ISTANBUL TECHNICAL UNIVERSITY ★ GRADUATE SCHOOL OF SCIENCE ENGINEERING AND TECHNOLOGY

SYNTHESIS AND ENCAPSULATION OF NANOSILICA WITH Poly(AAm-co-AMPS) AND APPLICATION ON VISCOSE FABRIC FOR PILLING

M.Sc. THESIS

Ebru CELEBI

Department of Nanoscience and Nanoengineering

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İSTANBUL TEKNİK ÜNİVERSİTESİ ★ FEN BİLİMLERİ ENSTİTÜSÜ

NANOSİLİKA SENTEZİ VE Poli(AAm-ko-AMPS) İLE KAPSÜLLENMESİ, VİSKON KUMAŞLARIN TÜYLENME SORUNUNUN GİDERİLMESİ

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ARALIK 2015

Ebru-Celebi, a **M.Sc.** student of ITU **Institute of / Graduate School of Science Engineering and Technology** student ID **513121003**, successfully defended the **thesis/dissertation** entitled "**SYNTHESIS AND ENCAPSULATION OF NANOSILICA WITH Poly(AAm-co-AMPS) AND APPLICATION ON VISCOSE FABRIC FOR PILLING**",which she prepared after fulfilling the requirements specified in the associated legislations, before the jury whose signatures are below.

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Date of Submission : 17 November 2015
Date of Defense : 22 December 2015

To my family,

FOREWORD

This thesis written for my Master degree in Nanoscience and Nanoengineering at the Istanbul Technical University. It is including researches and studies about nanoscience, polymer chemistry and textile chemistry. I tried to combine my textile knowledge from my work and nanoscience and polymer knowledge from my master degree.

With a deep sense of gratitude, I wish to express my sincere thanks to all the people who made this work possible.

Foremost, I am deeply indebted to my supervisor, Prof. Dr. A. Sezai Sarac, for his kind support and guidance. It was a great pleasure to me to conduct this thesis under his supervision. His guidance helped me in all the time of research and writing of this thesis.

I wish to express my special gratitude to Eksoy Chemicals family, who give me a chance to study M.Sc.

My sincere thanks also goes to Neda Soydan who gained a great and special place in my life. I want to thank for her guidance, support, encouragement, patience and sincerity.

I warmly thank to my friends; Ayşe Martin, Didem Varol and Ezgi Yurdakul. They always supported and helped me in all stages of this thesis.

Last but not the least, I would like to thank my family for their support, encouragement and endless patience. They are my greatest assets which I have in my life.

December 2015

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ABBREVIATIONS

AMPS	**:** 2-Acrylamido-2-methylpropane sulphonic acid
AAm	**:** Acrylamide
TEOS	**:** Tetraethylorthosilicate
AIBN	**:** 2,2′-Azobis(2-methylpropionitrile)
NH_4OH	**:** Ammonium Hydroxide
HF	**:** Hydrogen Fluoride
Co	**:** Copolymer
M_w	**:** Molecular Weight
M_n	**:** Number Average Molecular Weight
Tg	**:** Glass Transition State
CTA	**:** Chain Transfer Agent
FTIR	**:** Fourier Transform Infrared Spectroscopy
ATR	**:** Attenuated Total Reflectance
GPC	**:** Gel Permiation Chromatography
SEM	**:** Scanning Electron Microscope
EDX	**:** Energy Dispersive Detector
TEM	**:** Transmission Electron Microscopy
EDS	**:** Energy Dispersive
TG/DTA	**:** Thermogravimetric and Differential Thermal Analysis
BSL	**:** Bursting Strength Loss
nm	**:** Nanometer

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SYNTHESIS AND ENCAPSULATION OF NANOSILICA WITH POLY(AAm-co-AMPS) AND USAGE ON VISCOSE FABRIC FOR PILLING PROBLEM

