increase in thermal conductivity of nanoparticles $Al_2O_3$ of size 33nm suspended in water for a volume percentage of 5% (Eastman *et al*. 2001, Masuda *et al*. 1993).

Wang *et al*. (1999) numerically studied the natural convection in a cavity of two dimensions for various Grashof numbers and volume concentrations. They obtained a thermal conductivity refinement of 40% for $Al_2O_3$/ethylene glycol of 8% volume fraction and 14% for $Al_2O_3$ for a volume fraction of 4.5%.

Choi *et al*. (1999) studied the thermal conductivity of nanofluids. They employed $Al_2O_3$ and CuO. These particles are dispersed into ethylene glycol and pure water. They achieved a 10% improvement in thermal conductivity for the $Al_2O_3$/water nanofluid with a volume fraction of 4.3% and a 20% enhancement for $Al_2O_3$ in ethylene glycol for $\varphi = 5\%$ volume. The latter results differ significantly from those reported by earlier scientists, despite the fact that they used nanoparticles of the same size.

Das *et al*. (2003) were the first to study the influence of temperature on the increase of thermal conductivity of nanofluids in 2003. They found an increase in thermal conductivity when the temperature varies from 21°C to 51°C for both nanofluids $Al_2O_3$/water and CuO/water. The values of particle diameters are 38.4nm for $Al_2O_3$ and 28.6nm for CuO. The experimental results show that the thermal conductivity increases linearly with increasing temperature and depends on the volume fraction of nanoparticles in water.

Choi *et al*. (1999) measured the thermal conductivity of nanofluid using CuO nanoparticles of diameter 18.6 nm in water or ethylene glycol. They found that the thermal conductivity of the nanofluid used increases linearly as a function of the volume fraction of nanoparticles.

Li *et al*. (2006), also conducted experiments with CuO nanoparticles of 29 nm diameter in pure water with a volume fraction of 10% and a temperature of 34.7°C. They found a 52% improvement in the thermal conductivity of the nanofluid. They proposed the case of $Al_2O_3$ nanoparticles.

Pak *et al*. (2007) improved thermal conductivity by 10.7% % for a volume fraction of 4.35% by dispersing 27 nm nanoparticles in water. This value is inferior than the 32% obtained for the $Al_2O_3$/water nanofluid with the same the nanoparticles concentration.

Murshed *et al*. (2006) evaluated the heat transfer coefficients of spherical and cylindrical $TiO_2$ nanoparticles. They discovered that a nanofluid containing 15 nm spherical nanoparticles improves heat conductivity somewhat.

### 3.3 Non-metallic Nanoparticles Nanofluids

Wen *et al*. (2004) measured the enhancement of (MWCNTs) suspended in water as a function of temperature and concentration. They found that below 30°C, the thermal conductivity of the nanofluid increases linearly with increasing temperature. The maximum improvement obtained is 31% for $\varphi = 0.84\%$ and at the temperature of 60°C.

Ding *et al*. (2006) conducted the same experiment and found an improvement of 80% for $\varphi = 1\%$ and at a temperature of 30°C.

Assael *et al*. (2004) obtained 38% for a volume fraction of 0.6% of nanotubes in water or 150% for a concentration of 1% of nanotubes in ethylene glycol.

# 4. MATERIALS AND METHODS

## 4.1 General

In this study, nanofluids in which deionized water is the main fluid were prepared using silver oxide nanoparticles. Taguchi L9 experimental design was used in the study, and optimum nanofluid was determined with the help of Minitab 15 software. Taguchi experimental design is given in Table 4.1. Three different volumetric ratios, 0.01%, 0.05%, and 0.1%, were used in the experiments. These volumetric ratios correspond to mass ratios of 0.1164%, 0.5828% and 0.1637%, respectively. 150 mL of nanofluid was prepared for each experimental run. Nanofluids were prepared at three different mixing times, 60, 90, and 120 minutes. Another factor used for the stabilization that helps nanofluids to remain stable is surfactants. In the study, Tergitol NP-10 was used as a surfactant, 0.5 g for each test condition.

**Table 4.1** Specification of the sample $Ag_2O$/water nanofluids

| Experiment No | Volumetric Ratio (v/v) | Ultrasonic mixing Time (min) |
|---|---|---|
| 1 | 0.01 | 60 |
| 2 | 0.01 | 90 |
| 3 | 0.01 | 120 |
| 4 | 0.05 | 60 |
| 5 | 0.05 | 90 |
| 6 | 0.05 | 120 |
| 7 | 0.1 | 60 |
| 8 | 0.1 | 90 |
| 9 | 0.1 | 120 |

## 4.2 Chemicals Used in the Experiment: Source and Purity

**Table 4.2** Chemicals used in the experiment

| Chemical Name | Formula | Supplier | Purity Rate (%) |
|---|---|---|---|
| Silver oxide nanoparticles | $Ag_2O$ | Sigma Aldrich | 99.99 |
| Sodium Hydroxide | NaOH | Sigma Aldrich | >99 |
| Hydrochloric acid | HCl | Sigma Aldrich | 37 |
| Tergitol NP-10 | $(C_2H_4O)n$ . $C_{15}H_{24}O$ | Sigma Aldrich | 99.99 |
| Deionized water | $H_2O$ | Çankırı Karatekin University | 99.99 |

## 4.3 Synthesis of Silver/Water Nanofluids

As stated in the second chapter, there are two ways for preparing nanofluids: one-step and two-step. This study was employed with the two-step method. The phases of preparation for the two-step approach are as follows:

— Silver nanoparticles were weighed in accordance with the specified volumetric ratios and made up to 150 mL of deionized water,

— By using 0.01 M NaOH and 0.01 M HCl solution, pH adjustment was made at the specified value for each experiment,

— The prepared mixture was mixed in a magnetic stirrer at 1000 rpm for 30 minutes,

— The mixture, which was stirred for 30 minutes in the magnetic stirrer, was mixed in the ultrasonic stirrer for the time specified for each sample shown in Table 4.1. Figure 4.1 (1) shows the ultrasonic mixer used in the study.

