ISTANBUL TECHNICAL UNIVERSITY ★ GRADUATE SCHOOL OF SCIENCE ENGINEERING AND TECHNOLOGY

POLYMER/GLASS NANOCOMPOSITE FIBER AS AN INSULATING MATERIAL

M.Sc. THESIS

Cemre İpek Akkaya

Department of Chemical Engineering

Chemical Engineering Programme

Thesis Advisor: Prof. Dr. Sadriye Küçükbayrak

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İSTANBUL TEKNİK ÜNİVERSİTESİ ★ FEN BİLİMLERİ ENSTİTÜSÜ

YALITIM MALZEMESİ OLARAK POLİMER/CAM NANOKOMPOZİT FİBER ÜRETİMİ

YÜKSEK LİSANS TEZİ

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MAYIS 2016

To my family,

FOREWORD

I would like to begin with my deepest appreciate to my supervisor Prof. Dr. Sadriye KÜÇÜKBAYRAK for her guidance and kind support all the time. She always encouraged me throughout my thesis study. In addition, I would like to appreciate her for supporting me in my work life.

I would also like to thank Assoc.Prof. Dr. Melek Mümine EROL TAYGUN for her support in every step of my thesis. I was a very lucky person to work with her. Also, I would like to thank Chem. Eng. (MSc.) Ayşen AKTÜRK for her guiding me in laboratory. I am very pleased to meet such an informative lab-mate. In addition, I also appreciate to my lab-mate Chem. Eng. (MSc.) Özge GÖNEN for her support and friendship during our long working periods. I wish both of them success in their academic lives.

I would like to thank to Cansu ÜLKER, Tuğçe İNAN, Didem AYCAN, Özlem Haval DEMIREL, Fatih TEMİZER for their friendship throughout my thesis period.

Also, I would like to thank to my general manager, Kayhan MERT and to my boss Selman ÖLMEZ for taking time off from work, and supporting me always.

I would like to show my appreciation and gratitude to my lovely parents Fatma AKKAYA and Seyfettin AKKAYA, who never left me alone. Furthermore, I would like to thank all my family members.

MAY 2016

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ABBREVIATIONS

AC	: Alternative current
CA	: Cellulose acetate
DC	: Direct current
DCCA	: Drying control chemical additives
DMAC	: Dimethylacetamide
DMF	: Dimethylformamide
DW	: Distilled water
ECM	: Extracellular matrix
EtOH	: Ethanol
FTIR	: Fourier transform infrared spectroscopy
HEPA	: High efficient particulate air
MF	: Microfiltration
MOCVD	: Metal-organic chemical vapor deposition
NF	: Nanofiltration
OIHM	: Office for harmonization in the internal market
PCl	: Polycaproloctone
PEO	: Poly(ethylene oxide)
PSF	: Poly-silicic-ferric
PVA	: Polyvinyl alcohol
PVP	: Poly(vinylpyrrolidinone)
TEOS	: Tetraethyl orthosilicate
TMOS	: Tetramethyl orthosilicate
UF	: Ultrafiltration
SEM	: Scanning electron microscopy
v/v	: Volume per volume ratio
w/w	: Weight per weight ratio
wt%	: Weight percent
XRD	: X-Ray diffraction

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FABRICATION OF POLYMER/GLASS NANOCOMPOSITE FIBER AS AN INSULATING MATERIAL

SUMMARY

Nanotechnology is an area which is utilized in lots of applications such as filter media, fiber-reinforced plastics, solar and light sails and mirrors in space, application of pesticides to plants, biomedical applications (tissue engineering scaffolds, bandages, drug release systems), protective clothing aimed for biological and chemical protection and fibers loaded with catalysts and chemical indicator. The main idea of using nanofibers is increasing the surface to volume ratio by reducing the diameter into nanometer range. This supplies better mechanical properties such as wetting behavior and strength of fibers. Using nanofibers in insulation material is a unique idea. It provides better insulating facilities with compressing the air between the layers of nanofibers. It is known that glass wool is generally used to produce insulation materials. However, nanofiber glasses can be used for insulation purposes to obtain better insulation materials then glass wool.

In this study, glass nanofibers were produced with sol-gel method by using electrospinning technique. Firstly, sol-gel mixture (ethanol, tetraethoxysilane (TEOS), calcium nitrate tetrahydrate and hydrochloric acid) and *polyvinylpyrrolidone* (PVP)/ethanol solution (as polymer) were prepared, then they mixed together. The obtained mixture was then used to produce nanocomposite material by electrospinning method. After the heat treatment process, produced nanocomposite fibers showed specific insulating material characteristics such as nonflammability and low thermal conduction coefficient. SEM, FTIR, XRD, and BET analyses were also conducted to the produced composite materials.

A second sample was improved for the durability by varying the composition of the sol. It was concluded that the nanocomposite fiber may be a possible candidate for industrial applications as an insulating material.

YALITIM MALZEMESİ OLARAK POLİMER/CAM NANOKOMPOZİT LİF ÜRETİMİ

ÖZET

Nanoteknoloji, filtre malzemesi üretiminde, lif ile güçlendirilmiş plastik üretiminde, güneş ve ışık gemilerinde, uzaydaki aynalarda, çiçekler için böcek ilacı uygulamalarında, biomedikal uygulamalarda (doku mühendisliği yapı iskelelerinde, bandajlarda, ilaç salınım sistemlerinde), biyolojik ve kimyasal koruma amaçlı kıyafet üretiminde, lif yüklenmiş katalizör ve kimyasal indikatör uygulamalarında kullanılmaktadır. Nanolifleri kullanmanın başlıca nedeni, çapı nano boyutuna indirerek yüzey/hacim oranını artırmaktır. Bu sayede, ıslanma davranımı ve dayanım gücünün artması gibi daha iyi mekanik özellikler sağlanmaktadır.

Yapılarda ve tesisatlarda ısı kayıp ve kazançlarının sınırlandırılması için yapılan işleme "ısı yalıtımı" denir. Teknik olarak, ısı yalıtımı, farklı sıcaklıktaki iki ortam arasında ısı geçisini azaltmak için uygulanır. Her yalıtım malzemesinin kullanım alanı farklıdır. Ayrıca, aynı uygulama için kullanabilecek birden fazla yalıtım malzemesi mevcuttur. Binalarda ısı yalıtımı yapılırken çeşitli malzemeler kullanılmaktadır. Dış cephe ısı yalıtımında genellikle köpük kullanılır. Isı yalıtım malzemelerinin seçiminde dikkat edilmesi gereken özellikler ise; ekonomik açıdan uygun olması, çevreye zarar vermemesi, kolay uygulanabilir olması, asitlere ve asit yağmurlarına karşı dayanıklı olması, böcek ve mikroorganizmaların üremesine uygun olmaması, hafif, yanmaz ve elastik olması, ısı iletkenlik değerinde zamanla değişim olmaması, zaman içinde bozunup çürümemesi, aşınmaması ve paslanma yapmamasıdır. Isı yalıtımının avantajları ise; enerji tüketimini azaltması, çevrenin korunmasına katkı sağlaması, ısıl konfor sağlaması, sağlıklı yaşam sunması ve de ilk yatırım ve işletim maliyetlerini azaltmasıdır. Çeşitli yalıtım malzemeleri vardır; cam yünü, taş yünü, polistren köpük, poliüretan, odun talaşı levhaları, cam köpüğü, fenol köpüğü vb. malzemeler örnek olarak verilebilir.

Yalıtım malzemesi olarak nanolif kullanımı orijinal bir fikirdir. Bu sayede, havayı nanolif tabakalarının arasına sıkıştırarak daha iyi yalıtım özellikleri sağlanabilecektir. Bilindiği üzere, cam yünü genellikle yalıtım malzemesi olarak kullanılmaktadır. Ancak, nanolif camlar daha iyi yalıtım özelliğine sahip olmaları nedeniyle, cam yününün yerini alabilecektir.

Bu çalışmanın amacı, sol-jel yöntemi ile elektrosipining tekniğini birlikte uygulayarak cam nanolifler üretmektedir.

Sol- jel tekniği, hem inorganik hem de organik-inorganik hibrit malzemelerin elde edilmesi amacıyla uygulanmakta olan çok etkili bir yöntemdir. Bu tekniğin temel avantajı, tüm prosesin oldukça kolay ve sorunsuz yürütülebilmesidir. Sol-jel prosesi, katı hal proseslerinden oldukça farklı olup, tepkime sırasında son ürünün moleküler seviyede kontrol edilebilmesine imkan sunmaktadır. Bu sayede sol- jel prosesi, çok yüksek saflık ve homojenlikte, uniform kristal morfolojisinde ve iyi tanımlanmış nanotaneciklerin üretimine imkan vermektedir. Sol-jel kimyası, sentez protokolünün

iyi tekrarlanabilirliğini sağlamak için sürekli kontrol edilmesi gereken tepkime değişkenlerinin oldukça fazla olması (metal alkoloksit ön başlatıcılarının hidroliz ve kondenzasyon hızı, pH, sıcaklık, karıştırma yöntemi, oksidasyon hızı, vb.) nedeniyle oldukça karmaşık bir prosestir. Sol-jel yöntemi hem laboratuvar, hem de endüstriyel ölçekte üretime uygun olduğu için, kullanımı giderek yaygınlaşmaktadır. Sol-jel yöntemi; ön başlatıcının hidrolizi, sol- jel aktif türlerinin alkol ya da su kondenzasyonu, jelleşme, yaşlanma, kurutma ve yüksek sıcaklık işlemi gibi basamaklardan oluşmaktadır.

Elektrospinning yöntemi; üretilen liflerin çaplarının çok küçük olmaları sayesinde filtre üretimi, kompozitleri güçlendirme ve biyomedikal cihazlar gibi geniş uygulama alanlarına uygun olduğu için son yıllarda geniş uygulama alanları bulmaktadır. Avantajları; düşük maliyet, tekrar edilebilirlik ve fiber kalınlığının kontrolünün kolayca sağlanabilmesi olarak açıklanabilir.

5kV-30kV arasındaki elektrik akımı bir polimerin yüzey gerilimini yok etmektedir. Yüzey gerilimine sahip olmayan polimer çözeltisi de, özel tasarlanmış ve çok ince yapıdaki jet ipliğinden geçerek düzenin karşısına yerleştirilmiş olan topraklanmış plakaya yönlenmektedir. Lif ışını şeklinde topraklanmış yüzeyde biriken lifler ise elektrik akımı sayesinde sürekli olarak çekilmektedirler. Elektrospinning yöntemi; polimerlerin önce çok yüksek voltajlı elektrik akımı ile yüklenmeleri, daha sonra da topraklı bir plakaya doğru akmaları esnasında katılaşmaları (çözücünün buharlaşması ile) ve lif halini almaları şeklinde kısaca özetlenebilir. Nanoliflerin homojen ve sürekli olarak üretilebilmeleri için, jetin üstünde Taylor konisinin oluşması gerekmektedir. Bu oluşumu etkileyen birçok değişken bulunmaktadır. Bunlar, çözelti değişkenleri, elektrospinning değişkenleri ve çevre koşullarıdır. Çözelti değişkenleri; viskozite, yüzey gerilimi, iletkenlik, molekül ağırlığı, derişim ve buhar basıncı olarak özetlenebilir. Elektrospinning değişkenleri ise; jet ile toplayıcı arasındaki mesafe, polimerin beslenme hızı ve uygulanan voltajdır. Çevre şartları olarak da nem ve sıcaklık sayılabilir.

Deneysel çalışmalarda ilk olarak, sol-jel karışımı (etanol, tetraetiloksisilan (TEOS), kalsiyum nitrat tetrahidrat ve hidroklorik asit) ile polyvinilpiroolidone (PVP)/etanol çözeltisi hazırlanmış ve sonra bu çözeltiler karıştırılmıştır. Elde edilen karışıma, nanakompozit üretebilmek amacıyla, elektrospinning yöntemi uygulanmıştır.

Üretilen nanofiberler, polimerden kurtarabilmek ve cam nanofiber yalıtım malzemesi üretebilmek amacıyla 600°C'de ısıl işleme tabi tutulmuştur.Elde edilen son ürüne, yalıtım malzemesinin sağlaması gereken özellikleri sağlayıp sağlamadığını kontrol edebilmek için çeşitli karakterizasyon testleri uygulanmıştır. Bunlar, yanmazlık testi, ısıl iletim katsayısı testi ve yoğunluk ölçümleridir. Ayrıca, SEM, FTIR, XRD ve BET analizleri uygulanmıştır. Üretilen ilk numunenin yanmazlık testi kalorimetre bombası kullanılarak uygulanmış olup, ISO 1716 standartlarına göre 1. sınıf yalıtım malzemesi olduğu kanıtlanmıştır. Ayrıca, CE standartlarına göre bir malzemenin yalıtım malzemesi olabilmesi için ısıl iletim katsayısının 0.065 W/mK'den küçük olması gerekmektedir. Bu test, Armfield HT10XC Isı Transferi Ölçüm Cihazı kullanılarak uygulanmış olup, ısı iletim katsayısı 0.049 W/mK olarak ölçülmüştür. Yoğunluk ölçümü ise Micrometrics piknometre sistemi ile gerçekleştirilmiştir. Sonuç olarak numunenin yoğunluğu 2419 kg/m^3 olarak saptanmış olup, bu değer cam yününinkinden yaklaşık 20 kat daha büyüktür. XRD analizi sonucunda, hem ısıl işlem öncesi hem de sonrasında amorf yapının oluştuğu gözlemlenmiştir. FTIR analizinin sonucuna göre ise, beklenildiği üzere, ısıl işlem sonrası polimere ait olan tüm piklerin kaybolduğu gözlemlenmiştir. Ayrıca, BET analizinin sonucuna göre, üretilen nanolif camın yüzey alanının cam yününinkinden 140 kat daha fazla olduğu saptan-

mıştır. SEM analizinde ise, nanoliflerin çaplarının 37 ile 87 nm arasında değiştiği gözlemlenmiştir. Ancak, üretilen ilk numunenin mekanik dayanımı oldukça düşük olup, kolayca kırılmaktadır. Bu amaçla ikinci olarak geliştirilen numunenin bileşimi S-CAM içeriği temel alınarak saptanmıştır. S-CAM, yüksek dayanımı olan ve kompozit malzemelerde yapıyı güçlendirmek için kullanılan bir bileşimdir. Geliştirilen ikinci numune aynı yöntemlerle üretilmiş olup, aynı karakterizasyon testleri uygulanmıştır. Analizlerin sonuçları aynı doğrultuda olup, üretilen ikinci numunenin, ilk üretilmiş olan numuneden daha dayanıklı olduğu saptanmıştır.
Sonuç olarak, her iki numune de, birinci sınıf yanmazlık özelliği bulunan, geniş yüzey alanlı, yüksek yoğunluğa sahip, düşük ısı iletim katsayısı olan, cam nanofiberler özelliğine sahiptir. Üretilen nanoliflerin sahip oldukları bu özellikler nedeniyle, endüstriyel uygulamalarda kullanılabilecek uygun yalıtım malzemeleri oldukları sonucuna varılmıştır. Bilindiği kadarıyla, bu çalışma, polimer/cam nanokampozit liflerin yalıtım malzemesi olarak üretildiği ilk çalışmadır.

1. INTRODUCTION

Nowadays insulator consumption has been grown because of increasing raw material prices, necessary investments for environmental technologies, potential penalties for lacking compliance with environmental regulations, increasing public awareness on resource consumption and climate change [1].

Reductions of CO_2 emissions and energy consumptions are really great challenges for our world. The European Union has aimed a reduction of 20 % energy consumption until 2020. Sectors should share this percentages between each other [2].

There are many energy resources such as oil, coal, gas, nuclear, wind, geothermal, hydropower and solar [3]. According to Turkish Statiscal Institute, in 2013, energy consumption dissipates with sectors such as 35 % construction, 37 % industry, 18 % transportation and 10 % agriculture and others [4].

According to European Comission, energy efficiency, in other words, energy savings could be increased by improving thermal insulation and by using energy efficient products and automation systems [5].

Thermal insulation is the most important application for energy saving. Especially in Turkey, using great amount of energy for heating is a problem. Furthermore, the cost of it is so high [6]. Therefore, the thermal insulation in the buildings has become critical issue for us [7].

In addition to this, thermal insulation can be applied in the white appliances, for instances in the ovens, in refrigerators or in industrial applications such as valves, gas or electric boilers and HVAC systems [8].

As it stated in “State of the art in thermal insulation materials and aims for future developments” there are four different kinds of insulation materials such as inorganic material, organic material, combined materials and new technology materials as seen in Figure 1.1 [9]. Glass-wool is the mostly used thermal insulation material [10].

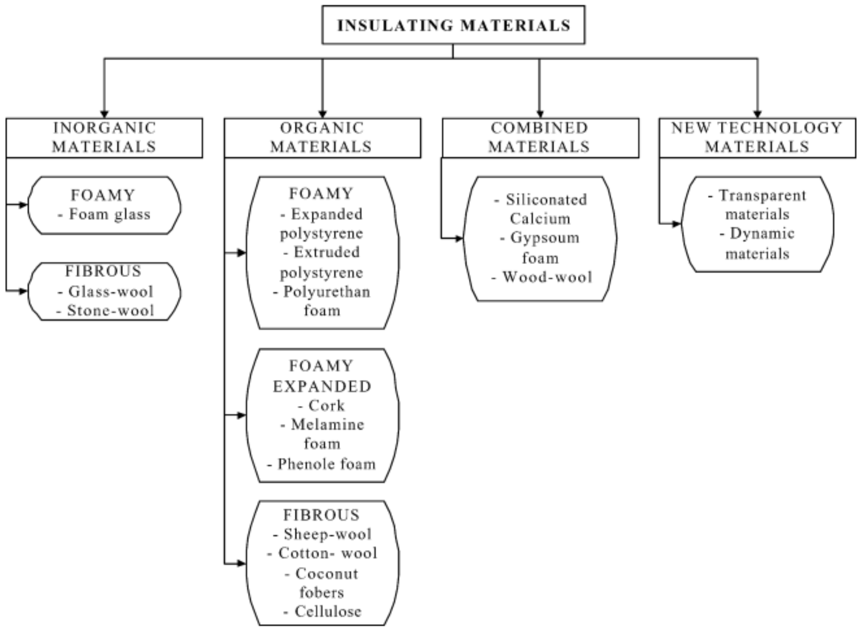

Figure 1.1 : Insulation materials. [9]

In this study, non-flammable nanofiber glass is aimed to produce as an insulation material. Sol-gel method is applied for producing glass nanofibers as the first step because of being a simple technology. However, finding the true precursor and carry out step-wise hydrolysis is important. While for sol part, ethanol, tetraethoxysilane (TEOS) as a precursor, calcium nitrate tetrahydrate and hydrochloric acid were mixed, for gel part %3 polyvinylpyrrolidone (PVP)/ethanol solution was prepared. Then they mixed together. Ethanol was used as a solvent for PVP and TEOS. To be able to increase the strength of the fiber, the ingredient of sol was changed in the second sample. For sol ingredients “S-GLASS” components were utilized to achieve high modulus, high strength and high stability at extreme temperatures. Its softening point is nearly 950 °C [11]. Furthermore, it can be used in reinforcement in composite materials. Therefore, while for sol part, ethanol, tetraethoxysilane (TEOS) as a precursor, magnesiumnitrate, aluminumnitrate and hydrochloric acid were mixed, for gel part %3 (PVP)/ethanol solution was arranged. Then they stirred together. Tetrahydrate salts were utilized for enhancing the viscosity of the solution for electrospinning. The obtained mixtures were then used to produce nanocomposite materials by electrospinning method. Electrospining method was utilized due to being cheapest, easiest method and lab-scale one. Then, heat treatment was applied to the samples at 600 °C. After heat treatment process, produced nanocomposite fibers showed specific insulating material characteristics such as nonflammability, low thermal conductivity coefficient and low density. In addition to this, the strength of the second sample is better then the first one perceptibly. SEM was applied to observe the morphology and size of the fibers. Then, FTIR was performed to determine the chemical structure of the samples. At last, XRD was applied to characterize the formation of glass fiber. In addition to these analyzes, nonflammability test, density and thermal conductivity coeffecient measurements were performed on the samples.