SUMMARY

Viscose fabric is prefered because of high moisture capacity, elasticity and user comfort. In recent years consume of viscose is increased due to high cost of cotton production. Nevertheless, demand has not reached the expected level yet, because shortness of use life. The reason is viscose fabric's pilling, tensile strength loss and the wrinkle problem. There is no permanent, cheaper and easy to apply product on the market to completely resolve these problems. Due to gain the permanent features may be possible during the fiber synthesis and it may be possible by using certain chemicals or crosslinking of the synthesized chemical to the fiber . Chemicals which are using for synthesis of fiber are not preferred because unpredictable changes of the some of fiber properties. Generally carcinogenic chemicals are used as crosslinker. Polymers has become an essential point in the textile chemical, however solely polymers or copolymer also can not show sufficient effect for some of the issues. Silica is an available resource for this subject owing to improve the resistance of fabric to abrasion by entering between the fibers. Therefore silica needs a functional groups to bond the fabric structure for showing washing resistance and it may be possible with creating a hybrid structure with silica. However, it is difficult to ensure accordance and stability between the organic and inorganic structures. Today, nanostructures are started to use in textile chemicals same as other technologies. In nanoscale; structures performance, synergy and also accordance are increasing. Nano hybrid structures have been a solution in this point. Likewise a nano-hybrid polymer can be the remedy for the problems of viscose fabric. Because of size and functionality of core structure, it can show good stability and dispersity by entering the fabric fibers.

NANOSİLİKA SENTEZİ VE POLİ(AAm-ko-AMPS) İLE KAPSÜLLENMESİ, VİSKON KUMAŞLARIN TÜYLENME SORUNUNUN GİDERİLMESİ

ÖZET

Viskon kumaşlar yüksek nem tutma kapasitesi, elastisitesi ve kullanıcı rahatlığı nedeniyle tercih edilmektedirler. Viskon %14 civarında rutubet toplamakta ve şişme özelliğinden dolayı %80-120'ye kadar su emebilmektedir. Viskon pamuktan çok daha yüksek olan bu nem alma özelliğinden dolayı boyanmaya ve baskıya çok elverişlidir ve renkler çok parlak görünür. Doğal liflerin üretiminin her geçen sene artmasına rağmen, nüfus talebini karşılayamaması dolayısıyla viskon,modal,bambu gibi rejenere liflerin de üretimi önem kazanmaktadır. Zaman içerisinde tekstil mamullerinin maliyetleri de düşürülmeye çalışıldığından viskon üretim teknolojileri geliştirilmiş ve böylelikle viskonun üretim rekabeti arttırılarak pazar içindeki payını koruması sağlanmıştır. Günümüzde gelinen noktada hoş tutuma, parlak bir görünüme ve dökümlülüğe sahip olan viskona talep git gide artmaktadır. Yeni üretim teknolojileri ile yeşil üretim faaliyetlerine de başlayan viskon üreticileri ; kayın ağacı gibi ağaçların kabuklarını , tekstil atıklarını ve çeşitli kaynakları kullanarak rejenere elyaf üretimlerini arttırmaktadırlar. Pamuklu mamulun satın alındıktan sonra çamaşır makinasında yıkama sıcaklığının viskona göre 10-20˚C yüksek olması; ayrıca pamuklu tekstil ürünleri için kurutma makinaları kullanılırken viskonun narin yapısından dolayı kurutma makinalarında işlem görmesinin tercih edilmemesi dolayısıyla; pamuğun kullanım esnasında viskona göre enerji tüketimi neredeyse iki kat olarak belirlenmiştir. Tekstil fabrikalarında ise pamuklu kumaşlara kasar yapılması zorunluluğu ve viskona göre pamuğun boyama sürelerinin daha uzun olması dolayısıyla son ürüne gelen enerji maliyeti pamuk için yine her zaman daha yüksek olmaktadır. Pamuğun yetiştirilmesinden nihai tekstil ürününün oluşmasına kadar geçen süreçte de ciddi anlamda su ve enerji tüketimi yapılmaktadır. Su ve enerji dünyanın en büyük sorun yaratan tükenişteki kaynakları olduğundan dolayı dünyada hızlı bir şekilde alternatif elyaf kullanımına gidişat görülmektedir. Bu