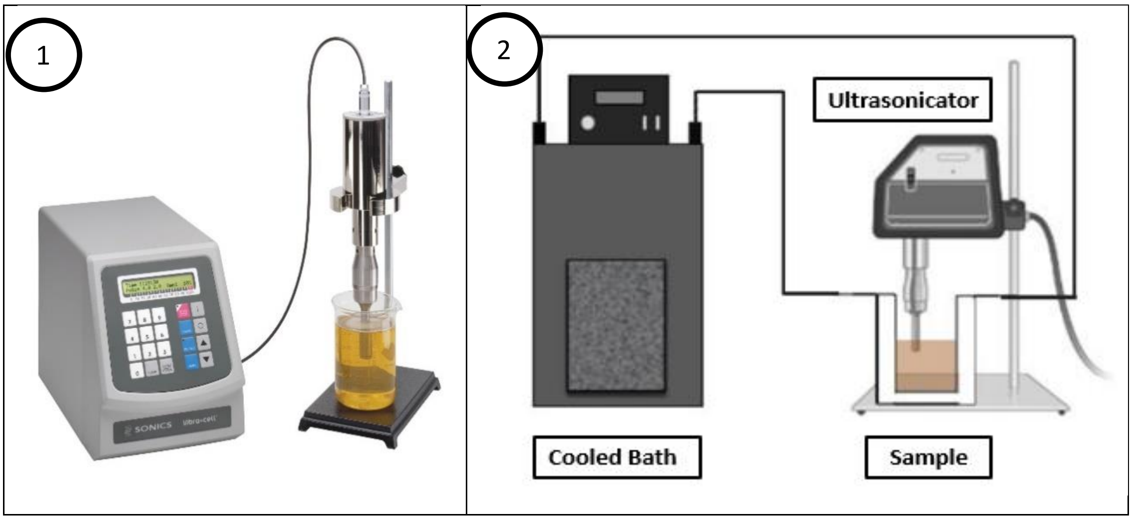


**Figure 4.1** (1) Ultrasonicator device (2) Experimental setup for ultrasonication

Ultrasonic mixing was done with 8/2 pulses at 400 W power. As the temperature rise during mixing is high and high temperatures affect the stability of the nanofluid, a cooling jacket was formed by using a circulating water bath, as seen in Figure 4.1 (2). Moreover, the temperatures of the nanofluids were kept at room temperature.

As previously stated, viscosity, thermal conductivity, zeta potential, and particle sizes were evaluated to identify the physical properties of the generated nanofluids.

In addition, as soon as the nanofluids were prepared, they were dropped on a glass surface and dried, and their images were taken under an optical microscope. All physical properties were measured at room temperature.

### 4.4 Structural Characterization of Silver-oxide/Water Nanofluids

For the characterization of silver/water nanofluids, a light optical microscope was used. An initial drop of nanofluid was placed on a microscope slide in the center. Afterwards, the slide was viewed and photographed under the microscope.


**Figure 4.2** Optical microscope (Microscopio De Luz Transmitida 2022)

## 4.5 Thermophysical Properties of Nanofluids: Measurement Techniques and Experimental Protocols

### 4.5.1 Thermal conductivity

The literature has adopted various methods for measuring thermal conductivity of liquids, and some of these techniques have also been applied to actual nanofluids. The experimental setup for measuring thermal conductivity is shown in Figure 4.3 (2). Transient short-wire approach was used to examine the different thermal conductivities of $Ag_2O$/water nanofluids using a KD2 pro thermal properties analyzer (Decagon devices, INC., USA). To control and measure the conduction in the probe, the instrument was equipped with a 60 mm length and 1.3 mm diameter probe, a thermoresistor, and a microprocessor. Its accuracy was specified as ± 5%. The sample was covered in parafilm to prevent air penetration and ensure reliable results. After temperature equilibrium was achieved, the sample was held for another

30 minutes. Ten measurements were made for each sample, and an average was determined.

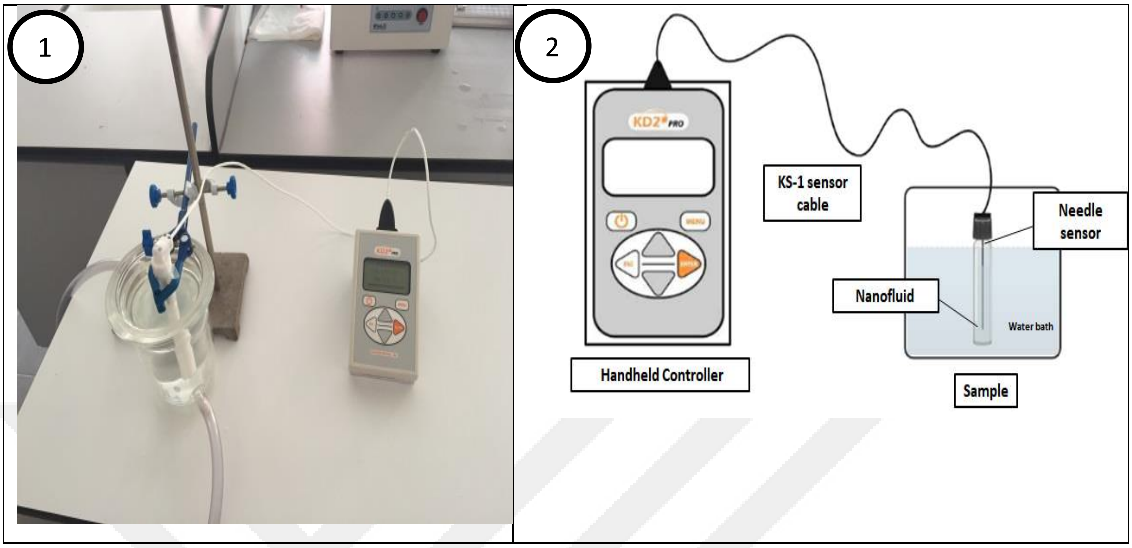


**Figure 4.3** (1) Thermal conductivity meter (2) Experimental setup for thermal conductivity measurement

In order to protect the KD2 Pro thermal conductivity measuring device from temperature changes during thermal conductivity measurements, a water bath was used during the measurements, as shown in Figure 4.3 (2).

### 4.5.2 Viscosity

For the measurement of viscosity data, a Vibro-viscometer SV-10/SV-100 with which a wide range of measurements can be made continuously, was used (Figure 4.4). The ranges are for SV-10: 0.3-10000 mPa.s and for SV-100: 1000- 100000 mPa.s. The instrument in question measures viscosity by detecting the electric driving current necessary to resonate the two sensor plates at a constant frequency of 30Hz and amplitude of less than 1mm and has an accuracy of ± 1%. For each sample, three different values of viscosity and temperature were noted. The viscosity was measured at ambient pressure and temperature between 10°C and 40°C. The suspension was deposited into the glass container of the viscometer with the help of a

Pasteur pipette while avoiding the air bubbles. Before any of these measurements were made, the viscometer was carefully calibrated manually.