2. NANOTECHNOLOGY AND NANOFIBERS

Nanotechnology is an area which is utilized in a wide range from space crafts to biomedical applications. According to the type of the application, there are different dimensional systems such as three-dimensional systems (photonic band gap materials), two-dimensional systems (quantum well structures) or one-dimensional systems (quantum wires, nanocables) [12]. Generally, one dimensional systems are nanofibers. The main idea of using nanofibers is increasing the surface to volume ratio by reducing the diameter into nanometer range [13]. This supplies better mechanical properties such as wetting behaviour and strength of fibers. Today, nanofibers are fabricated from synthetic or natural polymers in a controlled manner. To produce nanofibers, there are many ways to choose. Mainly, these can be divided as extrusion and electrospinning. Extrusion has two segments as fine-hole and islands in the sea. Firstly, fine hole is a meltblown process in which melted polymer is pressurized through small holes. In the islands for sea process, polymer blends are generally hydro-tangled, but firstly, they are extruded through thicker holes, then dispersed afterwards [14]. On the other hand, electrospinning is the most common method for producing nanofibers and has two types such as nozzle and nozzle-less. For laboratory scale, nozzle process in which solvent polymers are forced through a needle and generate an electrostatic field can be applied [15]. In addition to this, nozzle-less electrospinning is a technology for industrial processes. In nozzle-less process, electrical field is generated without needles by using higher voltage on roller-electrodes. This voltage is nearly between 30-120 kV, while nozzle electrospinning technology requires only 5-20 kV. To supply this mechanism, an instrument which is called "Nanospider" is required. To obtain large fiber sizes (800 – 2,500 nm diameters), extrusion method should be chosen, while 80 – 500 nm diameters of fibers can be formed by nanospider. These products can be specified with lots of parameters such as fiber diameter distribution (mean value and its standard deviation), basis weight of the nanofiber layer, etc. However, for the significant applications, functional product parameters like initial gravimetric filtration efficiency (IGE), differential filtration efficiency, and pressure drop has became much more important [16].

2.1. Application of Nanofibers

Nanofibers can be applied in a wide range area. Some of these areas were described below.

2.1.1. Filtration applications

Filtration is widely used in many engineering applications. In these applications, fibrous materials are chosen for filter media because of providing advantages in high filtration efficiency and low air resistance. The most common method for developing high efficient and effective filter media is electrospinning. By this method, unfriendly particles in submicron range are removed. Producing of nanosized filter is required to match channels and structural elements of a filter to the scale of the particles or droplets that are to be captured in the filter. Generally, due to the high ratio of surface area to volume, high cohesion forces are resulted. Therefore, tiny particles of the order of <0.5 mm can be easily kept in the electrospun nanofibrous structure, so that efficiency will be increased [17-19].

2.1.2. Tissue engineering applications

In tissue engineering applications, organs or tissues are treated by designing the drafting of ideal scaffolds or synthetic matrices which can be replaced with natural extracellurlar matrix (ECM). The basis of using fibers in tissue engineering applications is the capability of fibers (with diameters which are smaller than those of the cells) to attach to human cells and organizing well around. Therefore, human cells can be an optimum environment for fibers and they can seed, migrate, and grow [20]. According to Li et al.[20], conductive polymers such as polyacetylene, polypyrrole (PPy), polythiophene, polyaniline (PANi), and poly(para-phenylene vinylene) are preferred for tissue engineering applications because of cell function capability as attachment, proliferation, migration, and differentiation. These polymers supply to synthesis of nanoparticles, immobilization of proteins, and coating devices with PPy materials [21].

2.1.3. Wound dressing applications

Nanofibers can also be utilized in treatment of wounds or burns of human skin. These nanofibers should be designed for hemostatic devices with some special characteristics. To produce a fibrous mat dressing by the electric field, fine fibers of biodegradable polymers should be sprayed onto injured place of human skin. It provides wound healing with supplying the production of normal skin growth [22].

2.1.4. Drug delivery applications

Patients always have difficulties like taking medicine on time or carrying medicine by near themself, that's why the idea of absorbing medicine in human body is better. Main advantage of polymer nanofiber based drug delivery system is high dissolution rate of a particulate drug which increases with growing surface area of both the drug and the corresponding carrier. While drug and carrier materials are mixed during the electrospinning process, the resulting nanostructured products can be in different structures such as drug as particles attached to the surface of the carrier which is in the form of nanofibers [23-25].

2.1.5. Cosmetic applications

Generally, cosmetic materials are made by tropical creams, lotions or ointments which consist of dusts or liquid sprays. If these materials are fibrous, they can migrate into sensitive areas of the body such as the nose and eyes where the skin mask is being implemented to the face. These masks are utilized for the treatment of skin healing, skin cleaning, or other therapeutical or medical properties with or without various additives. This process pass gently and painlessly to improve healing or care treatment of the skin [22]. Qun et al.[26] stated that, the gold nanoparticles are potential vaccine carriers which are used in beauty clinics and saloons. They support blood circulation, skin elasticity, and decrease the formation of wrinkles. Furthermore, gold nanoparticles do not damage the human skin [26].

2.1.6. Electrical and optical applications

Conductive nanofibers are used in electrical and optical applications to produce small devices like junctions, sensors and actuators. In addition to this, conductive nanofibrous membranes are preferred to use in these applications because of high electrochemical reaction rates, so that high performance batteries will be produced.

These membranes have lots of usages such as electrostatic dissipation, corrosion protection, electromagnetic interference shielding, and photovoltaic device [22].

2.1.7. Protective clothing applications

In military, protective clothes must have some specialities such as maximize the survivability, sustainability, and combat effectiveness of the individual soldier system against extreme weather conditions, ballistics, and NBC (nuclear, biological, and chemical) warfare. If the clothes are produced by fibrous mat, then they will have these specialities, and also they will be lightweight and breathable [13].

2.1.8. Energy applications

Nanofibers are used in many energy applications such as lithium ion batteries, photovoltaic cells, fuel cells and supercapacitor electrodes.

2.1.8.1. Lithium ion batteries

Li-ion batteries have three layers such as an anode, a cathode, and a porous separator that is positioned between the anode and cathode layers. While the anode contains graphites and other conductive additives, the cathode consists layered transition metal oxides (e.g., lithium cobaltite ($LiCoO_2$) and lithium iron phosphates (LiFePO4)) [27-29].

Energy conversion and storage devices are critical subject nowadays. Applying nanotechnology in these area supplies benefits in catalysis for fuel cells. It will affect battery’s performance positively. The basic speciality of lithium-ion batteries is allowing lithium ions to pass quickly and reversibly according to intercalation, alloying or conversion reactions [30].

2.1.8.2. Fuel cells

Fuel cells convert chemical energy to electrical energy directly. They are electrochemical devices. The most important speciality of fuel cells is lowering the operating temperature which provides an increase in cathode polarization significantly. Nowadays, fuel cells are most popular in car industry [31].

3. INSULATION MATERIALS

Thermal insulation material is a material or mergence of materials which could delay the rate of heat flow by conduction, convection, and radiation. It also delays heat flow into or out of a building because of its high thermal resistance. Thermal conductivity is related with the effectiveness of an insulation material in conducting heat [32]. According to Munoz et al.[33], thermal conductivity is the most critical feature of an insulation material. The most accurate and reliable way to obtain its value for a specific sample is to measure the thermal conductivity according to a standard method. The most reliable sources of data are the European and ISO standards, the ASHRAE Handbook of Fundamentals and the CIBSE Guide-A [33].

There are so many benefits of thermal insulation. It helps to conserve energy and the associated natural resources and it has economical benefits. An energy cost is an operating cost, and by utilizing thermal insulation, capital expenditure in other words money and big amount of energy can be saved. While it decreases the operating cost, HVAC equipment initial cost decreases too because of reduced equipment size required. The other benefit could be environmental benefits.

The pollutants could be deacreased by utilizing thermal insulation. It can also decrease the level of noises. This will increase the acoustical comfort of insulated buildings. What is more ? An another benefit is fire protection.

There are five main groups in thermal insulation materials. These are glass wool, stone wool, expanded polystyrene, extruded polystyrene and polyurethane foam. Firstly, glass wool includes of quartz sand, dolomite, resovit and limestone. Besides, adhesive materials and water-repellent oils are jointed in order to enhance the mechanical strength of the materials by supplying a high fire-resistance. Secondly, stone wool includes of the same basic materials as glass wool. Stone wool is heavier and has a higher melting point; therefore it is proper for high temperature applications. Expanded polystyrene includes polymerised polystyrol (1.5–2%) and air (98–98.5%). Extruded polystyrene is related with polymerised polystyrol.

The features of these insulation materials are important in choosing the application place. The specialities of insulation materials can be divided into three categories such as physical properties, environmental impacts and public health influences. The physical properties are density, mechanical strength, thermal insulation ability, sound absorption, resistance to moisture and fire, etc. The traditional physical properties are certified according to specific standards. The second group is environmental impacts that contains features such as primary embodied energy, the gas emissions for the production of the material, the use of additives against biological impacts, the classification of their treatment as waste, etc., their re-usability and recyclability. Thirdly, public health influences contain features such as dust and fibers emissions, biopersistence, toxicity in case of fire, etc. [9].

The common and current insulation material is glass wool that is a fibrous material, which consist principally of sodium, calcium and magnesium silicates, however it can include smaller amounts of other elements [34]. It is categorized as non-combustible or limited combustible depending on the binder content. Thermal insulation properties of glass wool and some other insulation materials can be seen in Table 3.1 [35].

Table 3.1 : Insulation materials. [35]

Insulation	**Density Range (kg/m^3)**	**Conductivity Range (W/mK)**	**Flammbility Euroclass Range**
Glass Wool (GW)	10-100	0.030-0.045	A1-A2
Stone Wool (SW)	22-180	0.033-0.045	A1-A2
Extruded polystrene (XPS)	20-80	0.025-0.035	E-F
Expanded Polystrene (EPS)	10-50	0.029-0.041	E-F
Pheonolic (PhF)	30-40	0.029-0.041	B-C
Polyurethane (PUR)	30-80	0.029-0.041	D-E
Polyisocyonurate (PIR)	30-80	0.023-0.041	C-D

3.1. Production of Glass Wool

Glass fiber manufacturing symbolizes high-temperature conversion of various raw materials especially borosilicates into a homogeneous melt, contunies with the fabrication of this melt into glass fibers. There are two basic types of glass fiber products as, textile and wool, which are manufactured by similar processes. Glass fiber production can be divided into 3 phases: raw materials handling, glass melting and refining, and wool glass fiber forming and finishing. The general process is the same for both products, but only the last phase is slightly different for textile and wool glass fiber productions [36].

3.1.1. Raw materials handling

Glass fiber is made by sand, but it also includes varying quantities of feldspar, sodium sulfate, anhydrous borax, boric acid, and many other materials.

Raw materials are unloaded by different methods such as drag shovels, vacuum systems, and vibrator/gravity systems. These raw materials are transported to silos from storage piles by belts, screws, and bucket elevators. In this stage, the main step is weighing according to the desired product recipe [35,36].

3.1.2. Glass melting and refining

The glass melting and refining step is carried out in the glass melting furnace. In this step, the raw materials are heated to 1500-1700°C. With this influence, they become molten glass with chemical reactions. There are many furnace designs, but especially they have general specilities such as large, shallow, and well-insulated vessels that are heated from above. Raw materials are introduced continuously on top of a bed of molten glass, at this stage they start to mix slowly and dissolve. Mixing is affected by natural convection, gases rising from chemical reactions, and, in some operations, by air injection into the bottom of the bed. Then, the second stage of this step is refining, molten glass starts to pass into a refining unit, here bubbles and particles are annihilated by settling. Furthermore, the melt is cooling for validating the proper viscosity for the fiber forming operation [37,38].

3.1.3. Glass fiber forming and finishing

Wool fiber glass is produced for insulation and is formed into mats that are cut into batts. The insulation is used primarily in the construction industry. The production occurs in five steps: preparation of molten glass, formation of fibers into a wool fiber glass mat, curing the binder-coated fiber glass mat, cooling the mat, and backing, cutting, and packaging the insulation [37,38].

3.1.4. Textile glass fiber forming and finishing

For textile glass fiber production firstly, the molten glass is heated with platinum bushings containing numerous very small openings. The continuous fibers going out from the openings are passing a roller applicator, which supplies a coating of a water-soluble sizing and/or coupling agent. Then, the coated fibers are wounded into a spindle. After the spindles, the glass fibers are transported to a drying oven. The spindles are then sent in an oven to cure the coatings.

At last, they are sent to twisting, chopping, weaving, and packaging sections [37,38].

4. SOL-GEL

Sol-gel method was discovered in 1845 at Manufacture de Ceramiqués de Sevres in France by Mr. Ebelmen. Basically, sol-gel process is defined as formation of solid materials, especially inorganic nonmetallic metarials from solutions. These solutions can be monomeric, oligomeric, polymeric or colloidal precursors. In addition to this, if the dispersed phase is too small, gravitional forces can be neglected and interactions can be dominated by short-range forces as van der Waals attraction and colloidal surface charges. Furthermore, if the liquid consists colloidal suspension of solid particles, it will be called as “sol”. Sol fills the pores by gel which consists of sponge like three dimensional solid network [39-41].

4.1. History of Sol-Gel

The sol-gel usage stands to mid 1980s. This method has been utilized to synthesize inorganic polymer and organic–inorganic hybrid materials. At past, Schot Glass Company used this method almostly in 1900s [42]. The most important success was recognized with synthesized boronsilicate glass by heating bathes of oxide powders obtained by a low temperature sol-gel process by Dislich. After this success, the number of publications increased with sustainable exponential growth. The another important step is the first International Conference on Glasses and Glass Ceramics Obtained from Gels was held in Padova, Italy in 1981 that supplies improvement of sol-gel science [43]. From past to nowadays, it has been used in lots of applications such as catalysis, chemical sensors, membranes, fibers, optical gain media, photochromic and non linear applications, solid state electrochemical devices, in diverse range of scientific and engineering fields, such as ceramic industry, nuclear field industry and electronic industry because of its several advantages [42].

At last, the history of sol-gel technologies and the development of materials by utilizing them is far from ending. The possibility of changing both parameters and precursors are endless.

4.2. Chemical Reactions in Sol-Gel

In sol-gel synthesis, metal oxides are usually chemical precursors. They belong to organometallic compounds family. These have shapes like R-O-M or R-OH, have a metal atom, M, replacing the hydrogen H in the hydroxyl group. In addition to this, silicon dioxide is the rifest mineral in the world. It has several crystalline forms of silica, at least seven, including quartz. Tetrahedron (SiO_4) is the basic building block of all of these crystalline forms in which the ratio of oxygen to silicon is 2:1 on behalf of 4:1. Tetrahedrals' specific geometry has been shaped for long years by giving incredible terrestrial pressures at great depth , therefore, SiO_2 is main part of a glass. Because of setting conditions of crystallization especially the long cyrstallization time in laboratory is difficult, glasses' main part should be amorphous silica. Silicon tetraethoxide is a well-known and well-investigated alkoxide. It is also called as tetraethyl orthosilicate (TEOS) which has chemical formula as $Si(OC_2H_5)_4$, or $Si(OR)_4$ where the alkyl group R represents C_2H_5.

The first reaction is hydrolysis; alkoxides are utilized because they can easily make a reaction with water. In these reaction (4.1), a hydroxyl ion has attached to the silicon atom.

$$Si(OR)_4 + H_2O \rightarrow HO\text{-}Si(OR)_3 + R\text{-}OH \quad (4.1)$$

The process depends on the presence of water and catalyst. Hydrolysis can proceed to completion or it can continue with partial hydrolysis. If it is finished, all of the OR groups will be replaced by OH groups as it can be seen in equations (4.2) and (4.3).

$$Si(OR)_4 + 4\ H2O \rightarrow Si(OH)_4 + 4\ R\text{-}OH \quad (4.2)$$

$$(OR)_3\text{–}Si\text{-}OH + HO\text{–}Si\text{-}(OR)_3 \rightarrow [(OR)3Si\text{–}O\text{–}Si(OR)_3] + H\text{-}O\text{-}H \quad (4.3)$$

In partial hydrolysis, ($(OR)_2$–Si-$(OH)_2$) or ($(OR)_3$–Si-(OH)) should be noticed as intermediate species. Furthermore, these species will react with condensation to form a siloxane [Si–O–Si] bond as it can be seen in equation (4.4).

$$(OR)_3\text{–}Si\text{-}OR + HO\text{–}Si\text{-}(OR)_3 \rightarrow [(OR)3Si\text{–}O\text{–}Si(OR)_3] + R\text{-}OH \quad (4.4)$$

Polymerization occurs with the production of H-O-H and R-O-H species [44-45].

$$(OH)_x(OR)_ySiOH + ROSi(OH)_v(OR)_w \rightarrow$$

$$(OH)_x(OR)_ySi - O - Si(OH)_v\ (OR)_w + ROH \quad (4.5)$$

Silicon dioxide formation procedure can be seen in Figure 4.1 [46]. There are several parameters which affect this mechanism and these reactions. These can be arranged such as compositions and concentrations of alkoxides and solvent used, amount of added water, catalyst used (type & concentration), another additives such as NH_2CHO to desiccate controlling chemical additives, mixing time, and temperature.