nedenle dünyada doğal sentetik elyaf karışımlarının üretimi gitgide artmakta, pamuk üretimi gitgide azalmaktadır. Pamuğun yerini tutabilecek alternatif elyaf için arayışlar da sürmektedir ve alternatifler arasında pamuğa en yakın olan elyaf viskondur. Buna rağmen, viskonun kullanım ömrünün kısa olması nedeniyle beklenen talep seviyesine henüz ulaşamamıştır. Kullanım ömrünün kısa olmasının temel sebepleri ise tüylenme, mukavemet kaybı ve buruşma sorunudur. Bu sorunlar giderilirse kullanımı artacak ve pamuğun işlenmesi sırasında harcanan enerjiye göre ciddi anlamda tasarruf sağlanacaktır. Elyaflara kalıcı bazı özellikler kazandırmak ancak elyaf sentezi esnasında mümkün olabilir. Bazı kimyasallar kullanarak ya da sentezlendikten sonra kullanılacak kimyasalı elyafa çapraz bağlamak yoluyla yapılabilir. Elyaf sentezinde kullanılan kimyasallar elyafın ön görülemeyen bazı özelliklerini de değiştirdiğinden tercih edilmemektedir. Çapraz bağlamak için kullanılan kimyasalların da geneli kanserojendir. Elyaf sentezinde kullanılan kimyasallar elyafın öngörülemeyen bazı özelliklerini de değiştirdiğinden tercih edilen bir durum değildir. Bu çalışmada viskon ve pamuk elyaflarının yapısal özellikleri incelenip ; tekstil işletmelerince kolay uygulanabilecek bir apre işlemi ile viskon polimerinin modifiye edilerek doğal pamuk selulozik yapısına yaklaştırılması hedeflenmiştir. Bu sayede viskonun yüksek su alma ve esneklik özelliğini pamuğa göre avantajlı kalacak, sürtünmeye karşı olan dayanımı da geliştirilmiş olacaktır. Tezdeki amacımız viskonun modifikasyonunu tekstil işletmelerinde yeni ve farklı bir makina,teçhizat kullanımı gerekmeden kolay bir şekilde tamamlamak ve herhangi kanserojen bir bileşen kullanmamaktır. Başlangıç olarak projede viskona kazandırılması amaçlanan özellikler; viskonun boncuklanmasını önlemek, sürtünmeye karşı olan direncini arttırmaktır. Polimerik yapılar başlangıçtaki elyaf elyaf kaymasını azaltır ayrıca mukavemeti düşük olan kumaşlarda iplik uçları, bağlı oldukları diğer iplikleri sürtünme ile daha kolay bir şekilde bırakırlar. Pamuklu kumaşlarda selülaz enzimi zayıf iplik uçlarının kumaştan boncuklanma olmadan ayrılmasını sağlar, çoğu deterjanda da bir miktar selülaz enzimi bulunur. Selülaz enzimlerinin rejenere seluloz üzerinde kullanımları başarısızdır. Dolayısıyla viskon için boncuklanmayı önlemek amacıyla kullanılacak kimyasal kalıcı bir polimer kaplama olarak düşünülebilir. Polimerler tekstil kimyasalları içersinde vazgeçilmez bir noktaya gelmiştir. Ancak bazı konularda tek başına polimerler ya da kopolimerler de yeterli etkiyi gösterememektedir. Bu konuda silika kullanılabilir bir kaynak olabilir çünkü silika lifler arasına girerek viskon kumaşın aşınmaya karşı gösterdiği