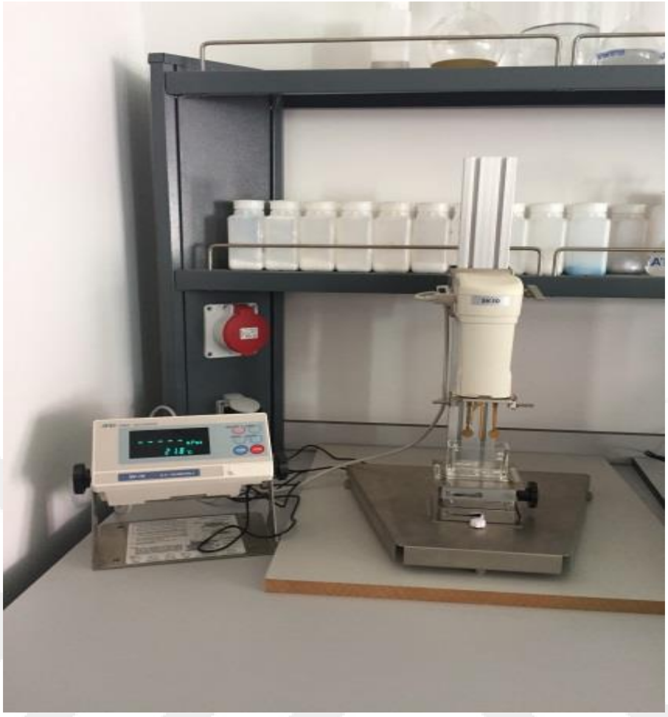

**Figure 4.4** Viscometer device

### 4.5.3 Zeta potential and Zeta size

To determine the stability of the nanofluids, Malvern Zetasizer Nano ZS was used, which uses a 632 nm HeNe laser with a 173°C detection angle to perform zeta potential and zeta size measurements. It is shown in Figure 4.5. With the described instrument, particle size, the zeta potential, and the molecular weight of the suspension can be measured using the dynamic light scattering techniques in a single unit. A sample is loaded with the nanofluid into a disposable folded capillary cell in the measurement. The cells are equipped with two conductive electrodes on the outside that contact the applied voltage and fold back to touch the liquid sample.

**Figure 4.5** Zetasizer device

# 5. RESULTS AND DISCUSSION

## 5.1 Synthesis of Silver-oxide/water Nanofluids: Results and Discussion

As mentioned before, nanofluids were prepared using the two-step nanofluid method. The images of the nanofluids prepared in accordance with the parameter in the Taguchi L9 experimental design in Table 4.1 are given in Figure 5.1. One of the methods of determining the stability of nanofluids is photography. In the photographs, it is seen that denser nanofluids are obtained as the solid amount increases. The volumetric ratios of all three nanofluids 1,2, and 3 are 0.01%. However, the ultrasonic mixing time varies to 60, 90, and 120 minutes, respectively. Although the solid ratios are the same, as can be seen from the images, nanofluids number 1 and three are more transparent than fluid number 2, and nanofluid number 1 appears more transparent than nanofluid number 3. Nanofluids 4,5 and 6 have 0.05% volumetric ratios and ultrasonic mixing times of 60, 90, and 120 minutes, respectively. Here, nanofluid 6 appears to be more stable compared to nanofluids 4 and 5. The ultrasonic mixing time of nanofluid 6 is 120 min. Nanofluids with test numbers 7, 8, and 9 have a volume ratio of 0.1% and a mixing time of 60, 90 and 120 minutes, respectively. While nanofluids 7 and 8 appear to be more stable than nanofluid 9, it is seen that nanofluid 8 is the most stable one. Ultrasonic mixing times of nanofluids have an optimum value, and collapse and instabilities are observed below and above this value. Therefore, it can be said that the 90 min ultrasonic mixing time with the photographing method in the silver oxide nanofluids obtained in this study is the most appropriate time.