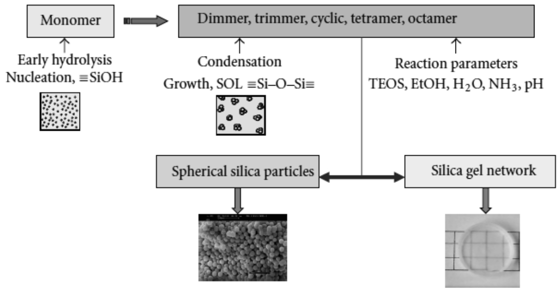

Figure 4.1 : Silica formation by sol-gel process. [46]

4.3. Steps of Sol-Gel

There are five steps in sol-gel process such as mixing, gelation, aging, drying, and sintering [47,48].

Mixing: The first step is mixing and it is the most interesting part of sol-gel process to find metalorganic compound to form a polymeric gel for polymerization. The key of design is finding the proper monomer that will polymerize to form M-O-M structures. Metal alkoxides, M(OR)n, where M is the metal and R an alkyl radical, fulfill these necessities. There are well-liked choices to utilize for preparation of silica-based gels are tetramethoxysilane, $Si(OCH_3)_4$, and tetraethoxysilane, $Si(OC_2H_5)_4$, known as TMOS and TEOS. Firstly, liquid silicon alkoxide precursor $Si(OR)_4$ reacts with water and starts hydrolysis and polycondensation reactions in the presence of a common solvent (normally alcohol), since water and the alkoxide are immiscible.

Gelation: After a while, the polycondensation of silicon alkoxide generates colloidal particles which link together to produce a 3-D network. The catalyst has a vital responsibility in this process because of the ionic charge of silica particles, with an effect on the polycondensation rate directly. Therefore, at low pH, for instance, silica particles bear very little ionic charge and that can collide and add into chains and form a polymeric gel. Because of this reasons, it is generated around at pH 1.7 which is the silica isoelectric point and the surface charge is zero. On the other hand, at high pH, where the rate of dissolution is higher, the particles grow in size and decreased in number as the smaller ones dissolve (positive curvature) and the silica is accumulated upon the larger ones. In this case, a colloidal gel is formed; this gel should be seen like a bean pot. As a direct consequence of the pictures outlined, the severe change in the rheological behavior of the sol could be utilized to determine the gel point [47,48].

Aging: The aging process is divided into three steps: continuing polymerization, synthesis, and coarsening. Connectivity of the gel network become higher by polymerization of unreacted hydroxyl groups. This process runs with some shrinkage. Irreversible shrinkage of the gel network is called synthesis which is spontaneous. It is concluded in expulsion of pore liquid. Also, solid network is drawn into the liquid by the driving forces of the liquid and produce compressive stresses. Finally, coarsening or ripening symbolizes to a process of dissolution and reprecipitation. This process effects the strengthening of the gel. Furthermore, it is affected by the solubility, temperature, pH, concentration, and type of the solvent [47,48].

Drying : One of the main problems in the preparation of bulk materials is avoiding cracking of the gel during drying, due to the stresses caused by the capillary forces associated with the gas-liquid interfaces There are two ways to dry. The first one is evaporation at a very low rate. This strategy, although effective, is not usable because it takes long time (weeks, even months). The second method is using chemical additives (DCCAs) that change the surface tension of the interstitial liquids, let fast elimination of unwanted residues. The most well-liked DCCAs used for drying silica gels is formamide. It is suggested that this additive supplies the nucleation of growing aggregates. On the other hand, because of the high viscosity of formamide, formation of a formamide layer on the gel surface occurs. This likely decreases the capillary pressure in two ways such as by forming a surface film and it reduces the contact angle and the vapor pressure. Due to its low vapor pressure, it evaporates very slowly, promoting a plasticizing effect that minifies crack formation [47,48].

Sintering : Sintering is a network densification process, which is driven by interfacial energy. Solid network can move according to viscous flow or diffusion to eliminate porosity. In gels with high pore surface areas the driving force is great enough to generate sintering at exceptionally low temperatures, where the transport processes are relatively slow. Furthermore, the kinetics of densification in gels are not simple, due to the concurrent processes of dehydroxylation and structural relaxation. The steps of sol-gel process can be seen in Figure 4.2 [46].

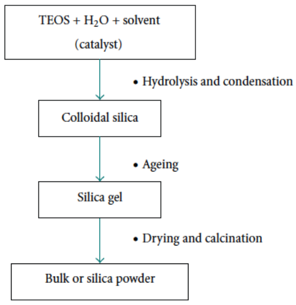

Figure 4.2 : Typical flowchart of sol-gel process. [46]

5. ELECTROSPINNING

Electrospinning is a unique process which can supply producing of continuous fibers with diameters down to a few nanometers. It is applied on to not only synthetic and natural polymers, polymer alloys, and polymers loaded with chromophores, nanoparticles, or active agents, but also to metals and ceramics. Furthermore, this technique can be utilized in both laboratory and industry scale [46]. The basis of electrospinning is unaxial stretching or elongation of a viscoelastic jet arise from a polymer solution or melt. The fiber is produced by electostatic repulsions between surface charges. By this electrical field, thinner fibers can be generated [49].

5.1. Devices of Electrospinning

Firstly, electrospinning has three major components such as a high-voltage power supply , a spinneret, and a collector. Collector can have different shapes such as rectangular, circular, or it can be a needle, too. It supplies to collect different fibrous assemblies. Furthermore, different materials can be brought together to make a collector to have advantage for production. For instance, these are rotating drum, paralel electrodes, rotating wire drum collector, drum collector with wire wound it, disc collector, array of counter electrodes, rotating drum with sharp pin inside, yarn collection using waterbath and multiple spinnerets. The common advantages of these collectors are seting-up simple and getting highly aligned fibers possible [50]. Both AC and DC currents can be used, however, direct current is generally used. Polymer solution is put into the syringe which is connected to the spinneret. By pump and syringe, with a constant rate, polymer solution can be fed to the spinneret. Also, high voltage which is usally between 1-30 kV is neccessary to start the production. When optimum parameters are set, there will be Taylor Cone on the tip of nozzle. Taylor Cone is occured when electrostatic forces overcome the Colombic forces. The schematic diagram of the electrospinning process can be seen in Figure 5.1 [51]. The homogenous production can be generated with the forming of Taylor cone as it was shown in Figure 5.1

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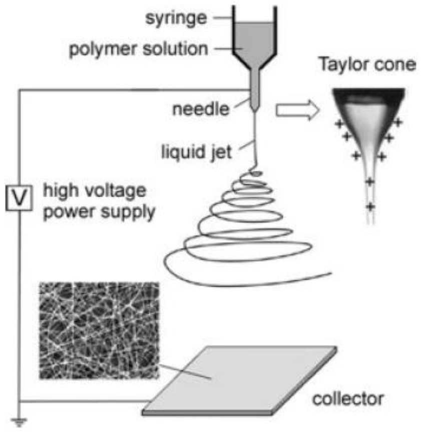


Figure 5.1 : Devices of electrospinning. [51]

5.2. The Working Mechanism of Electrospinning

Generally, for a typical electrospinning, firstly, solution is put in a syringe, then fed through a spinneret by the help of a pump. Then, a high voltage which should be the critical voltage and usually more than 5 kV is applied to the solution. In order to have a homogeneous and continuous solution, repulsive forces should be larger than solution's surface tension. At this moment, a drop will be occured at the tip of the spinneret. Therefore, jet enters a bending instability stage, so that by the solvent evaporation, fiber has occured on the road between the tip and the collector [50,52].

5.3. The Parameters of Electrospinning

Working parameters are very critical to understand not only the nature of electrospinning also the conversion of polymer solutions into nanofibers trough electrospinning. There are several parameters which affect the electrospining process directly. These parameters can divided into three groups such as solution properties, process parameters, and lastly ambient conditions. These parameters can be seen in Table 5.1.

Table 5.1 : Electrospinning parameters.

Solution Parameters	Process Parameters	Ambient Conditions
Viscosity	Feed Rate of Solution	Relative Humidity
Surface Tension	Geometry of Spinneret	Temperature
Conductivity	Shape of Collector	
Molecular Weight	Applied Voltage	
Vapor Pressure	Distance Between Tip and Collector	
Concentration		

5.3.1 Solution Parameters

5.3.1.1 Vapor pressure

Vapor pressure is an important issue for spinnability. It affects the viscosity, basically the solution should evaporate through the distance between the tip and collector and then it should converts into fiber. So that, vapor pressure affects the speed of evaporation [53].

5.3.1.2 Concentration

During the electrospinning process, concentration is an important issue for continuous and homogeneous fiber formation. The value of concentration should be optimum, if it is very low, polymeric micro (nano)-particles can be defined. Therefore, due to the low viscosity, electrospray will form instead of electrospinning. If the concentration is little higher than its optimum value, a mixture of beads will form. Furthermore, if the concentration is much more higher than its optimum value, then, helix-shaped microribbons will form. Generally, increasing concentration results in fiber diameter reduction. Also by changing concentration, viscosity can be set [53].

5.3.1.3 Molecular weight

Molecular weight is another important parameter for electrospinning process. It affects morphologies of electrospun fiber. Basically, molecular weight is related with solution viscosity directly because of entanglement of polymer chains in solutions. By decreasing the molecular weight at constant concentration, beads can be obtained instead of smooth fiber formation. Generally, smooth fiber, moreover micro-ribbon can be produced by increasing the molecular weight. However, it depends on the type of the polymer; finding optimum value is critical [53].

5.3.1.4 Viscosity

Solution viscosity is the most important point in electrospinning for fiber formation. Generally, solution viscosity has a critical range. If it is very low, continuous and smooth fibers cannot be obtained. On the other hand, if it is very high, there will be hard ejection of jets from solution. As it was mentioned before, polymer concentration, and molecular weight of the polymer hve relationship with each other. In addition to this, viscosity and surface tension are couple components, so, if viscosity is too low, surface tension will be the dominant factor, by that beads or beaded fiber can be produced [13,53].

5.3.1.5 Surface tension

Surface tension is a critical parameter for producing smooth and continuous fibers. It is related with solution characteristic. For instance, for PVP there are three solvents such as ethanol, DMF, and MC to use. All of the solvents have different surface tensions. Generally, to obtain smooth fibers instead of beaded fibers, surface tension should be reduced at constant concentration. Another way for setting surface tension is altering the composition of the solvent. For instance, Doshi & Reneker [54] stated that by reducing surface tension of a polymer solution, fibers could be defined without beads. However, in some applications, it should be applied with caution. It is negligibly dependent on the polymer concentration. On the other hand, a lower surface tension of a solvent is not always appropriate for electrospinning, it depends on the type of the polymer [53,54].

5.3.1.6 Conductivity

Surface charge density is called conductivity. Electrospinning uses electrical field to form fibers. Therefore, conductivity characteristic of a solution can be important. Conductivity should supply decrease the charge carrying ability of the polymer jet by ions, this improves lower surface tension and it concludes with smooth fiber formation. Polymers are divided into two groups such as natural and synthetic polymers. Using synthetic polymers will improves fiber formation. Conductivity can be adjusted by adding salts such as KH2PO4, NaCl, etc. Along with increasing conductivity, adding salts also decrease fiber diameter. Second method to obtain solutions with high conductivity is adding organic acid. Angammana et al.[55] investigated the effects of changing the conductivity of polyethylene oxide (PEO)/water solution on the electrospinning process and fiber morphology. Changing conductivity is occurred by adding NaCl. As a result, firstly, by increasing conductivity of the solution, the average jet current increases with the increase in the conductivity of the solution, after that, jet decreases slightly. Also, by increasing conductivity of the solution, the average fiber diameter decreases. Angammana et al.[55] claimed that conductivity should be at optimum value; if it is very low, electrospinning can not occur. On the other hand, if the conductivity is too high, Taylor cone will not form because of the depleted tangential electric field along the surface of the fluid droplet [53,55].

5.3.2 Process parameters

5.3.2.1. Applied voltage

Applied voltage is a considerable matter for electrospinning process. There is a minimum voltage which is threshold voltage that necessary for appearance of Taylor Cone. During the starting of electrospinning, a cone-shaped deformation of the drop of polymer solution can be seen by the applied voltage. The cone angle is nearly 30°. When a higher voltage is applied, a jet appears and moves toward the counter electrode and becomes narrower in the process as it can be seen in Figure 5.2. [52]. For an applied voltage of 20 kV, Fig.2B shows a jet perpendicular to the counter electrode. Fig.5.2C is at an applied voltage of 20 kV, with a jet diagonal to the counter electrode.

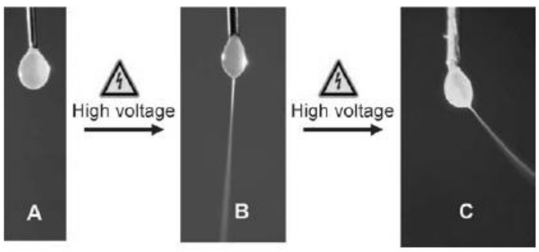


Figure 5.2 : The influence of applied voltage to the droplet. [52]

In addition to this, effect of applied voltage on fiber diameter is a little bit polemical. For example, Reneker and Chun [56] claimed that there is not much influence of electric field on the diameter of electrospun PEO nanofibers. Oppositely, there are different thoughts that higher voltages facilitated the formation of large diameter fiber [56]. For instance, Zhang et al. [17] investigated the effect of voltage on morphologies and fiber diameters distribution with poly (vinyl alcohol) (PVA)/water solution as model and they determined a direct relation between the fiber diameter and applied voltage. Furhermore, other researchers believed that higher voltages can arise the electrostatic repulsive force on the charged jet, so that, the narrowing of fiber diameter. As an example, the effect voltage on morphologies and fiber alignment with polysulfone (PSF)/DMAC/acetone as model and the fiber diameter was narrowed by the increasing of voltage [57]. In addition to this, there is another claim that higher voltage can be a problem such as the beads formation [53].

5.3.2.2 Flow rate

Another important process parameter is flow rate. Lower flow rate should be chosen generally for the continuation of polymerization. On the other hand, if it is adjusted too high, bead fibers with thick diameters will be generated due to not having time for solvent evaporation and low stretching forces [53].

5.3.2.3 Shape of collector

Shape of collector is significant for taking specimen after production. In basic laboratory scaled electrospinning, aluminum foil is usually used. However, it has disadvantage because it is difficulty to transfer the collected nanofibers to other substrates for several applications. Various nanofiber collections can be obtained by different shape of collectors such as wire mesh, pin, grids, parallel or gridded bar, rotating rods or wheel, liquid bath, and etc., as it can be seen in Figure 5.3. [53]. The collector structure has a significant influence on the properties of the resulting material (product), the collector design is always adapted to the product requirements in terms of the size and internal morphology of the nanomaterial. The differences between type of collectors; especially between rotating and fixed collector can be seen directly from Figure 5.3.

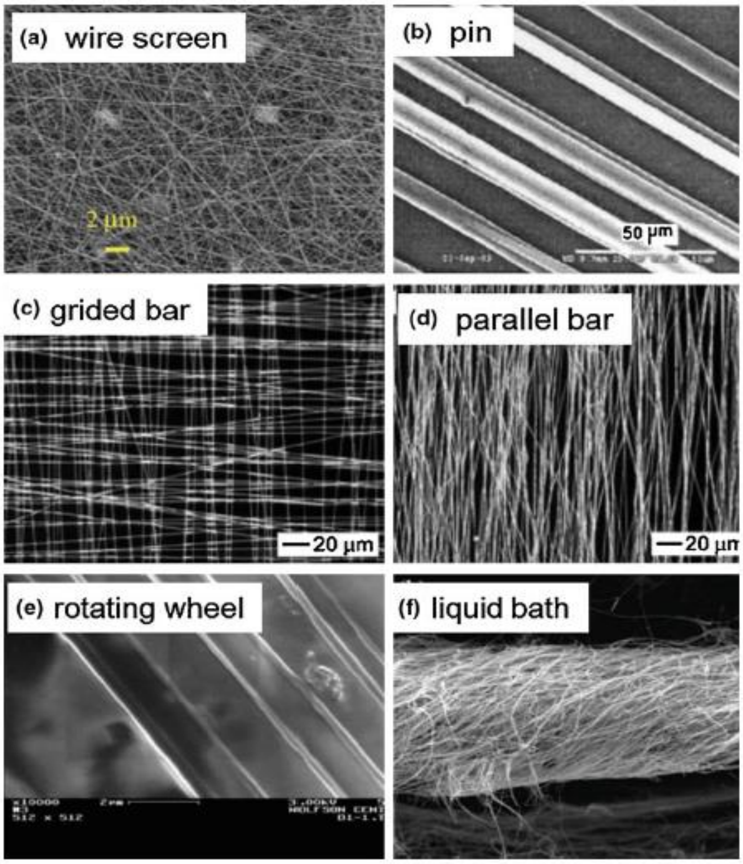


Figure 5.3 : SEM images for different collector shapes. [53]

5.3.2.4 Distance between jet and collector

The distance between jet and collector is a vital parameter for electrospinning process. Firstly, restriction is neccessary because solvent needs enough time for evaporation. Secondly, it will affect the fiber diameter and morphologies. If the distance is too short, electrospray can be formed. On the other hand, if the distance is too high, bead nanofibers can be obtained. So, optimum value for distance between jet and collector is significant for producing homogeneous and continuous nanofibers. Mazoochi et al. [58] stated that, by changing the distance between tip and collector, spinnability has been affected directly and at low distances such as 5 cm, electrospraying has occurred instead of electrospinning [58] .

5.3.3 Ambient conditions

5.3.3.1 Relative humidity

Relative humidity affect the solution properties directly. If it is too high, streching forces will be too small, charges on the jet can be neutralized. Therefore, thick fiber diameter can be produced. On the other hand, if it is too small, solvent can be dried totally, so that it can accelarate the solvent evaporation. Vrieze et al.[59] studied the influence of humidity and temperature on the formation and the properties of nanofibres using cellulose acetate (CA) and poly(vinylpyrrolidone) (PVP) as target materials [59]. According to Vrieze et al.[59], thick or thin fiber diameter producing possibility varries with chemical nature of polymer.

5.3.3.2 Temperature

Temperature is a significant parameter which affects electrospinning strongly. It changes fiber diameter by two mechanisms. These are the influence of humidity and temperature on the evaporation rate of the solvent and viscosity of the solution. According to Dao and Jırsak [60], the average fiber diameter decrease by increasing temperature in the electrospinning. This can be explained by having an inverse relationship between viscosity and temperature [60].