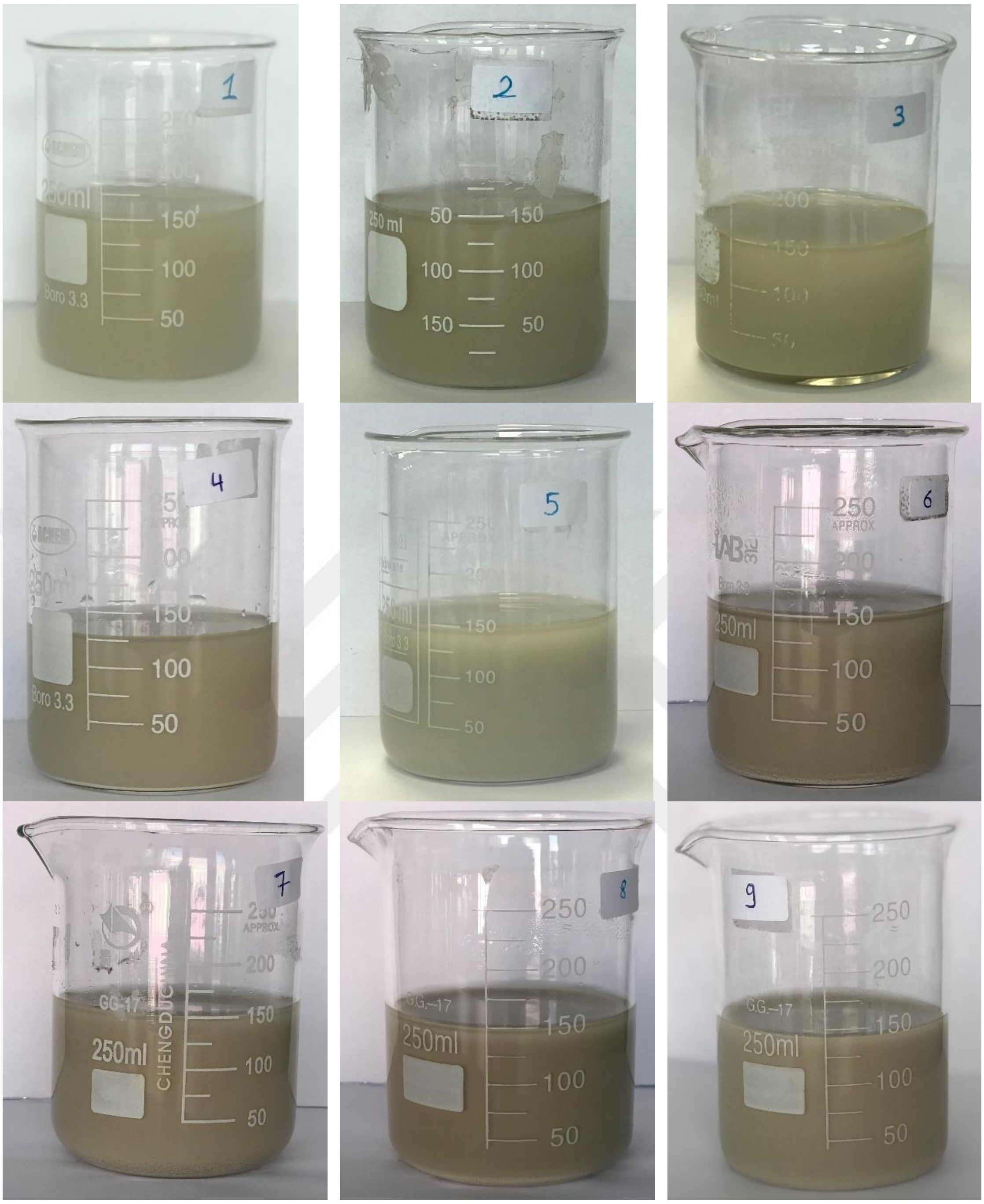


**Figure 5.1** Silver-oxide/water nanofluids samples

### 5.2 Optical Microscope images of Silver-oxide/water Nanofluids

As mentioned before, optical microscope images of nanofluids were taken. Figure 5.2 shows the images taken by drying a few drops of nanofluids on a glass surface.

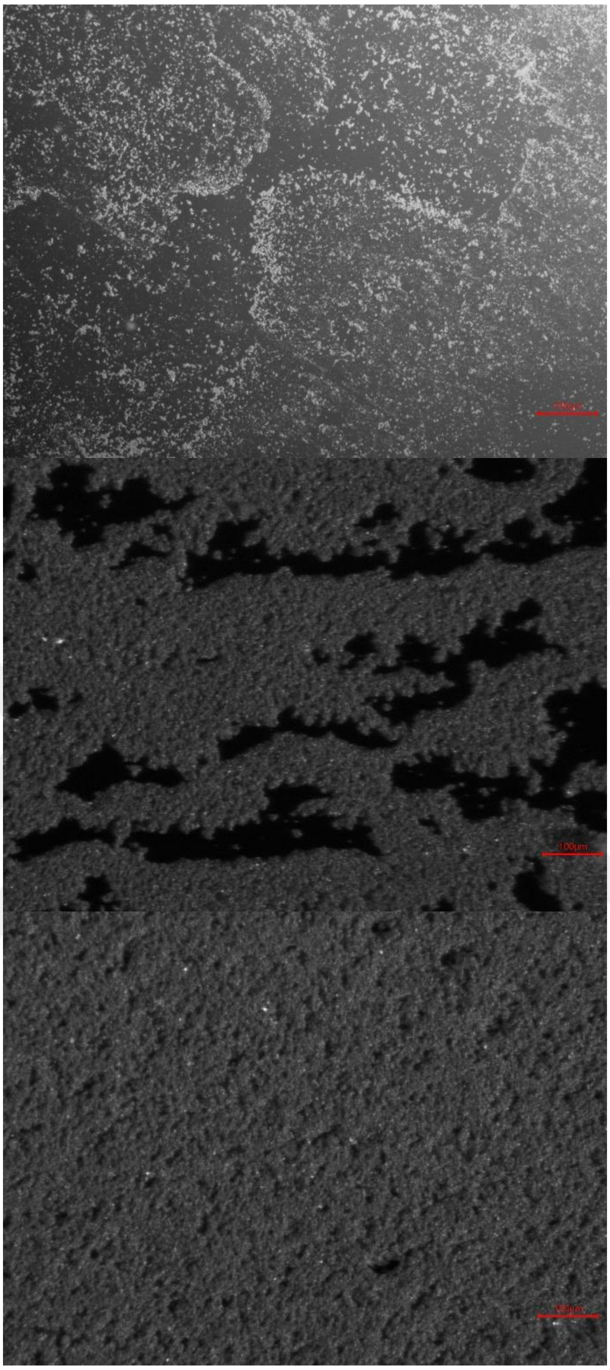

**Figure 5.2** Optical microscope images of $Ag_2O$/water nanofluids

### 5.3 Thermophysical Properties of Nanofluids: Results and Discussion

#### 5.3.1 Thermal conductivity

The thermal conductivities were determined with the help of the KD2 pro device, as we mentioned before. Each sample was measured 15 times, and the average was taken to determine the thermal conductivity. Firstly, deionized water thermal conductivities were measured at different temperatures, then compared to literature values before measuring nanofluids. Experimentally obtained thermal conductivities and the thermal conductivity values from the literature on deionized water are given in Figure 5.3. Measured values are below 5%, which is the error range of the device.

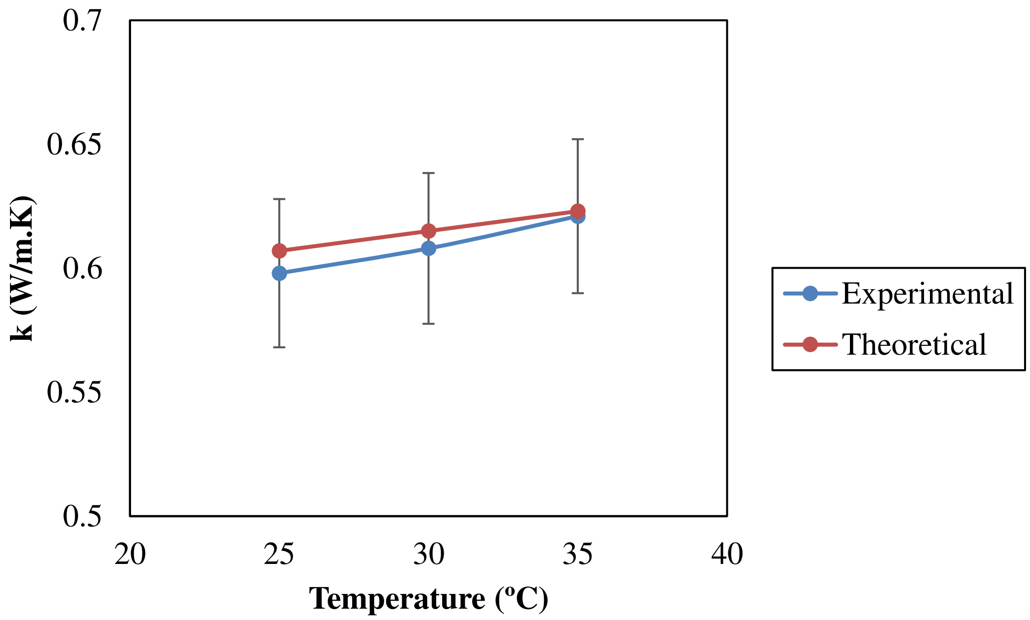


**Figure 5.3** Experimental and theoretical heat transfer coefficients of deionized water

In Figure 5.4, the effect of ultrasonic mixing times on the thermal conductivities is shown graphically. When the thermal conductivities were compared according to the ultrasonic mixing time for nanofluids 1,2, and 3 with a volumetric ratio of 0.01%, the highest thermal conductivity value was obtained at 120 min mixing time. However, an interpretation that the thermal conductivities increase as the ultrasonic mixing speed increases is incorrect because the thermal conductivity at the mixing time of 60 minutes is greater than the value of the thermal conductivity obtained at the mixing time of 90 minutes.