6. RHEOLOGY

Rheology is a science that studies the flow and deformation of solids and fluids under the effect of mechanical forces. Rheology is referred to flow as a Greek word. Rheology can be applied to all types of materials, from gases to solids. Rheology of the fluids can be determined with two components such as the viscosity and the elasticity. Practically, rheology is based on viscosity measurements, characterization of flow behavior and determination of material structure. These subjects are vital in process design and product quality evaluation. In rheology, shearing of a substance is the most important criteria of flow behavior and structure. A sheared flow is a flow between parallel planes; a rotational flow between coaxial cylinders. To determine the viscosity of a material, the shearing must cause stationary flow of the material. The flow happened by rearrangement and deformation of particles and by breaking of bonds in the structure of the material [61-64].

Shear stress is the force that acts on the cell layer (on the wall) and could be defined as:

$$\sigma_{yx} = F / A \tag{6.1}$$

F: force (N)
A: area (m^2)
Shear rate which shows the velocity profile of the medium could be obtained as:

$$\varphi = d\varphi / dx \tag{6.2}$$

φ : shear rate (sec^{-1})
$d\varphi$: shear strain (m.sec)
dx : length (m)

So, the viscosity could be determined as:

$$\eta_a = \sigma_{12}/\varphi = \eta(\varphi) \tag{6.3}$$

σ : shear stress (N/m^2)
η : dynamic viscosity (Pa.sec)
φ : shear rate (sec^{-1})

Defining shear rate, shear stress and viscosity is related with classification of fluids directly.

6.1. Classification of Fluids

Fluids can be classified according to their mechanical features as Newtonian and Non-Newtonian fluids. Newtonian fluids have a constant viscosity dependent on temperature; however independent of the applied shear rate. Also, a Newtonian fluid could be determined by a single viscosity value at a specified temperature. Water, mineral and vegetable oils and pure sucrose solutions could be given as an example of Newtonian fluids. In addition, low-concentration liquids; for instance whole milk, skim milk and most single strength fruit juices, could be qualified as Newtonian fluids.

On the other hand, Non-Newtonian fluids that cannot be defined by a single viscosity value at a specified temperature have two types such as time-dependent and time-independent. Time-dependent fluid's viscosities are not changing with only shear stress but also changing with time. On the other hand, viscosity of time-independent fluids is not changing with time. The classification of fluids can be seen in Figure 6.1. Bingham plastic, pseudoplastic and dilatant fluids are Non-Newtonian fluids and they are time independent. Firstly, bingham liquid's behaviors look like solid under static conditions. An obtained force should be recognized for stimulating any flow which is called yield value. For instance, tomato ketchup could be given as an example. Not only simple but also very effective way of checking a fluid's possible plastic properties is turning the jar upside down. If the fluid is not going to flow by itself it probably has a significant yield value. If it flows by itself or very slowly, it probably has no yield value; however a high viscosity. Secondly, pseudoplastic liquid behavior is named as shear-thinning. As it can be seen in Figure 6.1, it has a decreasing viscosity with an increasing shear rate. Paints and emulsions could be given as examples. The main reason for shear thinning flow behavior is an increased shear rate deforming and rearranging particles, concluding in lower flow resistance and lastly lower viscosity. At last, dilatant fluid behavior is related with the shear-thickening liquids. For example, clay slurries, candy compounds, corn starch in water, and sand/water mixtures can be dilatant. They have increasing viscosity with an increase in shear rate. The flow behavior could be explained with the solvent acts as a lubricant between suspended particles at low shear rates; however it is squeezed out at higher shear rates, concluding in denser packing of the particles [61,62,64].

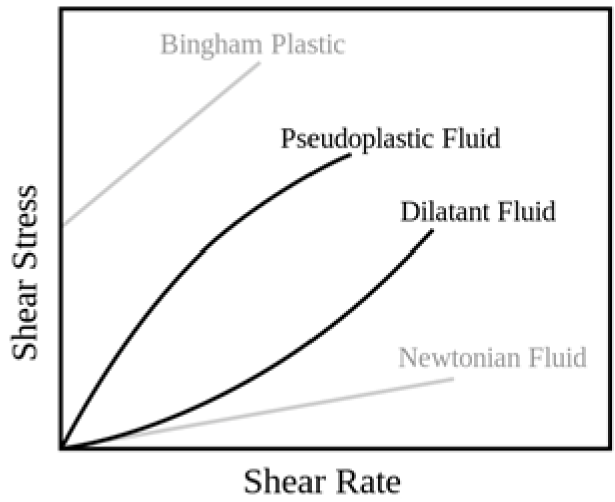


Figure 6.1 : Newtonian and non-newtonian fluids classification. [61]

7. THE STUDIES ABOUT NANOFIBER PRODUCTION BY SOL-GEL VIA ELECTROSPINNING IN LITERATURE

Nano-structured materials have been produced using a variety of different substances including polymers, ceramics, metals, composites, and hybrid materials. Recently, nanosized silica or silica based composites have drawn more and more attention for their potential applications in photonic crystal, electronic and thermal insulators, catalysis, biolabeling, magnetics, etc. Some characters of nanosized silica which are size, size distribution and morphology are of great importance to its applications. So, to seek facile fabrication methods which can effectively control these characters is very attractive and demanding to the scientists. Due to its low thermal conductivity, low thermal expansion coefficient, good thermal stability, and good mechanical properties, silica fiber has been widely used as a reinforcing fiber in composites and heat insulating materials. One way for obtaining the silica is sol-gel process. The main advantages of using sol-gel method compared with traditional glass melting or ceramic powder methods are higher purity and homogeneity of resultant matrices and the lower processing temperatures. Due to this properties, materials with a variety forms have been prepared through sol-gel method. Especially, nano-structured sol-gel derived materials have attracted attentions for a variety of applications. One of recently reported nano-structured materials obtained through sol-gel process is electrospun fibers. Electrospinning is a versatile, simple and economically feasible process for fabricating ultra-fine fibers from a wide range of materials. It is possible to produce silica-based nano-structured materials by electrospinning method [65-66]. Throughout this chapter, the fabrication of silica based nanofibers by electrospinning technique is reviewed.

Supplying best electrospinning conditions is the critical issue for getting homogenous nanofiber production. Therefore, there are many ways for proving these parameters. For example, one way is adding polymer to the sol-gel solution. According to Xia et al.[67], PVP is used for forming bioactive glasses. They investigated the influence of the polymer concentration, types of polymer and electric field strength on the fiber diameter. During the fabrication of the nanofibers, TEOS, TEP and $Ca(NO_3)_2 \cdot 4H_2O$ were stirred with HCl solution and ethanol. Then the sol was mixed with PVP and P123. After that the electrospinning is applied. They obtained nanofiber glasses in the system of SiO_2-P_2O_5-CaO. It was stated that the diameter and morphology of nanofiber glassess could be arranged by the polymer concentration, types of polymer and electric field strength [67].

Asgharnia and Alizadeh fabricated nanofiber glasses based on SiO_2–CaO–MgO–P_2O_5 system by using electrospinning. In this study, highly concentrated PVP solution was used. Then, they applied the calcination at 600 °C for 5 hours to reduce the diameter of nanofibers. Also, they performed a second calcination step at 800 °C to obtain glass-ceramic nanofibers [68].

According to Wang et al.[69], silicon oxycarbide (SiOC) fibers can be formed by electrospinning of the solution containing vinyltrimethoxysilane and tetraethoxysilane via sol–gel reaction. The aim of this study was to determine the influence of PVP on the spinnability. Also, in this study, nitric acid was used as an initiator instead of hydrochloric acid. Nitric acid is more effective; therefore its concentration is more important. On the contrary of other studies, the mixing time of sol and polymer solution was 4 hours. Then, electrospining was applied. The feed rate of solution was 2.5 ml/h, the voltage was 15 kV, and the distance between tip to collector was 10 cm. The calcination was applied at 1000 °C for 1 hour to supply high tensile modulus. As a result, they reached the optimum spinnability with a solution which has a mass ratio of PVP/alkoxides = 0.05 and a maximum spinnable time (t) of 50 min [69].

On the other hand, according to Lotus et al.[70], composite fibers of TiO_2–Al_2O_3 were fabricated by electrospinning to obtain template polymeric fibers followed by calcination. In this study, methanol is used for initiator instead of ethanol due to having a homogeneous solution. If ethanol would be utilized, white precipitate could be formed during the mixing of ethanol–PVP solution that did not electrospin well. However, methanol can easily evaporate. The electrospinning parameters were determined as 20 kV, 15 cm distance and a feed rate of 20 μl/min. They claimed that they produced nanofibers having the diameters between 150–400 nm [70].

Zhao et al.[71] stated that mesoporous silica nanofibers were produced by electrospinning method. Pluronic, P123 was utilized for the structure direction agent and polyvinyl pyrrolidone (PVP) was ensured to form refining nanofibers. It was found that the average diameters of the mesoporous silica fibers were about 200–300 nm [71].

There are other studies that used different polymers to increase spinnability instead of PVP. Chen et al.[72] reported that polyacrylonitrile (PAN) nanofiber membranes functionalized with calixarenes by electrospinning. The feed rate of polymer was 1 ml/h, the applied voltage was 20 kV, and the distance between tip to collector was 15 cm. In their study they practiced for determining the functional nanofibrous PAN membranes [72]. Another polymer which can increase the spinnability is PVA. As stated by Pirzada et al.[73] silica nanofibers were formed by the acid catalysis of a silica precursor solution and aqueous PVA. It was determined that the ratio of (silica/PVA) in solution affects the electrospinnability, fiber morphology, and diameter of fibers because of enhancing viscosity and conductivity of the solution [73].

In a similar study, Changlu et al.[74] fabricated fiber mats of organic–inorganic hybrids by using poly(vinyl alcohol) (PVA) and TEOS. In this study, H_3PO_4 used as a catalyst instead of HCl. In contrast to other studies, they did not apply any calcination process. The sample was only dried under vacuum for 12 h at 70 °C. It was reported that they produced the first fiber mats of organic–inorganic composite materials [74].

Another way for enhancing spinnability and also changing the morphology of nanofibers is adding surfactant. One of the main current surfactant is Triton-X. In addition to this, there are many different surfactants which could be used for nanofiber production. Aykut et al.[75] studied the effect of surfactants on the processability of electrospun polyacrylonitrile (PAN) nanofibers and their carbonized analogs. Used surfactants were Triton X-100 (nonionic surfactant, SF-N), sodiumdodecyl sulfate (SDS) (anionic surfactant, SF-A), and hexadecyltrimethylammonium bromide (HDTMAB) (cationic surfactant, SF-C). The solution which was applied contained 8 wt % of PAN. It was found that the addition of surfactant changes the properties of the electrospining solution such as viscosity, surface tension, and conductivity, therefore, it could easily affect the morphologies of the nanofibers. On the other hand, fiber breakage occured when the electrospining solution contained a nonionic surfactant and a bead-on-a-string structure could be seen in the case of a cationic surfactant sample. In addition to this, adding all of the surfactants provides more ordered structures after calcination [75]. On the other hand, another surfactant could be F127 to alter the morphology of fibers. He et al.[76] fabricated silica nanofibers with refined mesoporous microstructures by the combination of electrospinning and sol–gel methods. The aim was investigating the effect of the concentration of nonionic surfactant F127 on the morphology and mesoporous structure of silica nanofiber. Silica precursor was composed of TEOS, ethanol, water and HCl. The viscosity of the silica sols was adjusted via surfactant pluronic F127(EO106PO70EO106) additive to check the most stable electrospinning scenario and the sol effect on the fiber internal construction. On the contrary of other studies, 3-Mercaptopropyl trimethoxysilane(MPTMS) was also jointed to confirm the accessibility of functional group attachment in the internal pores. It was reported that the dimension and the distribution of the mesopores within the silica nanofibers became regular if low mass ratio of (F127/TEOS) could be set between 0.18–0.14 during the synthesis [76].

On the other hand, electrospining can be done without adding any polymer. According to Sakai et al.[65], electrospinning of the sol could be realized with the hydrolysis and polycondensation of TEOS to produce silica nanofibers. The silica sol was prepared by adding ethanol, TEOS, distilled water and hydrochloric acid in a molar ratio of 1:2:2:0.01. The mixture was heated to 80 ^{o}C for 8 hours. Then, it was cooled to 4 ^{o}C. After that electrospinning was applied to the sol. The optimum electrospinning parameters were determined as 25 kV for applied voltage, 10 cm for distance between tip to collector and 8 ml/h for feeding rate. According to these parameters, the viscosity of the solution was 0.14 Pa.s and the diameter of the nanofibers was 305 nm (the thinnest one) [65].

In another study, Min et al.[66] produced silica nanofibers including silver nanoparticles by the combination of sol-gel chemistry and electrospinning technique without using polymer. It was reported that silica nanofiber webs showed antibacterial activity [66].

Another study which was about the production of inorganic nanofiber without polymer was done by Choi et al. [77]. The silica sol was formed by tetraethyl orthosilicate (TEOS), distilled water, ethanol, HCl in a molar ratio of 1:2:2:0.01, respectively. The mixed solution was heated to 80 ^{o}C for half an hour and then the temperature decreased to room temperature. Then, the electrospinning was applied at a voltage between 10-16 kV. Results showed that silica nanofibers were fabricated with diameters between 200-600 nm [77].

8. MATERIALS AND METHODS

8.1. Materials

Tetraethyl orthosilicate (TEOS, ≥98.0%, Fluka), calcium nitrate tetrahydrate (Merck), aluminum nitrate (Merck) and magnesiumnitrate-hexahydrate (Merck) were used to prepare sol-gel glass (BG). Calcium nitrate tetrahydrate, aluminum nitrate and magnesiumnitrate-hexahydrate were utilized as salt components of the sol to be able to adjust the viscosity of the solution. Ethanol and hydrocloric acid were supplied from Merck. Polyvinylpyrrolidone (PVP, MW= 360,000 g/mol) was provided by Sigma Aldrich. Moreover, Triton X (Sigma Aldrich) was used to adjust the surface tension of the solutions. All chemicals were used as provided without further purification.

8.2. Viscosity Measurement

Rheological tests were performed using a Rheomat RHM180 rheometer that is a portable and stand-alone rotational viscometer, which utilizes a motor driven bob rotating in a fixed cup. The solution is sheared in the gap between bob and cup. The measured shear stress is utilized to calculate the viscosity. All measurements were performed at constant temperature of 25 ºC (± 0.2). The parameters determined throughout the rheometer studies were mixing ratio of sol and PVP, the weight ratio of salt over TEOS, the polymer solution ratio (v/v) and the concentration of Triton X. The parameters can be seen in Table 8.1. The mixing ratios of sol and PVP were chosen as 1:1, 1.5:1, and 2:1. The weight ratios of salt over TEOS were preferred as 1, 2 and 4. In addition to this, the polymer concentrations were selected as %2, %3 and %5. At last, for the last parameter Triton X concentrations were picked %2, %4.2 and %5 that can be seen in Table 8.1 directly. These values were chosen to make rheological analysis clearly.

Table 8.1 : Parameters for viscosity measurements.

Parameter			
Mixing ratio of sol-pvp (v/v, %)	1:1	1.5:1	2:1
Salt /TEOS (w/w, %)	1	2	4
Polymer solution (v/v, %)	2	3	5
TritonX concentration (%)	2	4.2	5

8.3. Preparation of Sol-gel Solution

The glass compositions in mole 70% SiO_2 and 30% CaO and 65% SiO_2, 25% Al_2O_3 and 10% MgO were prepared by hydrolysis and polycondensation reactions. Initially, tetraethoxysilane (TEOS) was added to the mixture of deionized water, ethanol, 1 M hydrochloric acid; then the mixture was allowed to react for 60 minutes for the acid hydrolysis of TEOS. After 60 minutes, calcium nitrate tetrahydrate was added to the stirring solution and mixed for 45 minutes. This sol-gel solution was coded as S1. The same procedure was also applied to be able to produce Al-Mg containing glass with the addition of both aluminum nitrate and magnesium nitrate hexahydrate in to the solution with 45 minutes intervals. The second sol-gel solution was coded as S2. Then the obtained solutions were stirred for one day to allow the completion of hydrolysis.

8.4. Preparation of Electrospinning Solution

PVP was dissolved in ethanol at a concentration of 3% at room temperature for one day during stirring. Then obtained sol-gel and polymer solutions were mixed with each other. The PVP/sol-gel ratio of the solutions were set as 2/1 and 1/1 (v/v). The obtained mixtures were stirred at room temperature for one day. Triton X (4.2% (w/w)) was added to solutions as a surfactant to decrease the surface tension before electrospinning. These mixtures were stirrred for 2 hours.

8.5. Electrospinning

The as-prepared solutions were transferred to a plastic syringe equipped with a flat stainless steel needle, which was connected to a high-voltage supply. For the S1 sample, the applied voltage to the needle tip was 16.5 kV. The flow rate was set as 3 ml/h by a syringe pump. Nonwoven electrospun fibers were deposited onto an aluminum foil wrapped around a grounded collector placed at a distance of 11 cm perpendicular to the needle tip. Electrospinning conditions for the S2 sample were set as: the feed rate of polymer 3 ml/hr, the voltage 17.5 kV and the distance between tip and collector 10 cm. Electrospinning procedure was performed under ambient conditions.

8.6. Heat Treatment

After the electrospinning process, the samples were removed from aluminum foil by a curved knife. Then the resultant fiber mats were dried at 37^{o}C for a couple of days to remove residual solvent and then transferred to a desiccator for further investigations. The samples were placed into the muffle furnace and heated at a rate of 5 ^{o}C/minute to 600 °C and held at this temperature for 3 hours to obtain glass fibers. Glass fiber samples were then cooled in the furnace.After calcination process PVP was removed from the fiber mat.

8.7. Characterization Techniques

8.7.1. Density measurement

The density of the produced samples were measured with gas displacement technique. This measurement was carried out with Micrometrics pcynometry system using helium as an inert gas.

8.7.2. Fourier transform infrared spectroscopy (FTIR)

Perkin Elmer FTIR Spectrum One B Spectrometer was used for the determination of the chemical structure of the produced glass nanofibers, both before and after calcination. The differences occurred in functional groups after calcination were detected by FTIR analysis. Each sample was analyzed by KBr pellets. The spectra were recorded between 650-4000 cm^{-1} and the functional groups were obtained.

8.7.3. Scanning electron microscopy (SEM)

Surface morphologies of the produced samples before and after calcinations were observed by Jeol JSM-5410 Model Scanning electron microscopy. Samples were coated with platinum before observation. Analysis was performed at 5 kV at different magnifications.

8.7.4. X-Ray diffraction analysis (XRD)

X-ray diffraction was utilized to determine the amount of amorphous glass present in the samples. X-Ray diffraction (XRD) patterns were recorded by Pan'alytical X'Pert Pro X-beams. Analyses were determined between $5° < 2\theta < 60°$ with an increment rate of 0.0167° and 26.67 s at 25 °C utilizing CuKa radiation. The resulting diffraction patterns were analyzed using a software package program. The phases were identified by comparing the peak positions and intensities with those in the JCPDS (Joint Committee on Powder Diffraction Standards) data files.

8.7.5. Nonflammability test

As stated in ISO 1716, a test specimen of obtained mass is burned under specified conditions, at constant volume, in an atmosphere of oxygen, in a bomb calorimeter calibrated by combustion of certified benzoic acid. Heat of combustion can be calculated under these situations which is determined on the basis of of the observed temperature increase, and also taking care of heat loss and the latent heat of vaporization of water.

The test appratus can be seen in Figure 8.1. The temperature is recorded every 0.01 K for 10 minutes as can be seen in Figure 8.2.

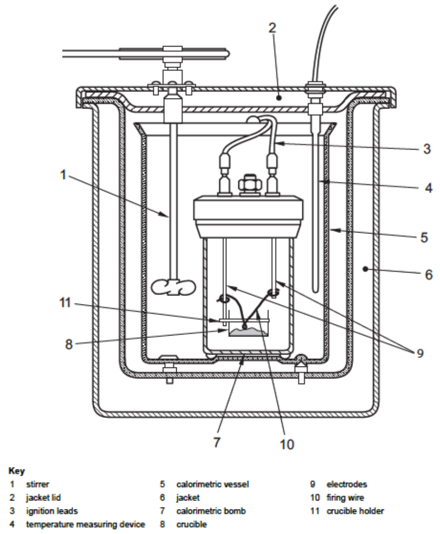


Figure 8.1 : Apparatus of the nonflammability test.

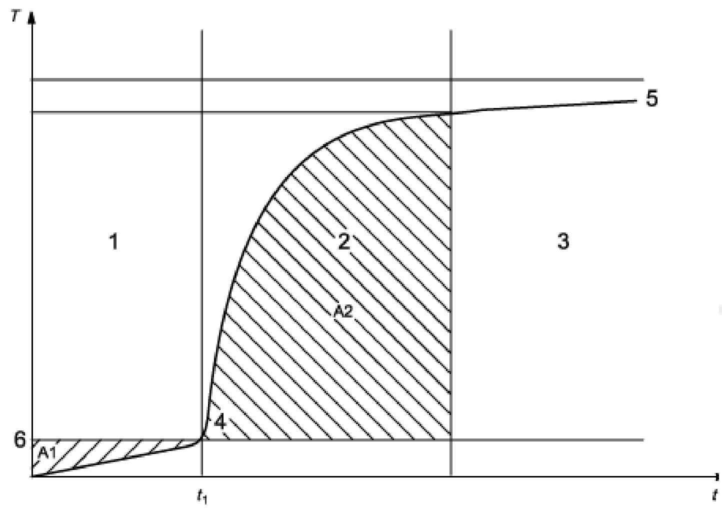


Figure 8.2 : Temperature/time curve of flammability test.

T: Temperature; t: time; 1 : preliminary period; 2 : main period; 3: final period; 4: ignition; 5 : Temperature of the jacket (T); 6 : Time when the temperature of the water in the crucible vessel is equal to the temperature of the jacket (T0).

After determining the unknown variables, the heat of combustion of the samples could be calculated by using Equation 8.1.

$$Q_{pcs}= (E(T_M-T_i+c)-b)/m \quad (8.1)$$

Where Q_{pcs} is the gross heat value of combustion, in MJ/kg; E is the water equivalent of the calorimeter, in MJ/kg; T_i is the initial temperature, in K; T_M is the maximum temperature in K; b is the correction, expressed in megajoules, required for the combustion heat of the “fuels” used during the test, i.e. firing wire, cotton thread, cigarette-making paper and benzoic acid or combustion aid; c is the tempearture correction factor, expressed in K; m is mass of the test specimen,in kg.

According to ISO 13501 classification of construction materials can be seen in Table 8.2.

Table 8.2 : ISO 13501 class of fire performances of construction materials.

Class	Test Method(s)	Classification criteria	Additional classification
A1	EN ISO 1182 And	$\Delta T \leq 30$ °C and $\Delta m \leq 50$ % and $t_1 = 0$ (i.e. no sustained flaming)	-
	EN ISO 1716	PCS ≤ 2.0 MJ /kg and PCS ≤ 2.0 MJ /kg and PCS ≤ 1.4 MJ /kg and PCS ≤ 2.0 MJ /kg	-
A2	EN ISO 1182 or	$\Delta T \leq 30$°C and $\Delta m \leq 50$ % and $t_1 = 20$ s	-
	EN ISO 1716 and	PCS ≤ 3.0 MJ /kg and PCS ≤ 5.0 MJ /kg and PCS ≤ 3.0 MJ /kg and PCS ≤ 4.0 MJ /kg	-
	EN 13823	FIGRA ≤ 120 W/s and LFS < edge of specimen and $THR_{600s} \leq 7.5$ MJ	Smoke production and Flaming droplets/particles
B	EN 13823 and	FIGRA ≤ 120 W/s and LFS < edge of specimen and $THR_{400s} \leq 7.5$ MJ	Smoke production and Flaming droplets/particles
	EN ISO 11925-2 : Exposure = 30s	$F_s \leq 150$ mm within 60 s	
C	EN 13823 and	FIGRA ≤ 250 W/s and LFS < edge of specimen and $THR_{400s} \leq 15$ MJ	Smoke production and Flaming droplets/particles
	EN ISO 11925-2 : Exposure = 30-s	$F_s \leq 150$ mm within 60 s	
D	EN 13823 and EN ISO 11925-2 : Exposure = 30s	FIGRA ≤ 750 W/s $F_s \leq 150$ mm within 60 s	Smoke production and Flaming droplets/particles
E	EN ISO 11925-2 : Exposure = 15s	$F_s \leq 150$ mm within 20 s	Flaming droplets/particles

8.7.6. Thermal conductivity test

Thermal conductivity of samples was measured with Armfield HT10XC Heat Transfer Service Unit. The measurement was applied with conventional heat flow meter setup based on ASTM C 518. This method is very successful for relatively low ($<$ 0.1 W/mK) thermal conductivity materials. It means that only a small amount of heat can pass through the material although a high temperature difference can occur between its two faces. These kind of materials are called as insulators that are suitable for using to reduce heat loss from a hot body to its surroundings. In the experiment, the heated and cooled parts were clamped tightly together with the cork disk in between to make a composite bar with the insulated disk of undetermined thermal conductivity sandwiched between two brass sections. The thermal conductivity coefficient was calculated on the basis of one-dimensional heat flow. The rate of heat transfer Q is given by Fourier's Law.

$$Q = k\,A\,\Delta T / L \tag{8.2}$$

Where Q is the heat flow; k is the thermal conductivity; A is the surface area; ΔT is the temperature difference and L is the thickness of the sample.

A constant heat flux was applied. Then the difference in temperature was measured over a period. By using the equation 8.2, thermal conductivity of the samples were calculated [78].

8.7.7. BET Analysis

The BET surface areas of the produced samples were determined by nitrogen adsorption at -196 ^{o}C using a surface analyzer (Quantachrome NOVA 1200 model). Prior to gas adsorption experiments, the samples were degassed under vacuum at 100 ^{o}C for a period of 1.5 h to remove the adsorbed atmospheric gases from the sample. According to BET surface area measurements, the surface area of the samples were determined.

9. RESULTS AND DISCUSSION

9.1. Pre-experimental Studies

Pre-experimental studies were performed to decide the optimum composition of sol-gel, polymer concentration, surfactant concentration and the to these studies, the mixing ratio was set as 2:1, polymer concentration mixing ratios of sol to polymer solution. These studies can be seen in Appendix A. According to Appendix A, polymer concentration was detected as %3 and the concentration of Triton-X was determined as %4.2. Furthermore, different sol compositions were prepared in this study. First composition was set as 70% SiO_2- 30% CaO (S1) and the second one was 65% SiO_2-25% Al_2O_3-10% MgO (S2).

9.2. Rheology of Solutions

During the process of electrostatic spinning, the rheological behaviour of polymer solutions is significantly influenced by the solution properties. In principle, the parameters contributing to the quality of nanofibrous webs can be divided roughly into four basic groups: polymer properties, solvent properties, polymer solution properties and the process parameters. Viscosity represents one of the crucial parameters characterizing the properties of a polymer solution. Solution concentration and viscosity are among the most important factors affecting the size and morphology of electrospun nanofibers. Therefore, one of the main aims of this thesis was to study rheological properties as a function of solution composition. For this purpose first of all, viscosity measurements of solutions were performed by altering shear rate, torque and shear stress in the meanwhile changing the parameters such as mixing ratio of sol and PVP solution, the weight ratio of salt/TEOS, polymer concentration and Triton X concentration. These measurements were given in Appendix B.

9.2.1. The effect of mixing ratio of sol-gel and polymer solution to the rheology of electrospinning solution

The effect of mixing ratios (1:1, 1.5:1 and 2:1) of sol-gel and polymer solution to the rheology of electrospinning solution was investigated. The densities of these mixed solutions were measured as 0.8505, 0.8561 and 0.8581 g/ml respectively. Figure 9.1. shows the plots of shear stress versus shear rate of the sol-gel and polymer solution in the mixing ratios of 1:1, 1.5:1 and 2:1. As seen from the Figure 9.1, the relationship between the shear rate and the shear stress is linear. The lines are passing nearly from the center. The interceptions could be assumed as zero. Therefore, these solutions can be considered as Newtonian type fluids. The plots of viscosity versus shear rate of the sol-gel and polymer solution in the mixing ratios of 1:1, 1.5:1 and 2:1 can be seen in Figure 9.2. It can be seen that viscosity stays constant while shear rate increases. This behavior also indicates that all the solutions are Newtonian type fluids. It was also detected that the viscosity of the electrospinning solution decreased with the increase in sol-gel content in the whole solution.

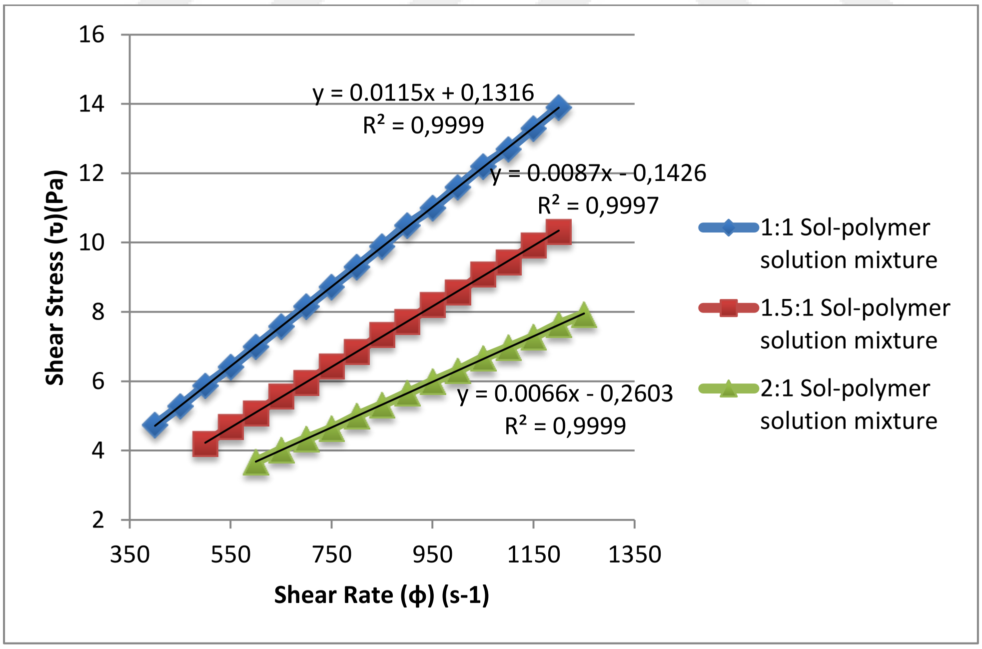


Figure 9.1 : Plots of shear stress vs. shear rate of sol-polymer solutions in the mixing ratios of 1:1, 1.5:1 and 2:1.

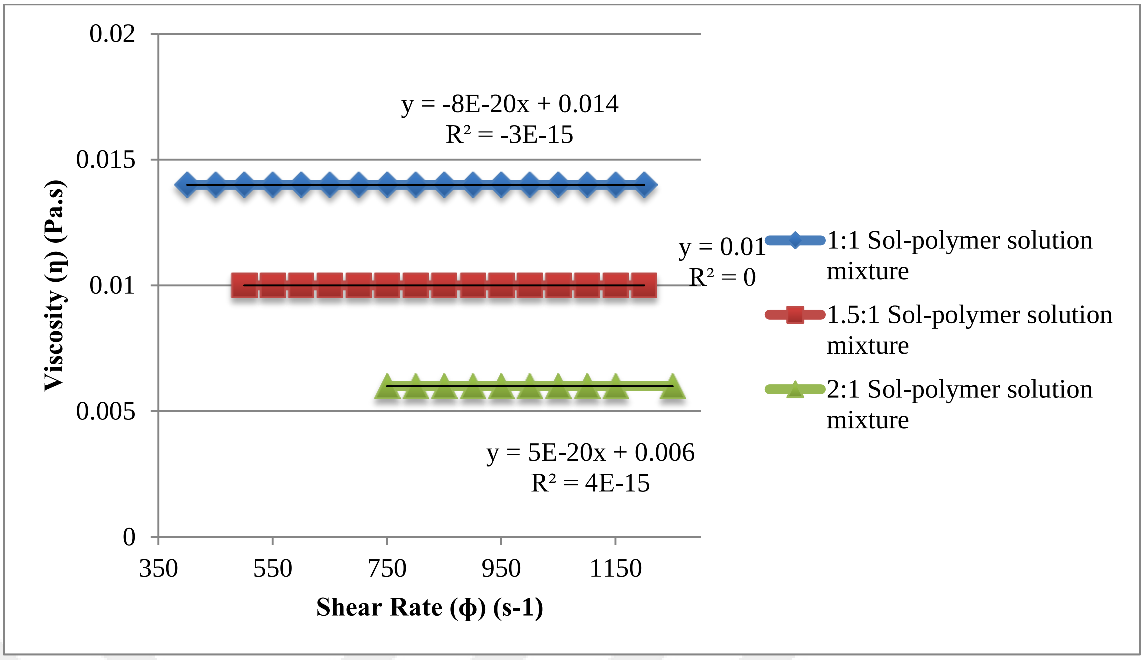


Figure 9.2 : Plots of viscosity vs. shear rate of sol-polymer solutions in the mixing ratios of 1:1, 1.5:1 and 2:1.

9.2.2. The effect of polymer concentration (wt.%) to the rheology of electrospinning solution

The effect of polymer concentration (in wt., 1%, 3% and 5%) to the rheology of electrospining solution was investigated. The densities of these solutions determined as 0.8510, 0.8512 and 0.8581 g/ml, respectively. The plots of shear stress versus shear rate of the polymer solutions in weight percentages of 1, 3 and 5 were given in Figure 9.3. As seen from the Figure 9.3, the relationship between the shear rate and the shear stress is linear. The lines are passing nearly from the center. The interceptions could be assumed as zero. Therefore, these plots indicate that all solutions show Newtonian behavior. The plot of viscosity versus shear rate of the polymer solutions (in wt., 1%, 3% and 5%) can be seen in Figure 9.4. As seen from Figure 9.4, the viscosity is stable as shear rate increases. This results also proved that all solutions are Newtonian type fluids. The viscosity measurements also revealed that the viscosity of the electrospinning solution increased with the increase in polymer (PVP) concentration. According to prelimenary studies of electrospinning, PVP concentration is very important parameter as it can be obtained from Appendices.

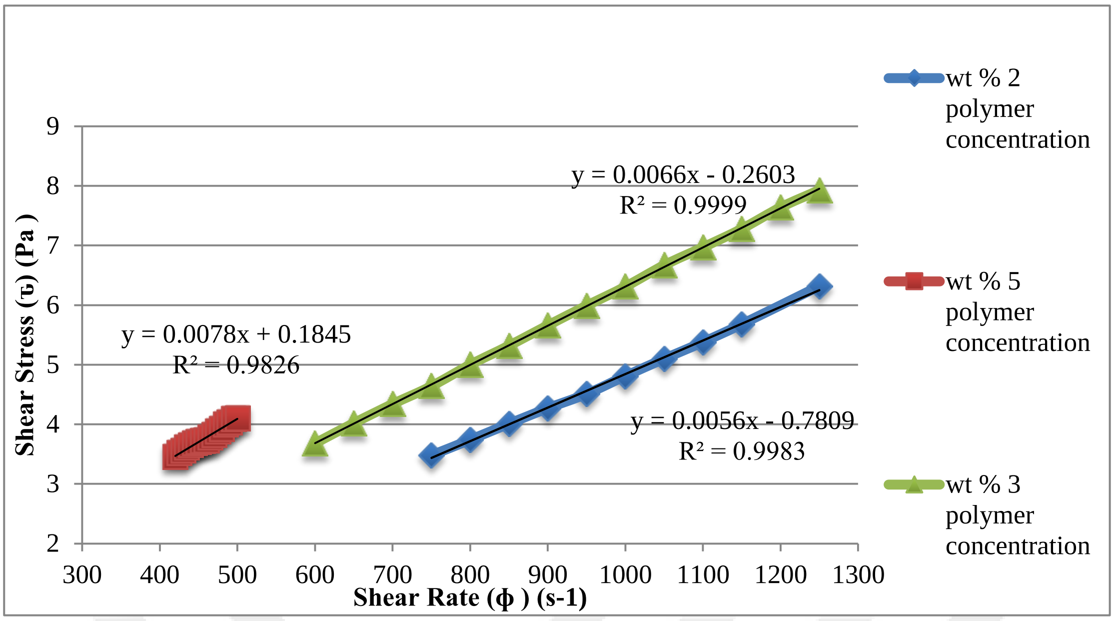


Figure 9.3 : The plots of shear rate vs. shear stress of the polymer solutions (concentrations in wt., 1%, 3% and 5%).