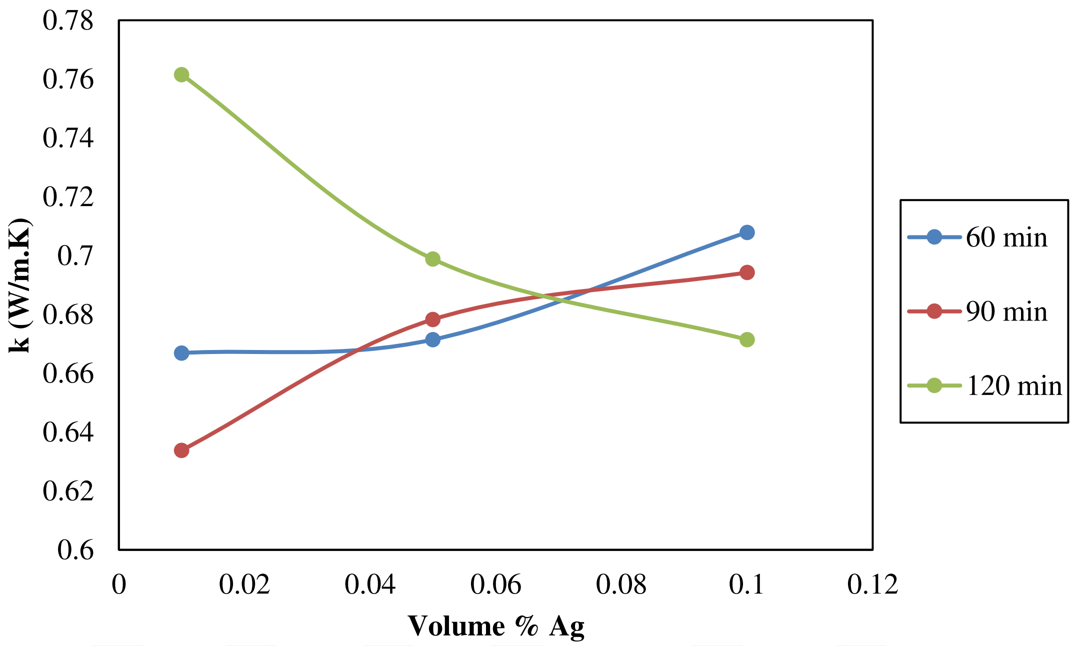


**Figure 5.4** Thermal conductivity ratio of $Ag_2O$/water nanofluid as a function of volume concentration

In experiments 4,5 and 6 with a 0.05% volumetric ratio, the thermal conductivity value increased as the ultrasonic mixing time increased. For a 0.05% volumetric ratio, the highest thermal conductivity was obtained at 120 min ultrasonic mixing time.

In experiments numbered 7, 8, and 9 with a 0.1% volumetric ratio, the thermal conductivity values decreased as the ultrasonic mixing time was increased. The highest thermal conductivity value was obtained at 60 min ultrasonic mixing time.

In general, thermal conductivity values increase as the solid ratio increases. For example, the thermal conductivity value of deionized water at room temperature is given as 0.607W/m.K in the literature (Dinçer *et al*. 2015).

As expected, the thermal conductivities of the nanofluids are higher than thermal conductivities of deionized water for each ratio.

### 5.3.2 Viscosity

As mentioned earlier, viscosities were determined using a viscometer. First, the viscosity values of the 99% pure ethylene glycol solution in the temperature range of 20-40 °C were measured with a viscometer and compared with the literature values in Figure 5.5. The device has an error range of 1%. Therefore, the measured values are within the error range of the device.

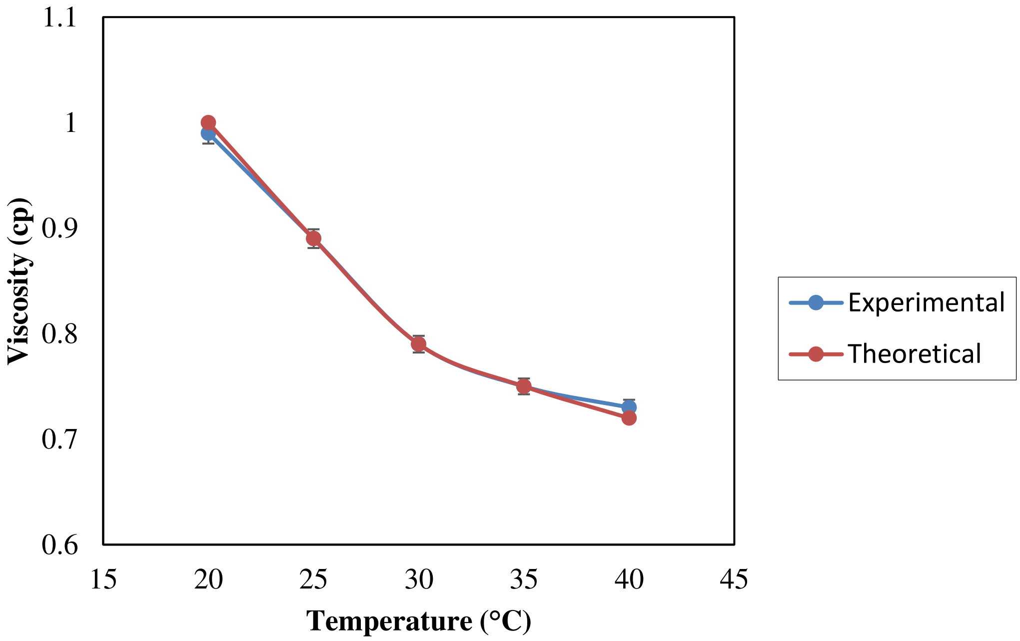


**Figure 5.5** Experimental and Theoretical viscosity values

Figure 5.6 shows the variation of viscosity values with ultrasonic mixing times. When the graph is examined, it is seen that the viscosity values increase as the volume % solid ratio increases. When each solid ratio is considered separately, it can be said that increasing the ultrasonic mixing times causes a linear increase in the viscosity values.