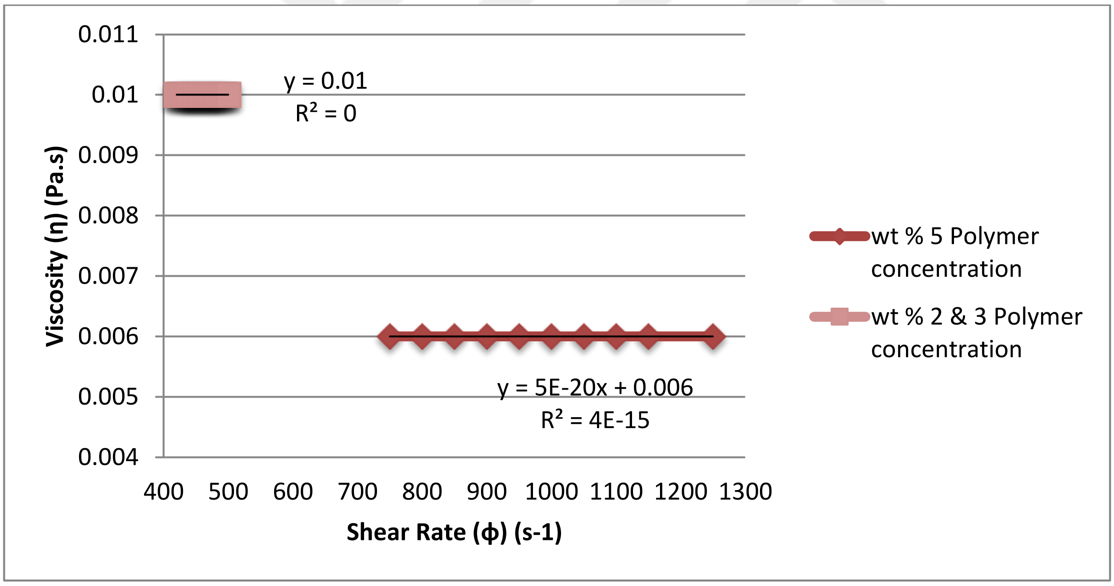


Figure 9.4 : The plots of viscosity vs. shear stress of the polymer solutions (concentrations in wt., 1%, 3% and 5%).

9.2.3. The effect of salt / TEOS ratio (w/w%) to the rheology of electrospinning solution

The effect of salt/TEOS ratio (in w/w%: 1, 2 and 4) to the rheology of electrospining solution was investigated. The densities of these solutions were measured as 0.8512, 0.8533 and 0.8596 g/ml, respectively. Figure 9.5. shows the plots of shear stress versus shear rate of solutions in different salt/TEOS ratios (in w/w%: 1, 2 and 4). As seen from the Figure 9.5, the relationship between the shear rate and the shear stress is linear. The lines are passing nearly from the center. The interceptions could be assumed as zero. Therefore, these solutions can be considered as Newtonian type fluids. The plots of viscosity versus shear rate of solutions in different salt/TEOS ratios (in w/w%: 1, 2 and 4) can be seen in Figure 9.6. It can be seen that viscosity stays constant while shear rate increases. This behavior also indicates that all the solutions are Newtonian type fluids. It was also determined that the viscosity of the electrospinning solution increased with the increase in the salt content.

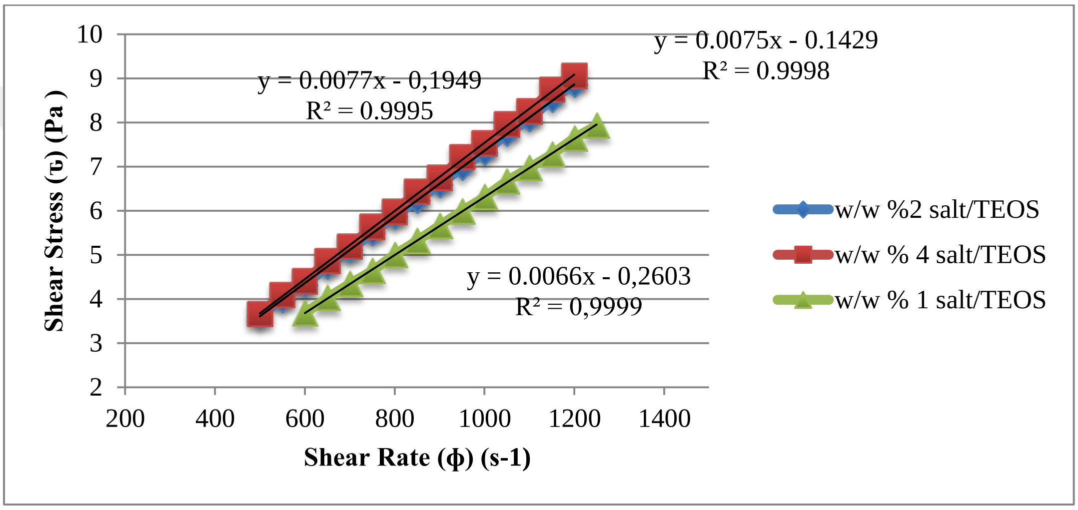


Figure 9.5 : The plots of shear rate vs. shear stress of solutions in different salt/TEOS ratios (in w/w%: 1, 2 and 4).

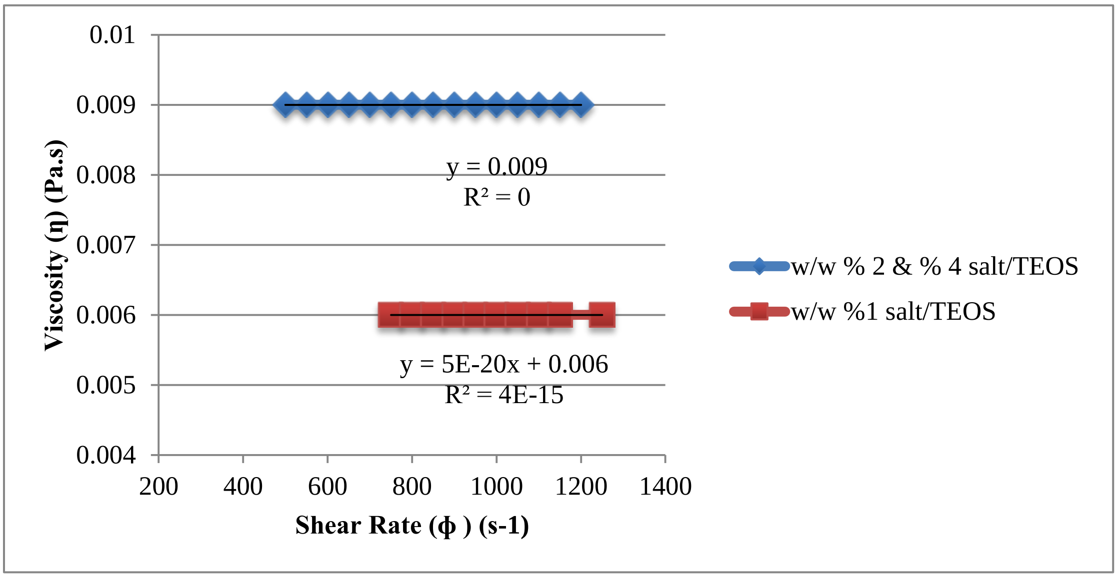


Figure 9.6 : The plots of viscosity vs. shear stress of solutions in different salt/TEOS ratios (in w/w%: 1, 2 and 4).

9.2.4. The effect of Triton X concentration (w/w%) to the rheology of electrospinning solution

The effect of surfactant concentration (in w/w%: 2, 4.2 and 5) to the rheology of electrospining solution was investigated. The densities of these solutions were determined as 0.8514, 0.8580 and 0.8628 g/ml, respectively. The plots of shear stress versus shear rate of the solutions in different surfactant concentrations (in w/w%: 2, 4.2 and 5) were given in Figure 9.7. As seen from the Figure 9.7, the relationship between the shear rate and the shear stress is linear. The lines are passing nearly from the center. The interceptions could be assumed as zero. Therefore, these plots indicate that all solutions show Newtonian behavior. The plot of viscosity versus shear rate of the solutions in different surfactant concentrations (in w/w%: 2, 4.2 and 5) can be seen in Figure 9.8. As seen from Figure 9.8, the viscosity is stable as shear rate increases. This result also proved that all solutions are Newtonian type fluids. The results also indicated that the viscosity of the electrospinning solution enhanced with the increase in concentration of surfactant.

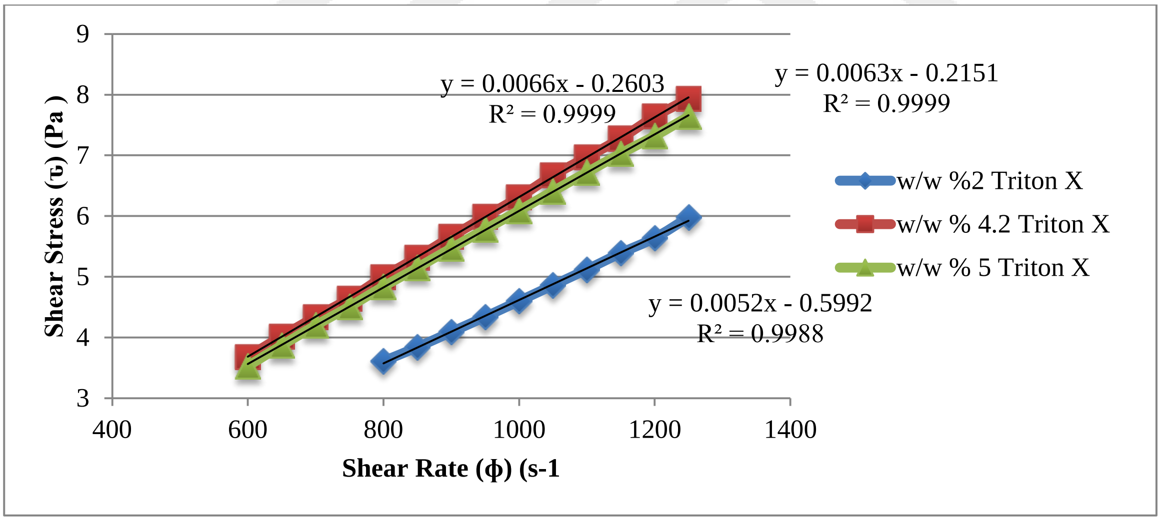


Figure 9.7 : The plots of shear rate vs. shear stress of the solutions in different Triton X concentrations (in w/w%: 2, 4.2 and 5).

In addition to this, from Figure 9.7, it can be obtained that if Triton X concentration is higher than %4.2, the line become to be similar each other. However, according to prelimenary studies of electrospinning which can be seen in Appendices, % 5 Triton X solution could not be spinned.

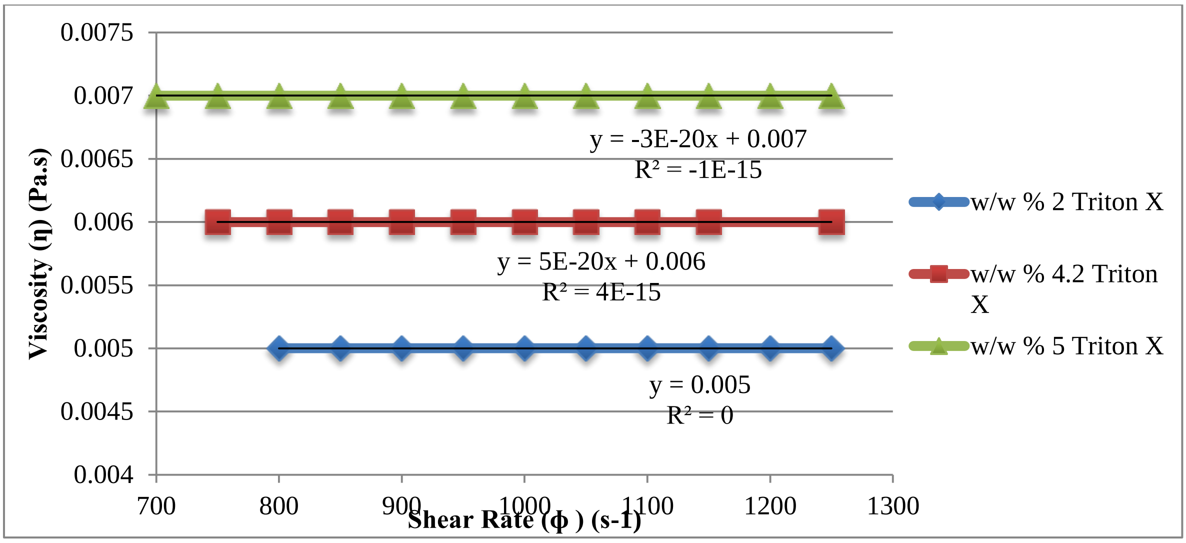


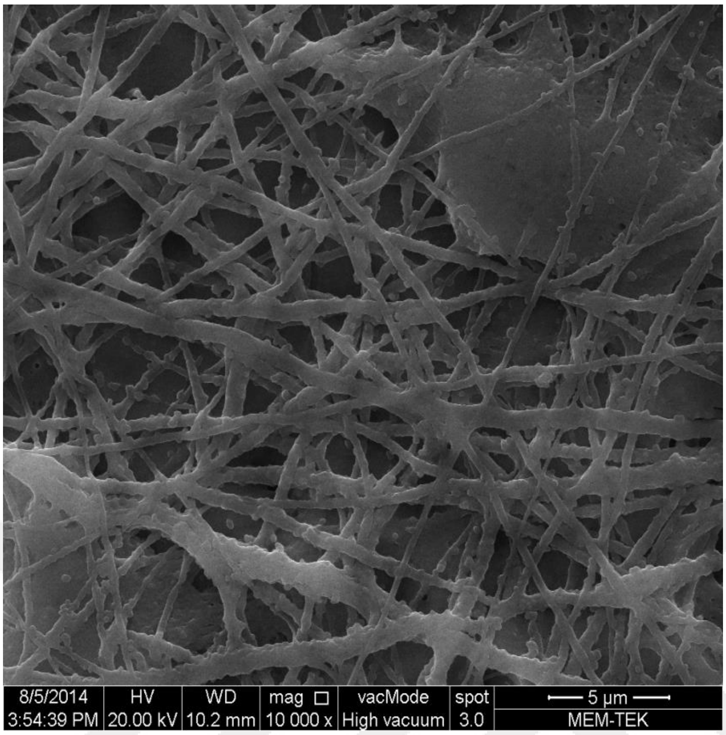
Figure 9.8 : The plots of viscosity vs. shear stress of the solutions in different Triton X concentrations (in w/w%: 2, 4.2 and 5).

In the light of the rheological and the prelimanary electrospinning studies (Appendix A), the optimum viscosity value of the solutions was 0.006 Pa.s to be able to perform the electrospinning process. The viscosity is one of the most important parameter for the electrospinning process.

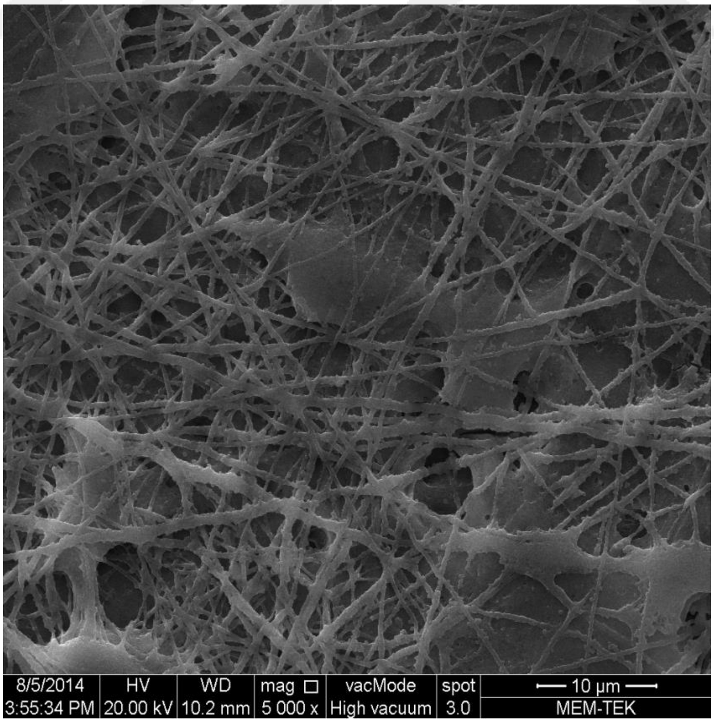
9.3. Characterization of the Produced Nanofibers

9.3.1. SEM analysis

The nanofibrous mats were prepared by the process of electrospinning, and their quality was evaluated using SEM analysis. Figure 9.9.(a) shows the SEM image of glass/PVP fibers that were electrospun from the final solution with 0.8514 g/ml concentration . SEM micrograph, given in Fig. 9.9(a), revealed that the sample S1 (before calcination) was composed nanofibers. The nanofibers randomly oriented throughout the structure. There were also some beads in sample S1 (before calcination). In comparison, a similar structure was also obtained for that sample after calcination. It is clear that the calcination process did not change the morphology of the nanofiber mat. The diameter of the electrospun fibers was measured by using Image J software (National Institute of Health, USA). The diameter of glass/PVP fibers for S1 was changed in the range between 37±10 nm and 87±10 nm. As can be seen from Figure 9.9(b), the diameter of fibers was reduced due to loss of PVP polymer after calcination at 600 ^oC. The diameter of fibers for S1 after calcination changed between 33±10 nm to 70±10 nm.



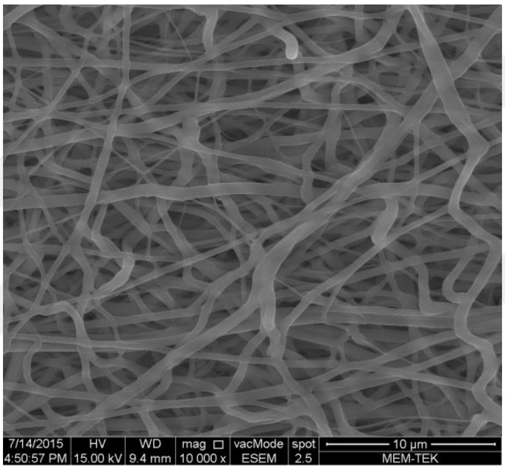
(a)



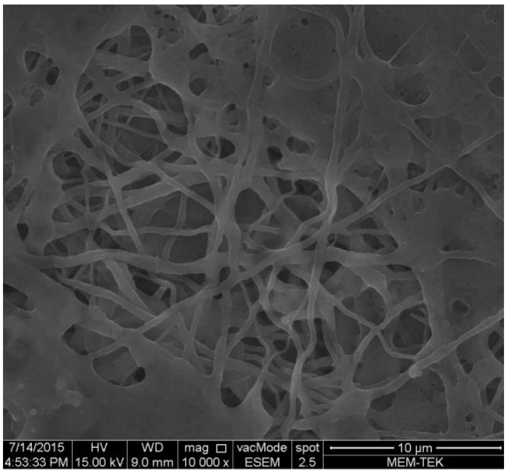
(b)

Figure 9.9 : SEM images of sample S1; (a) before calcination and (b) after calcination.

SEM micrograph, given in Fig. 9.10(a), revealed that the sample S2 (before calcination) was composed of randomly oriented, uniform, and bead free nanofibers. In comparison, a well-developed fibrous morphology with good electrospinnability was also obtained for the sample S2 after calcination. As can be seen from Figure 9.10, the average diameters of nanofibers were 82±8 nm and 59±8 nm for sample S2 before and after calcination, respectively. Sample S2 still maintained its fibrous morphology after calcination. However, because of the calcination process some parts of the nanofiber mat have smooth surfaces. This means that the calcination temperature can be too high for this sample.



(a)



(b)

Figure 9.10 : SEM images of sample S2; (a) before calcination and (b) after calcination.

9.3.2. FTIR analysis

FTIR analysis was performed to identify the surface properties of the fibrous mats. Figure 9.11. shows the FTIR Spectrum of PVP/glass nanofiber mat for sample S1 before and after calcination. Characteristic infrared bands of PVP was given in Table 9.1 [44]. As it can be seen in Figure 9.11, PVP presents the characteristic bonds at 3232, 1631, 1270, and 923 cm^{-1} corresponding to –OH, C=O, C-N, and H-CH=CH_2, respectively that is well accord with the literature according to Table 9.1. The sample S1 (before calcination) have all chacteristic bonds on it, however, after calcination all characteristic bonds lost because of the decomposition of the polymer. On the other hand, both before and after calcination, it can be seen that, the nanofiber mat contains glass fomation due to 798 adsorbtion peaks corresponding to Si-O bonds which is well accord with literature [67].

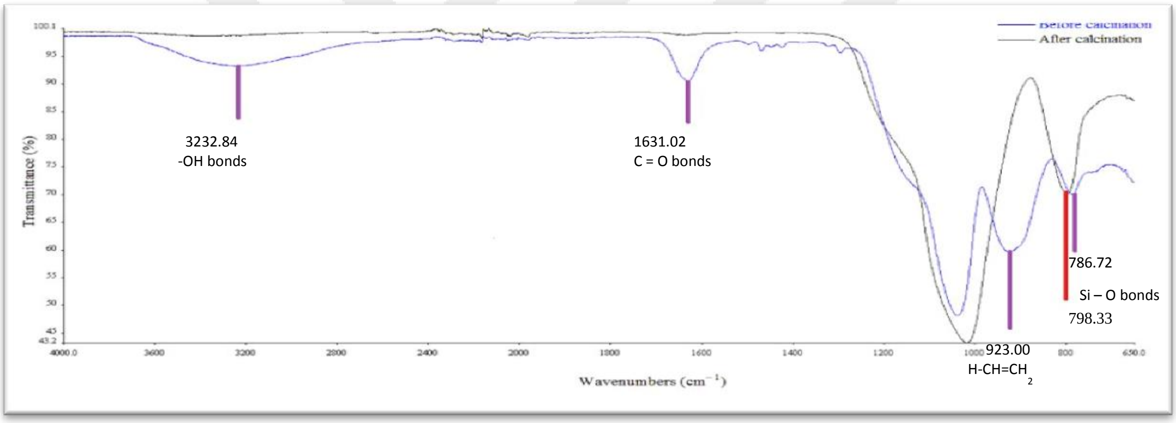


Figure 9.11 : FTIR spectra of PVP/glass nanofiber before and after calcination (sample S1).

Table 9.1: Characteristic infrared bands of PVP. [74]

Wavelength (cm^{-1})	Structure of Bonds
2949	Asymmetric CH_2 stretching
2865	Symmetric CH_2 stretching
1727	Carbonyl stretching
1293	C-O and C-C stretching (in crystalline phase)
1240	Asymmetric C-O-C stretching
1170	Symmetric C-O-C stretching

As it like in Figure 9.11, the characterics bond of PVP could be seen in Figure 9.12 for sample S2 before calcination. According to FTIR graphic, there is a strong adsorption peak at 3237 cm^{-1} which corresponds the –OH bands of PVP. Adsorption at 1634 cm^{-1} is referred to the vibrations of C=O from PVP, which gives a strong support to the hydrogen bond. Moreover, for adsorption peak at 937 cm^{-1} is stretching mode of $CH=CH_2$. However, these bonds were disapeared after calcination. The peaks at 798 and 667 cm^{-1} were associated with the Si-O-Si vibrations. In addition, the broad band from 3000 cm^{-1} to 3600 cm^{-1} is related with the occuring silanol groups (Si-OH) on the surface of prepared nanofibers [74].

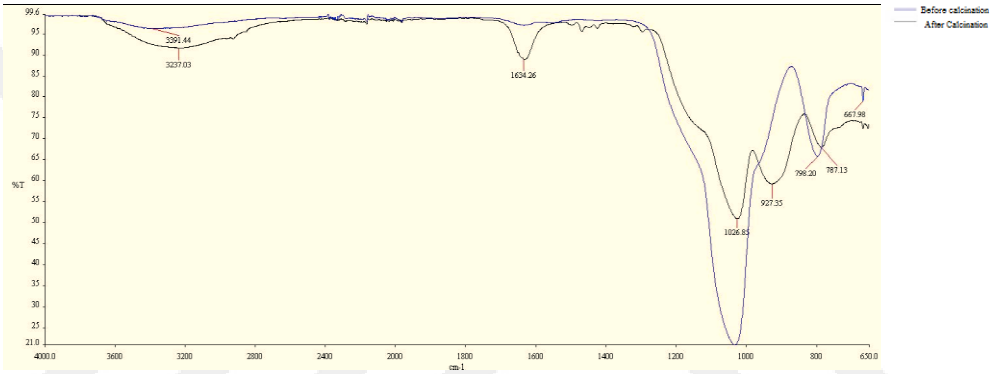


Figure 9. 12 : FTIR spectra of PVP/glass nanofiber before and after calcination (sample S2).

9.3.3. XRD analysis

The XRD measurements were performed to examine the crystalline structure of the fibrous mats. It is known that both glass and PVP show no peak in XRD pattern, which indicates its amorphous nature. Figure 9.13 shows the XRD pattern of PVP glass nanofiber sample S1 before and after calcination. The XRD Spectrum of glass nanofibers showed a broad diffraction peak at approximately 24 °, corresponding to characteristic of amorphous construction of SiO_2 [78]. At last, it is reported that the amourphous phases present in the fibres both before and after calcination treatment with using X-ray diffraction analysis (XRD). Therefore the results observed from the XRD measurements strongly recommend that the crystallinity of PVP.

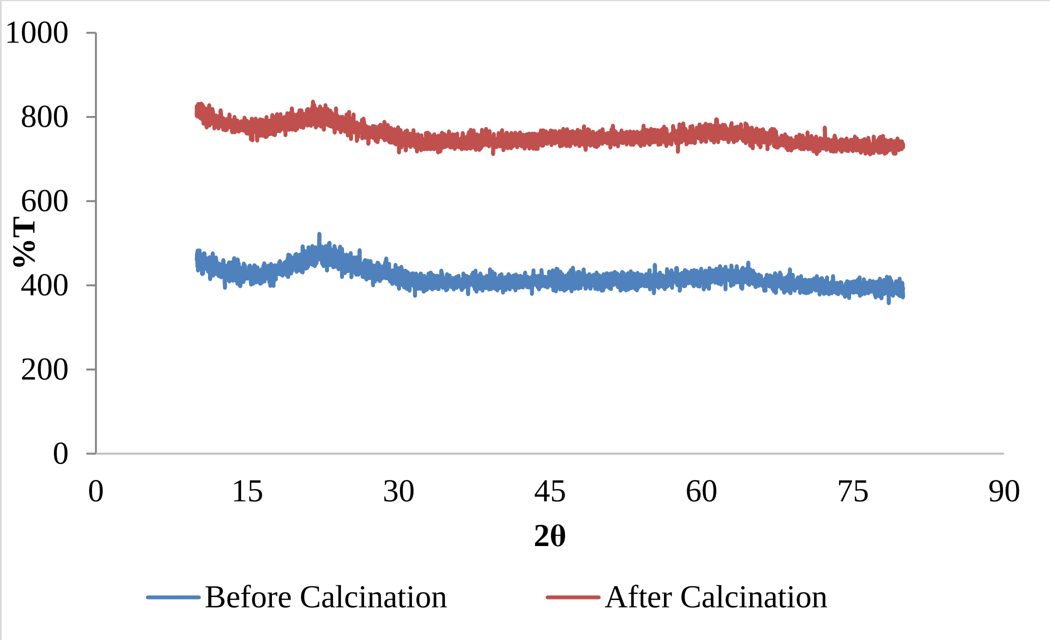


Figure 9.13 : XRD patterns of PVP glass nanofiber before and after calcination (sample S1).

XRD patterns of sample S2 were given in Figure 9.14. As can be seen from Figure 9.14, it has the same peak which exists at approximately 24° that showing characteristic of amorphous construction of SiO_2 when compared with sample S1. XRD studies showed that the glass structure was obtained succesfully for both samples S1 and S2.

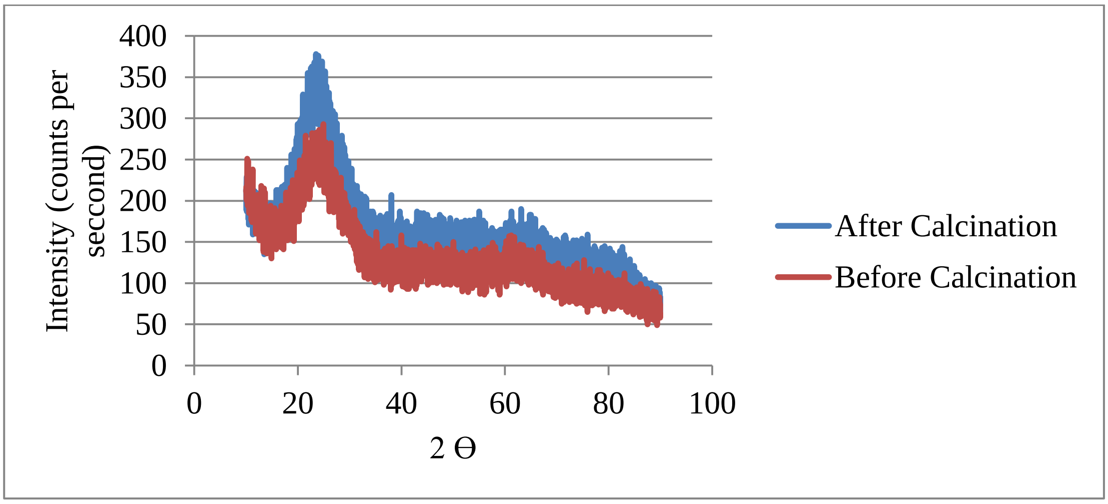


Figure 9.14 : XRD patterns of PVP glass nanofiber before and after calcination (sample S2).

9.3.4. Nonflammability test

As it stated before, according to ISO 1716, the fire performance of specimen can be obtained with the calculation of the gross heat value of combustion. The Q_{pcs} values for the samples S1 and S2 were calculated as 0.098 and 0.099 MJ/kg, respectively (see Appendix C). According to fire performance table (Table 8.2), both samples S1 and S2 can be classified as A1 Class since the Q_{pcs} values are smaller than 2 MJ/kg. On the basis of these analysis both samples can be considered as nonflammable.

9.3.5. Thermal conductivity measurements

Thermal conductivities of the samples S1 and S2 were measured as 0.049 and 0.034 W/mK, respectively. According to EN 14303, these values must be lower than 0.065 W/mK. Therefore, the produced nanofiber mats can considered as insulation materials which well accords with the CE Standarts to being insulation material [79].

9.3.6. Density measurements

Table 9.2 shows the density values of the samples. As it can be seen in Table 9.2, the density values of samples S1 and S2 were measured as 2419.9 kg/m^3 and 2033.7 kg/m^3, respectively. According to ISO EN 13162 standard, the density of glass wool is between 10 kg/m^3 and 100 kg/m^3[80]. The obtained values for both samples are 20 times greater than that of glasswool. This result showed that the low thermal conductivity values obtained with high density values which is a desired property for insulation materials with high capacity.

Table 9.2: The density measurement results.

Sample	S1	S2
Sample Mass	0.1110 g	0.1760 g
Temperature	22.63 °C	22.47 °C
Sample Volume		
Average	0.0459 cm^3	0.0865 cm^3
Standard Deviation	0.0001 cm^3	0.0001 cm^3
Sample Density		
Average	2.4199 g/cm^3	2.0337 g/cm^3
Standard Deviation	0.0027 g/cm^3	0.0023 g/cm^3

9.3.7. BET analysis

BET analysis were also used to further characterization of the fibrous mats. Surface areas were measured based on both single point and six point analysis. Nanofiber mat S1 gave specific surface area of 39.54 m^2/g and a density of 2419.9 kg/m^3, while S2 nanofiber mat was found to have a specific surface area of 36.32 m^2/g and a density of 2033.7 kg/m^3. These results indicated that the increase of fiber diameter reduces specific surface area and density. According to Ogasawara and Kohyama, the surface area of glass wool is 0.27 m^2/g [81]. The surface areas of the produced nanofiber glass samples are approximately 140 times bigger than that of glass wool. This is the most important reason of production of nanofibers.

Table 9.3: BET analysis results.

	Six Point BET	
	S1	S2
Specific Surface Area (m^2/g)	39.54	36.32
Surface Area (m^2)	1.38	1.93
	Single Point BET	
	S1	S2
Specific Surface Area (m^2/g)	38.16	36.84
Surface Area (m^2)	1.33	1,96

10. CONCLUSION AND RECOMMENDATIONS

10.1. Conclusions

This study was conducted to produce a nanofiber material, which could be suitable to be used as an insulation material for industrial applications. For this purpose, nanofiber glass was successfully fabricated via electrospinning process. The following conclusions can be drawn:

1. According to rheological mesurements, the characteristic of electrospinning solution was Newtonian Fluid due to linear relation between shear stress and shear rate; and also constant viscosity observed with the increase in shear rate.

2. The viscosity of the electrospinning solution decreased with the increase in sol-gel concentration in the mixture solution.

3. The viscosity of the electrospinning solution increased with the increase in the salt content.

4. The viscosity of the electrospinning solution increased with the increase in polymer (PVP) concentration.

5. The viscosity of the electrospinning solution enhanced with the increase in concentration of surfactant (Triton X).

6. According to rheological studies, the optimum electrospining viscosity which is required to produce homogenous nanofiber structure was found as 0.006 Pa.s.

7. SEM results showed that sample S2 has continous nanofibers structure with an average diameter of 59±8 nm after calcination while S1 has some beads both before and after calcination.

8. XRD results revealed the amorphous glass structure of the samples.

9. FTIR results indicated that the PVP decomposed after calcination and glass structure of the samples was also detected.

10. Nanofiber samples can be classified as A1 Class nonflammable insulation materials according to nonflammability test.

11. Thermal conductivity measurements showed that both samples can be considered as insulation materials according to CE Standards, due to having thermal conductivities smaller then 0.065 W/mK.

Overall results indicated that both samples have superior properties as insulation materials since they are nonflammable, have high density, low thermal conductivity and high spesific surface area. On the other hand, it was observed that sample S2 is more durable than the sample S1 since sample S2 has a better nanofiber morphology according to SEM results.

10.2. Recommendations

Based on the experimental data collected so far, the following future works are suggested:

1. Further investigations on mechanical properties will provide valuable information to be able to determine the suitable application areas for the produced nanofiber materials.

2. The composition of the sol-gel can be changed to produce more durable samples.

3. These produced samples can be coated with titanium to increase the durability of them.

4. These produced samples can be added into glass wool to improve the properties of glass wool, for instance; decreasing of thermal conductivity of glass wool while increasing the density of it.

5. Having less molecular weight PVP can be investigated to produce smooth and no bead nanofiber.

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APPENDICES

APPENDIX A

1) According to below composition ;

sol:
0.5 ml 1 M HCl
7.18 ml TEOS
0.543 ML TEP
1. 48 gr $Ca(NO_3)_2$. $4H_20$
50 ml EtOH

polymer:
3 g PVP
1.2 g F127
20 ML EtOH

Result : Prepared solution was so viscous, so that it could not be spinned.

1) For sol, 70S30C ratios were used. % 17 PVP solution was used. It was still so viscous. And it was dried and it could not be spinned.
2) For sol, 70S30C ratios were used again. To supply electrospinning, less viscous solution preaparing was aimed. Therefore, % 4.3 PVP polymer solution was prepared. It was spinned however, there were many interruptions and branches occured.
3) Later, less viscous forming was aimed. So that, % 3.8 PVP, % 3.4 PVP and % 2.9 PVP solutions were used for producing electrospinning solution. All three solutions were tried to electrospin. The best result was obtained for % 2.9 PVP. For the other ones, interruptions were existed.
4) In the next study, 1 g PVP, 0.4 gr P123 were added. F127 was utilized instead of P123. 8 solutions were prepared. The aim was long term production and to obtain sample for calcination.

Table A.1 : Experimental Results.

Composition (wt)	Electrospinning conditions	Result
% 1.33	15 kV, 10 cm, 3 ml/hr	Slim fibers were produced. The amount of production is good.
% 2	17 kV, 10 cm, 3 ml/hr	Thicker fibers were produced. Branches were determined.
% 0.96	No production	
% 1.43	17 kV, 10 cm, 3 ml/hr	Thicker fibers were produced.
% 0.08	No production	
% 1.25	15 kV, 10 cm, 3 ml/hr	Slim fibers were produced. The amount of production is good.
% 0.74	No production	
% 1.1	15 kV, 10 cm, 3 ml/hr	Slim fibers were produced. The amount of production is good.

Result : Below % 1 PVP solution could not be spinned because of they could not gain the surface tension due to their low viscosity.

Improvement : According to these results, in the following studies % 3 PVP solutions were used. Furthermore, for setting the surface tension surfactant Triton X was utilized (as % 1 weight of sol PVP mix solution).

5) For sol, 10 ml EtOH, 1M O.1 ml HCl, 0.08 ml TEOS were added. After 45 minutes, for homogenization, 0.03606 gr $Ca(NO_3)2.4H_2O$ was added. On the other hand, % 3 PVP polymer solution was prepared. After two hours, these two solutions were mixed. Before 2 hours of electrospinning, surfactant Triton X (as % 1 weight of

sol PVP mix solution) was added. The electrospinning conditions were 14 kV as voltage, 10 cm for distance between tip and collector, 3 ml/hr as the feed rate of polymer.