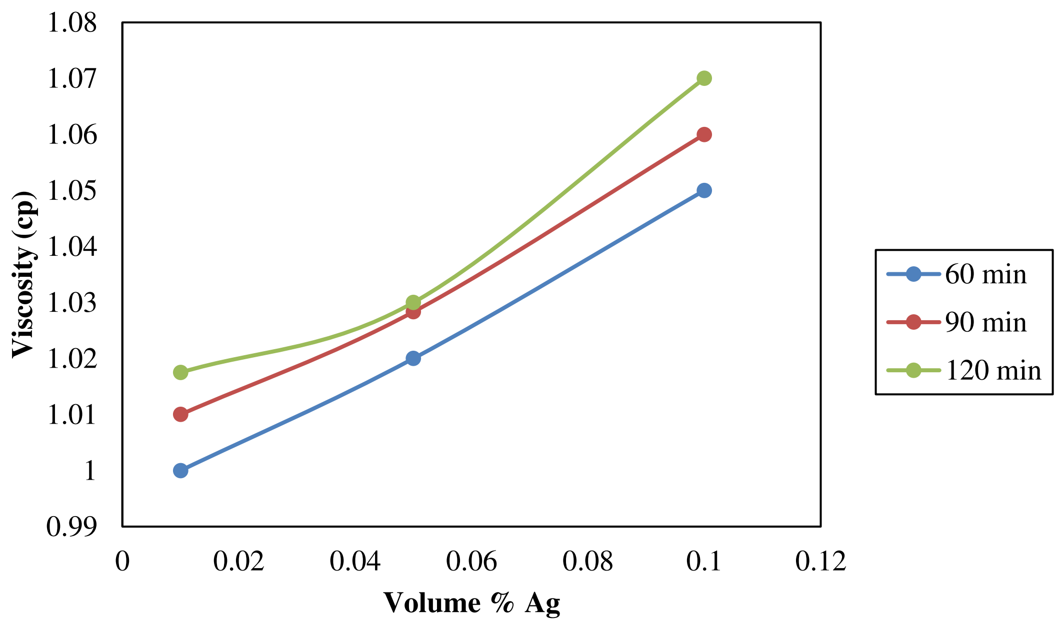


**Figure 5.6** Viscosity of $Ag_2O$/water nanofluid as a function of volume concentration

The viscosity of nanofluids gives very effective information about their stability. In general, the viscosity values of nanofluids should be higher than the viscosity of their base fluids. In this study, the viscosity values for all solid ratios were higher than the viscosity value of pure water at room temperature.

The increase in viscosities with the increase of ultrasonic mixing times shows that the amount of suspended nanoparticles increases as the mixing time increases.

### 5.3.3 Zeta potential

The zeta potential value is one of the most important parameters that give information about the stability of a nanofluid. As mentioned before, the zeta potential value of a stable nanofluid should be between ±30 - ±40. Figure 5.7 shows the zeta potential values of silver oxide nanofluids.

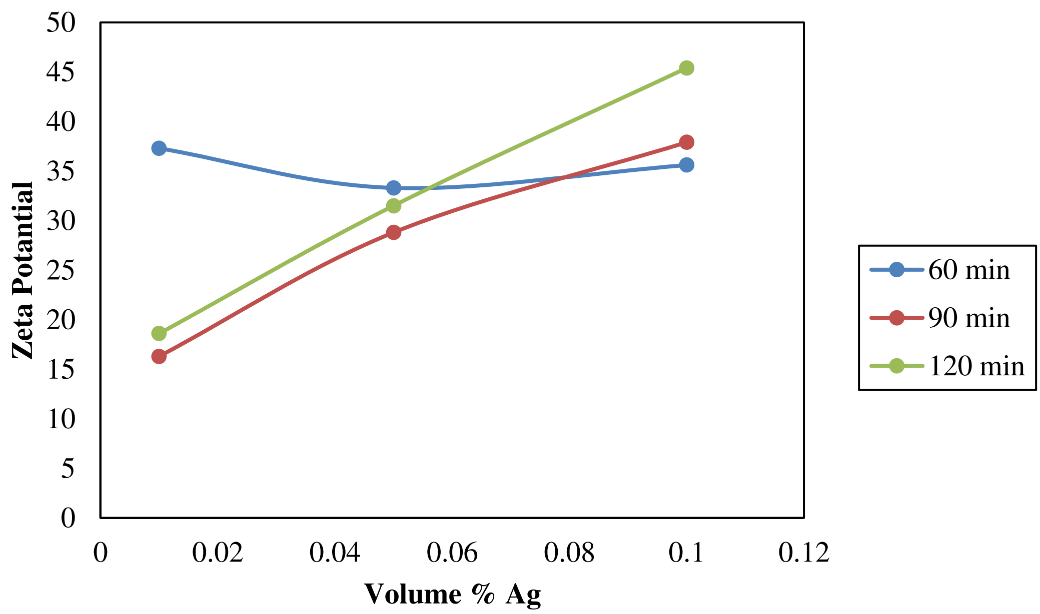


**Figure 5.7** Zeta potential of $Ag_2O$/water nanofluid as a function of volume concentration

When the effect of ultrasonic mixing speed on the zeta potential is examined, it is seen that as the ultrasonic mixing speed is increased, the zeta potential values get closer to the 30-40 range. In other words, as the ultrasonic mixing speed is increased, more stable nanofluids with better zeta potential values are obtained. The 120 min ultrasonic mixing time is the mixing time with the best results for all three volumetric ratios.

When the effect of the volumetric solid ratio on the zeta potential is examined, it is seen that as the solid ratio increases, the number of suspended particles increases, and therefore the zeta potential also increases.

### 5.3.4 Optimization of nanofluid parameters

Using nanofluid preparation parameters experimental design approach was used to find optimum parameter levels. Main effects plots, contour plots were drawn, and linear regression models were calculated for each of the outputs namely, viscosity, thermal conductivity, and zeta potential.

Main effects plots were drawn using volumetric solid ratio and ultrasonic prop time parameters was given in Figure 5.8 for viscosity.

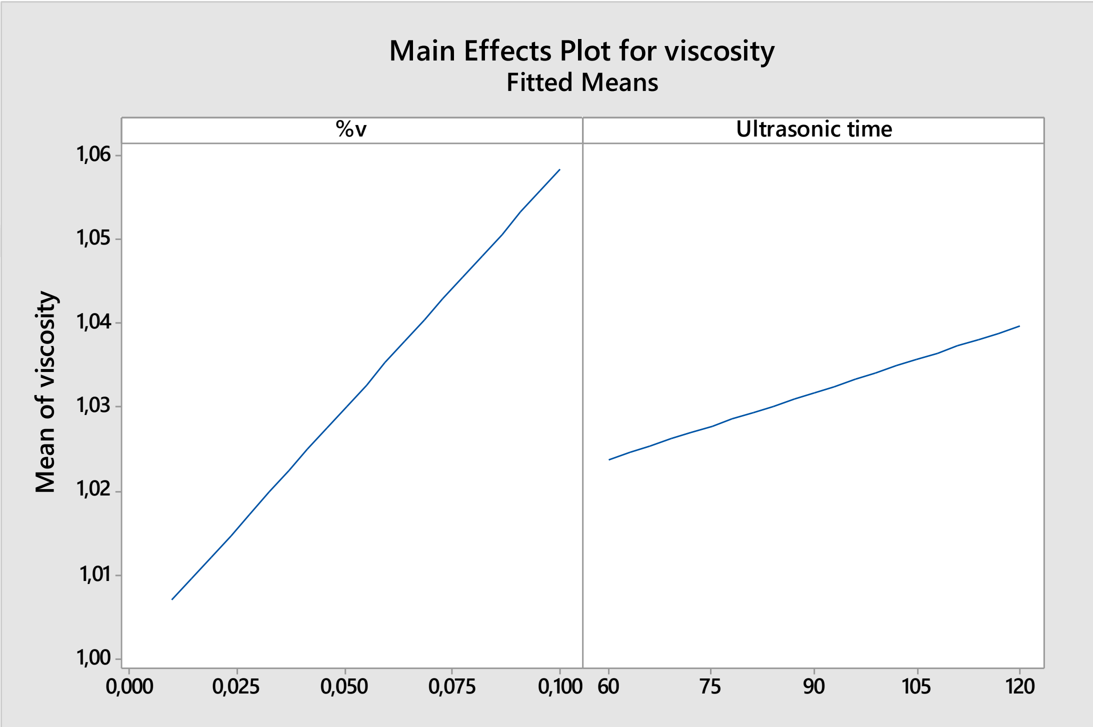


**Figure 5.8** Main effects plots for viscosity

A linear relationship between volumetric solid ratio and viscosity can be seen on the plot. Similarly, a linear relationship between ultrasonic prop time and viscosity can be seen on the plot. Both parameters have increasing effect to the viscosity.

Contour plot was drawn using volumetric solid ratio and ultrasonic prop time parameters was given in Figure 5.9 for viscosity.

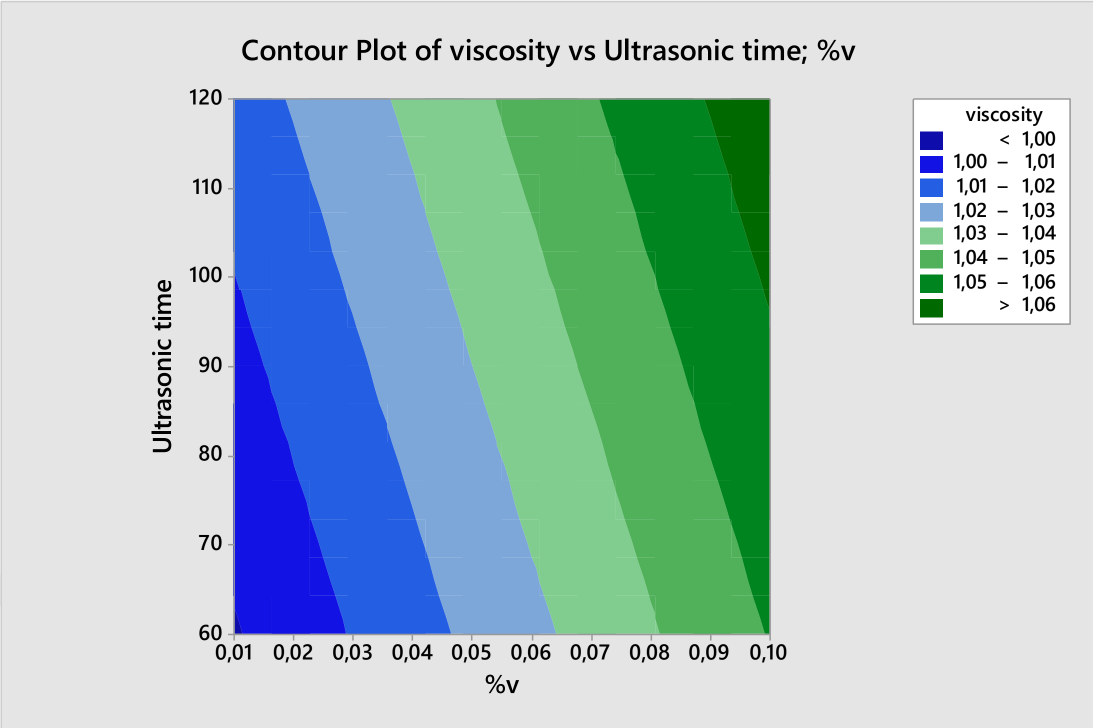


**Figure 5.9** Contour plot for viscosity

Contour plot clearly shows the effects of parameters on viscosity. For higher viscosities both volumetric solid ratio and ultrasonic prop time should be increased.

A regression equation was calculated using volumetric solid ratio (v) and ultrasonic prop time (u) parameters was given below for viscosity.

$$\text{viscosity} = 0.97764 + 0.5694v + 0.000264u \ (R^2 = 97.02\%)$$

Coefficient of determination ($R^2$) for the equation was found as 97.02%. High value this coefficient shown that linear model is useful on explaining relation between parameters and response.

Response optimizer tool was used to determine optimum viscosity output. Determined optimum results were given in Table 5.1.

**Table 5.1** Response optimizer predictions

| Solution | %v | Ultrasonic Time | Predicted Viscosity | Composite Desirability |
|---|---|---|---|---|
| 1 | 0.10 | 120 | 1.066 | 0.95 |
| 2 | 0.10 | 60 | 1.051 | 0.72 |
| 3 | 0.01 | 120 | 1.015 | 0.21 |

Solution 1 was determined as optimum result since it has the highest desirability. Optimum parameter level for volumetric solid ratio was determined as 0.100 and optimum level for ultrasonic prop time was determined as 120 minutes. When the predicted result compared with experiment 9 it can be seen that predicted result and the experimental result was quite close.

Main effects plots were drawn using volumetric solid ratio and ultrasonic prop time parameters was given in Figure 5.10 for thermal conductivity.


**Figure 5.10** Main effects plots for thermal conductivity

A linear relationship between volumetric solid ratio and thermal conductivity can be seen on the plot. Similarly, a linear relationship between ultrasonic prop time and thermal conductivity can be seen on the plot. Both of the parameters have increasing effect to the thermal conductivity.