Result : Interruptions were observed. However, there were fiber production.

Improvement: The amount of surfactant should be changed and the effect of the concentration of surfactant on fiber production should be investigated.

6) With the same sol and polymer sol composition, the concentration of surfactant varied such as % 1.8, %2, %2.2, %2.4, %3, % 4.2, % 5.2. Here is the aim to find the smallest distance between tip and collector for supplying bigger fiber production.

Table A.2 : Experimental Results.

Surfactant concentration	Electrospinning conditions	Fiber production
% 1.8	At 17 kV 11.5 cm	Smaller
% 2	At 17 kV 10.5 cm	Smaller
% 2.2	At 14 kV 10.5 cm	Better
% 2.4	At 14 kV 10 cm	Good Taylor cone, better fiber production
% 3	At 14 kV 11 cm	Good Taylor cone, better fiber production
% 4.2	At 14 kV 10 cm	Good Taylor cone, best fiber production
% 5.2	At 14 kV 11 cm	Fiber was dried. Branches were occured, the amount surfactant was too much.

Result : % 2.4 and % 4.2 are the best usable surfactant concentrations for best electrospinning conditions. Its supplied for gaining surface tension and forming best Taylor cone.

Improvements : With this two surfactant concentrations, long term production were possible, and the features of two samples both before calcination and after calcination were investigated.

7) For sol, 10 ml EtOH, 1M 0.1 ml HCl, 0.08 ml TEOS were added. After 45 minutes, to be sure that the homogenization occured , 0.03606 gr $Ca(NO_3)2.4H_2O$ was added. On the other hand, % 3 PVP polymer solution was prepared. After two hours, these two solution were mixed. Before 2 hours from electrospinning, surfactant Triton X as weight % 2.4 was added to sol-PVP mixture. The electrospinning conditions were 14 kV as voltage, 10 cm for distance between tip and collector, 3 ml/hr as the feed rate of polymer.

After the electrospinning, the sample was got through from aluminum foil. The sample was kept waiting at ambient conditions for 1 day. On the following day, the sample was put in the oven for calcination at 600^oC for 3 hours with 2^oC/min.

Result : The sample was too small. Furthermore, it was too fragile.

Improvement : % 4.2 surfactant would be tried for recovering the quality of the sample.

8) For sol, 10 ml EtOH, 1M 0.1 ml HCl, 0.08 ml TEOS were added. After 45 minutes, to be sure that the homogenization occured, 0.03606 gr $Ca(NO_3)2.4H_2O$ was added. On the other hand, % 3 PVP polymer solution was prepared. After two hours, these two solutions were mixed. Before 2 hours from electrospinning, surfactant Triton X as weight % 4.2 was added to PVP mix solution. The electrospinning conditions were 14 kV as voltage, 10 cm for distance between tip and collector, 3 ml/hr as the feed rate of polymer.

After the electrospinning, the sample was got through from aluminum foil. The sample was kept waiting at ambient conditions for 1 day. On the following day, the sample was put in the oven for calcination at 600^oC for 3 hours with 2^oC/min.

Result : The sample produced with % 4.2 Triton X was bigger than produced with % 2.4 Triton X. Furthermore, it was still too fragile.

Improvement : Here after, % 4.2 Triton X will be used for forming best Taylor cone. The another idea will be increasing the amount of sol, while decreasing the polymer solution.

9) The same sol and polymer composition was utilized. % 4.2 Triton X was used. However, the mixing ratio of sol and PVP solution was changed such as 2:1, 2.5:1, 3:1, 3.5:1, 4:1, 5:1.

Table A.3 : Experimental Results.

Mixing ratio	Electrospinning conditions	Fiber production
2:1	At 17 kV 10 cm	Good fiber production
2.5:1	At 17 kV 10 cm	Good fiber production
3:1	Could not be spinned (it could not gain surface tension)	
3.5:1	Could not be spinned (it could not gain surface tension)	
4:1	Could not be spinned (it could not gain surface tension)	
5:1	Could not be spinned (it could not gain surface tension)	

Result : Samples with 3:1 and bigger than 3:1 ratios could not be spinned. 2:1 and 2.5:1 could be used.

10) For sol, 10 ml EtOH, 1M 0.1 ml HCl, 0.08 ml TEOS were added. After 45 minutes, to be sure that the homogenization occured, 0.03606 gr $Ca(NO_3)2.4H_2O$ was added. On the other hand, % 3 PVP polymer solution was prepared. After two hours, these two solutions were mixed by the ratio of 2:1. Before 2 hours from electrospinning, surfactant Triton X as weight % 4.2 was added. The electrospinning conditions were 17 kV as voltage, 10 cm for distance between tip and collector, 3 ml/hr as the feed rate of polymer.

After the electrospinning, the sample was got through from aluminum foil. The sample was kept waiting at ambient conditions for 1 day. On the following day, the sample was put in the oven for calcination at 600^oC for 3 hours with 2^oC/min.

Result : The sample was still to fragile and small.

Improvements : The composition of sol should be changed because of the brittleness. The TEOS / EtOH ratio should be changed for providing more fiber production.

11) The EtOH / TEOS ratio could be 1.59 or 1. Therefore, the compostion was completely changed. In adition, mixing ratios were changed too. In addtion to this,

%3 PVP polymer solution was used. Again, % 4.2 Triton X was added before 2 hours from electrospinning

For both ratios of EtOH/TEOS (1:1 and 2:1) were tried.

Table A.4 : Experimental Results.

Sol composition	Mixing ratio	Electrospinning conditions	Fiber production
25 ml EtOH 1 M 0. 25 ml HCl 15.73 ml TEOS 0.0902 $Ca(NO_3)2.4H_2O$	1:1	No fiber production, it was dried.	
	2:1	At 16.5 kV, 12 cm	Contunious and good fiber production. Good Taylor cone
10 ml EtOH 1 M 0.1 ML HCl 10 ml TEOS 0.03606 $Ca(NO_3)2.4H_2O$	1:1	At 17 kV, 12.5 cm	Sample was taken
	2:1	At 17 kV, 12.5 cm	Sample was taken

Result : After the SEM results, it could be seen that, the second compostion was not spinned, it was similar to electrospraying. The best result was obtained with the second ratio.

Improvement : With this best result, long term production should be done, and all analyses should be made.

12) For sol, 10 ml EtOH, 1M 0.1 ml HCl, 5 ml TEOS were added. After 45 minutes, to be sure that the homogenization occured, 0.03606 gr $Ca(NO_3)2.4H_2O$ was put. On the other hand, % 3 PVP polymer solution was prepared. After two hours, these two solutions were mixed by the ratio of 2:1. Before 2 hours from electrospinning, surfactant Triton X as weight % 4.2 was added. The electrospinning conditions were 16.5 kV as voltage, 12 cm for distance between tip and collector, 3 ml/hr as the feed rate of polymer.

After the electrospinning, the sample was scraped from an aluminum foil. The sample was kept waiting in ambient conditions for 1 day. On the following day, the sample was put in the oven for calcination at 600^oC for 3 hours with 2^oC/min. The production lasted nearly 4 hours.

Result : The sample was big enough for testing.

Improvement: A more durable sample with better properties should be researched.

13) S GLASS has high modulus and its strength is better. The composition of this glass is %65 SiO_2, %25 Al_2O_3 and %10 MgO. The ratios distilled water/(TEOS+salts)=10/1 and EtOH/distilled water=100/1 were used.

According to these calculations, for sol, 10 ml EtOH, 1M 0.1 ml HCl, 6.3 ml TEOS were added. After 45 minutes, to be sure that the homogenization occured, 0.963 gr $Mg(NO_3)2.6H_2O$ and 2.72 gr $Al(NO_3)3.9H_2O$ were added. On the other hand, % 3 PVP polymer solution was prepared. These solution was prepared by 2 items.

After two hours, these two solutions were mixed by the ratios of 1:1 and 2:1. 2 hours before electrospinning, surfactant Triton X as weight % 4.2 was added. The electrospinning conditions were 17.5 kV as voltage, 11 cm for distance between tip and collector, 3 ml/hr as the feed rate of polymer.

After the electrospinning, the sample was scraped from an aluminum foil. The sample was kept waiting in ambient conditions for 1 day. On the following day, the sample was put in the oven for calcination at 600°C for 3 hours with 2°C/min. The production lasted nearly 4 hours.

Result : Both samples, before calcination and after calcination, have good strength and high modulus. However, after the SEM analysis, it could be seen that, the one produced with 2:1 sol/ polymer solution was not spinned.

Improvement: All analyses will be applied to the sample which was produced by electrospinning of 1:1 sol/polymer solution.

APPENDIX B

Table B.1 : The influence of the mixing ratio of sol and polymer solution (v/v).

Shear Rate (γ), (s^{-1})	Torque, M (mN.m)			Shear Stress, τ (Pa)			Viscosity, μ (Pa.s)		
	1:1	1.5:1	2:1	1:1	1.5:1	2:1	1:1	1.5:1	2:1
400	0.359	-	-	4,74	-	-	0.014	-	-
450	0.401	-	-	5,28	-	-	0.014	-	-
500	0.446	0.318	-	5,87	4.19	-	0.014	0.01	-
550	0.486	0.355	-	6,41	4.68	-	0.014	0.01	-
600	0.531	0.38	0.28	7,0	5.07	3.68	0.014	0.01	0.007
650	0.576	0.422	0.305	7,58	5.57	4.02	0.014	0.01	0.007
700	0.618	0.453	0.329	8,15	5.96	4.34	0.014	0.01	0.007
750	0.662	0.488	0.352	8,73	6.44	4.64	0.014	0.01	0.007
800	0.705	0.518	0.378	9,3	6.85	5	0.014	0.01	0.007
850	0.749	0.556	0.404	9,89	7.34	5.32	0.014	0.01	0.007
900	0.793	0.586	0.426	10,5	7.72	5.66	0.014	0.01	0.007
950	0.837	0.622	0.454	11,0	8.2	5.99	0.014	0.01	0.007
1000	0.879	0.648	0.478	11,6	8.57	6.31	0.014	0.01	0.007
1050	0.926	0.689	0.505	12,2	9.09	6.67	0.014	0.01	0.007

Table B.1 (continued) : The influence of the mixing ratio of sol and polymer solution (v/v).

Shear Rate (γ), (s^{-1})	Torque, M (mN.m)			Shear Stress, τ (Pa)			Viscosity, μ (Pa.s)		
1100	0.964	0.716	0.527	12,7	9.44	6.97	0.014	0.01	0.007
1150	1.01	0.753	0.55	13,3	9.93	7.28	0.014	0.01	0.007
1200	1.05	0.784	0.579	13,9	10.3	7.64	0.014	0.01	0.007
1250	-	-	0.603	-	-	7.93	-	-	0.007

Table B.2 : The influence of the ratio of salt/TEOS (w/w).

Shear Rate (γ), (s^{-1})	Torque, M (mN.m)			Shear Stress, τ (Pa)			Viscosity, μ (Pa.s)		
	%1	%2	%4	%1	%2	%4	%1	%2	%4
400	-	-	-	-	-	-	-	-	-
450	-	-	-	-	-	-	-	-	-
500	-	0.275	0.277	-	3.62	3.65	-	0.009	0.009
550	-	0.303	0.311	-	3.99	4.09	-	0.009	0.009
600	0.28	0.328	0.332	3.68	4.32	4.4	0.007	0.009	0.009
650	0.305	0.359	0.37	4.02	4.74	4.86	0.007	0.009	0.009
700	0.329	0.387	0.393	4.34	5.11	5.97	0.007	0.009	0.009
750	0.352	0.418	0.428	4.64	5.5	5.63	0.007	0.009	0.009
800	0.378	0.444	0.453	5	5.87	5.97	0.007	0.009	0.009
850	0.404	0.473	0.487	5.32	6.24	6.43	0.007	0.009	0.009
900	0.426	0.501	0.51	5.66	6.59	6.74	0.007	0.009	0.009
950	0.454	0.528	0.546	5.99	6.98	7.21	0.007	0.009	0.009
1000	0.478	0.555	0.571	6.31	7.32	7.52	0.007	0.009	0.009
1050	0.505	0.588	0.603	6.67	7.75	7.95	0.007	0.009	0.009
1100	0.527	0.613	0.625	6.97	8.08	8.25	0.007	0.009	0.009

Table B.2 (continued) : The influence of the ratio of salt/TEOS (w/w).

Shear Rate (γ), (s^{-1})	Torque, M (mN.m)			Shear Stress, τ (Pa)			Viscosity, μ (Pa.s)		
1150	0.55	0.646	0.665	7.28	8.52	8.74	0.007	0.009	0.009
1200	0.579	0.674	0.686	7.64	8.86	9.05	0.007	0.009	0.009
1250	0.603	-	-	7.93	-	-	0.007	-	-

Table B.3 : The influence of the polymer concentration.

Shear Rate (γ), (s^{-1})	Torque, M (mN.m)			Shear Stress, τ (Pa)			Viscosity, μ (Pa.s)		
	%2	%3	%5	%2	%3	%5	%2	%3	%5
550	-	-		-	-		-	-	
600	-	0.28		-	3.68		-	0.007	
650	-	0.305		-	4.02		-	0.007	
700	-	0.329		-	4.34		-	0.007	
750	0.264	0.352		3.48	4.64		0.006	0.007	
800	0.285	0.378		3.74	5		0.006	0.007	
850	0.304	0.404		4.01	5.32		0.006	0.007	
900	0.322	0.426		4.27	5.66		0.006	0.007	
950	0.342	0.454		4.51	5.99		0.006	0.007	
1000	0.364	0.478		4.81	6.31		0.006	0.007	
1050	0.386	0.505		5.1	6.67		0.006	0.007	
1100	0.408	0.527		5.38	6.97		0.006	0.007	
1150	0.43	0.55		5.68	7.28		0.006	0.007	
1200	0.478	0.579		6.32	7.64		0.006	0.007	
1250	-	0.603		-	7.93		-	0.007	

Table B.4 : The influence of the polymer concentration for % 5.

Shear Rate (γ), (s^{-1})	Torque, M (mN.m)	Shear Stress, τ (Pa)	Viscosity, μ (Pa.s)
420	0.262	3.45	0.01
425	0.265	3.52	0.01
430	0.269	3.56	0.01
435	0.274	3.61	0.01
440	0.277	3.65	0.01
445	0.282	3.7	0.01
450	0.276	3.62	0.01
455	0.279	3.68	0.01
460	0.283	3.73	0.01
465	0.286	3.78	0.01
470	0.291	3.82	0.01
475	0.294	3.88	0.01
48	0.303	3.92	0.01
485	0.306	4	0.01

Table B.4 (continued) : The influence of the polymer concentration for % 5.

Shear Rate (γ), (s^{-1})	Torque, M (mN.m)	Shear Stress, τ (Pa)	Viscosity, μ (Pa.s)
490	0.306	4.03	0.01
495	0.31	4.09	0.01
500	0.11	4.1	0.01

Table B.5 : The influence of surfactant (TritonX) concentration.

Shear Rate (γ), (s^{-1})	Torque, M (mN.m)			Shear Stress, τ (Pa)			Viscosity, μ (Pa.s)		
	%2	%4.2	%5	%2	%4.2	%5	%2	%4.2	%5
550	-	-	-	-	-	-	-	-	-
600	-	0.28	0.267	-	3.68	3.53	-	0.007	0.007
650	-	0.305	0.293	-	4.02	3.87	-	0.007	0.007
700	-	0.329	0.318	-	4.34	4.2	-	0.007	0.007
750	-	0.352	0.342	-	4.64	4.51	-	0.007	0.007
800	0.274	0.378	0.367	3.61	5	4.84	0.005	0.007	0.007
850	0.292	0.404	0.391	3.84	5.32	5.15	0.005	0.007	0.007
900	0.31	0.426	0.416	4.09	5.66	5.47	0.005	0.007	0.007
950	0.329	0.454	0.44	4.34	5.99	5.78	0.005	0.007	0.007
1000	0.35	0.478	0.461	4.6	6.31	6.09	0.005	0.007	0.007
1050	0.368	0.505	0.488	4.86	6.67	6.41	0.005	0.007	0.007
1100	0.388	0.527	0.511	5.12	6.97	6.72	0.005	0.007	0.007
1150	0.408	0.55	0.532	5.39	7.28	7.03	0.005	0.007	0.007
1200	0.428	0.579	0.557	5.64	7.64	7.32	0.005	0.007	0.007
1250	0.454	0.603	0.579	5.98	7.93	7.64	0.005	0.007	0.007

APPENDIX C

Table C.1 : 1st sample flammability test temperature/time results.

	T (°C)	T
Prelimenary Experiment	26.0116	6.01 (t_1)
Main Experiment	26.0786	11.01
Final Experiment	26.8731	19.01
Tmax	26.9199	24.01

T_i: 25.7 °C (T1)

c= $(t-t_1)*T_2-t_1*T1$

c=(8-6.01)*-(26.8731-26.0786)-6.01*(26.0116-25.7)

c=-7.66

Q= (2.6*10-5 (26.9199-25.7+273-7.66)-0.0477*10-3)/0.07

Q=0.098 megajoules per kg

Table C.2 : 2nd sample flammability test temperature/time results.

	T (°C)	T
Prelimenary Experiment	23.7441	6.11 (t1)
Main Experiment	23.7544	11.09
Final Experiment	24.5009	18.13
Tmax	24.5065	23.10

Ti= 22.7 °C

c= $(t-t_1)*T_2-t_1*T_1$

c=(7.4-6.11)*-(24.5009-23.7544)-6.11*(23.7441-22.7)

c=-6.1433

Q= (2.6*10-5 (24.5065-22.7+273-6.1433)-0.0477*10-3)/0.07

Q=0.099 megajoules per kg

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PRESENTATIONS ON THE THESIS:

- Akkaya C. İpek, Küçükbayrak S., Erol M. (2015). Glass Nanofiber Material as an Insulating Material. İstanbul, Turkey. International Conference on Advance in Composite Materials and Structures (Oral Presentation)

SUBMITTED ABSTRACTS :

- Akkaya C. İpek, Küçükbayrak S., Erol M. (2015). Glass Nanofiber Material as an Insulating Material. İstanbul, Turkey. *International Conference on Advance in Composite Materials and Structures* (OralPresentation)
- Taygun M. E., Akkaya I., Gönen S. Ö., Küçükbayrak S., (2015). Glass Nanofiber Material as an Insulating Material. İstanbul, Turkey. *International Advances in Applied Physics and Materials Science Congress & Exhibition* (OralPresentation)