Contour plot was drawn using volumetric solid ratio and ultrasonic prop time parameters was given in Figure 5.11 for thermal conductivity.

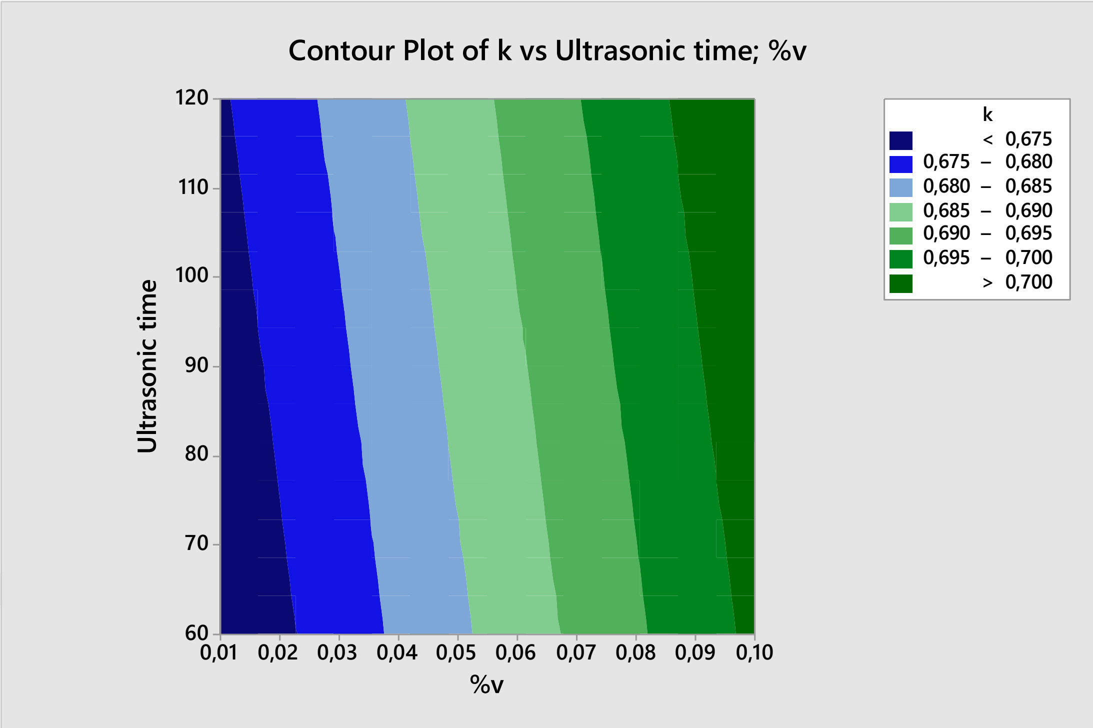


**Figure 5.11** Contour plot for thermal conductivity

Increasing effects of parameters on output can be seen on the contour plot.

A regression equation was calculated using volumetric solid ratio (v) and ultrasonic prop time (u) parameters was given below for viscosity.

$k = 0.6634 + 0.338v + 0.000063u$ ($R^2 = 14.19\%$)

Coefficient of determination ($R^2$) for the equation was found as 14.19%. Low value of this coefficient shown that linear model is not useful on explaining relation between parameters and response. This may be due to the nonlinear relationship between parameter and response.

Response optimizer tool was used to determine optimum thermal conductivity output. Determined optimum results were given in Table 5.2.

**Table 5.2** Response optimizer predictions

| Solution | %v | Ultrasonic Time | Predicted Thermal conductivity (W/m.K) | Composite Desirability |
|---|---|---|---|---|
| 1 | 0.10 | 120 | 0.705 | 0.56 |
| 2 | 0.10 | 60 | 0.701 | 0.53 |
| 3 | 0.01 | 120 | 0.675 | 0.32 |

The highest desirability value for the optimum parameter levels found in solution 1. When the predicted result was compared with the experimental result it was seen that predicted result was much higher than the experimental result. This is due to the fact that linear regression is not successful on explaining the relationship between parameter and the output. Therefore Solution 3 was selected as optimum solution to maximize thermal conductivity. Optimum parameter level for volumetric solid ratio was determined as 0.01 and optimum level for ultrasonic prop time was determined as 120 minutes. The predicted value for thermal conductivity was found as 0.675 W/m.K and experimental result was found as 0.708 W/m.K.

For a good stability of nanofluids it was desired to have zeta potential over 30-40 mW. Though zeta potential is not an output of nanofluid preparation a similar response prediction can be made to compare predicted results and the experimental results. Response optimizer tool was used to determine predicted zeta potential output. Determined results were given in Table 5.3.

**Table 5.3** Response optimizer predictions

| Prediction | %v | Ultrasonic Time | Predicted zeta potential (mW) | Composite Desirability |
|---|---|---|---|---|
| 1 | 0.01 | 120 | 40.9 | 0.85 |
| 2 | 0.10 | 120 | 37.8 | 0.74 |
| 3 | 0.01 | 60 | 25.3 | 0.31 |

The optimum parameter levels for two different outputs (viscosity and thermal conductivity) were found to be same. Optimum parameter level for volumetric solid ratio was determined as 0.100 and optimum level for ultrasonic prop time was determined as 120 minutes. These parameter levels are also adequate for the desired zeta potential.

# 6. CONCLUSION AND RECOMMENDATIONS

In this study, in which 9 samples of $Ag_2O$/water nanofluids were prepared with Taguchi L9 experimental design and the physical properties of these nanofluids were determined, the following results were obtained.

- Increasing the ultrasonic mixing time allowed the nanofluids to remain suspended.
- Thermal conductivity of nanofluids increased with increasing ultrasonic mixing time.
- Increasing the amount of solid resulted in an increase in the thermal conductivity.
- Viscosity values increased as ultrasonic mixing time, and solid amount increased.
- The zeta potential value increased as the solid amount increased
- As the amount of solid is increased, denser image nanofluids are obtained.
- As the ultrasonic mixing times of the nanofluids are increased, more intensely imaged nanofluids are obtained.

Preparation of nanofluids and determination of their physical properties is a very laborious process. Various methods have been applied in the literature to obtain stable and long suspended nanofluids. There are many methods to increase the stability of nanofluids. It is possible to obtain highly stable nanofluids by using different surfactant amounts and various surfactants.

Another important parameter for stable nanofluids is pH. Optimum pH values can be determined by examining the effect of pH change on stability.

In order for a good nanofluid to be used in an application, it is crucial that it remains stable for a long time. Therefore, determining how many days the nanofluids can remain stable will contribute.

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