direnci arttırmaktadır. Ancak silika tek başına kullanıldığında, kumaş yüzeyinde ve içersinde homojen bir dağılım sağlayamamakta ve yıkama dayanımı da gösterememektedir. Bunun için, silikanın viskon kumaşa bağlanmasını sağlayacak fonksiyonel gruplara ihtiyacı vardır. Bu da hibrit bir yapı oluşturarak mümkün olabilir ancak organik ve inorganik yapılar aradasındaki uyum ve stabiliteyi sağlamak da zor bir süreçtir. Günümüzde her teknolojide olduğu gibi tekstil kimyasallarında da nanoyapılar kullanılmaya başlanılmıştır çünkü nano boyutlara inildiğinde hem yapıların gösterdiği performas düzeyi hem de sinerjisi ve uyumu artmaktadır. Nano hibrit yapılar bu anlamda bir çözüm olmuşlardır, aynı şekilde viskon kumaşlardaki bu sorunlar için de nano hibrit polimerler çözüm olabilir. Boyutu ve fonksiyonel kor yapısı sayesinde, iyi bir stabilite ve dispersiyon gösterebilir ve liflerin arasına girerek de kumaşın her bölgesine etki edebilir. Poli(AAm-ko-AMPS) nanokompozit hidrojellerin mekanik olarak esnek ve aynı zamanda sağlam yapılar oluşturmaları ve seluloza çapraz bağlanması ile hem hidrojen bağlarının yerine daha sağlam kovalent bağların kurulmasını hem de selulozik polimerlerin stres altında viskoelastik davranış göstermesini sağlar. Bu iki özellik birleşince mukavemet kaybı olmadan elyaf elyaf sürtünmesi azalacağından boncuklanmanın da önüne geçilmiş olur.

1. INTRODUCTION

Regenerated fibers such as viscose, modal and bamboo production is becoming more important, because the production of natural fibers is increasing but demand is below the population [1]. Using the cotton fabric creates problems in pretreatment and dyeing stages of the process [2]. Moreover cotton has some disadvantages to viscose. Energy consumption during the production and the use of cotton is almost doubled the viscose. Also viscose dehumidification capability is higher than cotton. Viscose collects about 14% moisture and due to the swelling property can absorb water 80% up to 120%. Because of this high moisture capacity and swelling property, viscose is more convenient than cotton for dyeing and printing and the colors look too bright [3]. Despite all these shortcomings, viscose limits the use of positive properties. But if these restrictions have been eliminated, savings will increase and use of energy during the processing of cotton will be provided. Therefore the target should be; improving the resistance to friction while maintaining the elasticity and high water absorption capability of viscose.

1.1. Purpose of Thesis

Antipilling chemicals which is sold in the market are not enough to satisfy this demand. Some of them is improving the fabric's resistance to pilling, nevertheless they cause to lose their tensile strength. Besides some of them does not damage to fabrics's properties but they can not give expected effect for pilling resistance. Purpose of this thesis to design a product which possess unique properties to improve the antipilling characteristic of the viscose fabric while maintaining the existing properties. Padding is useful and preferred system for textile applications because of simplicity of application method so a product which can be applied by padding system was aimed to synthesize.

1.2. Literature Review

1.2.1 Disadvantages of cotton usage

1.2.1.1 Energy consumption

Energy consumption is an intensive process for textile manufacturing. Between the years of 1971 and 2004, energy usage in the textile sector has doubled in the world. The most commonly used energy type is electric energy. In textile; especially for heating and cooling control systems, lighting, and office equipments working with electric energy. Textile is an important sector for Turkey, it is including 35,000 companies and over than 2 million employee. For this sector total production volume was $30billion USD. Over last two decades, Turkish textile has remained until elimination of quotas in textile sector for China in 2005. Competition is getting difficult because of increasing costs of energy, textile raw materials, labor, finance and other related expenses [4]. World cotton fibre production was 19 million tonnes from 33.4 million hectares in the year 2005. Turkey has the sixth largest cotton cultivation area [5].

1.2.1.2 Water consumption

The production of 1 kg of cotton fibre can require more than 20,000 L of water. Although cotton fibre products are widely recognized by the consumers as being soft, natural and comfortable but there is a need to find a suitable alternative to the conventional cotton fibre. The consumers not only desire an increase in comfort but there is also growing awareness among consumers for eco-friendly products. Research to enhance the eco-friendliness of textile products has generally been concentrated on wet processing of textile substrates to produce the dyed and finished fabrics with minimal use of chemicals, water and energy [6].

1.2.2 An Alternative to cotton : viscose

For instance bamboo viscose, it is a green raw material, which is renewable and sustainable with little harmfull impact to the enviroment. Bamboo plant requires no irrigation and can be grown in a natural environment without the use of pesticides and it grows really fast. Organic Crop Improvement Association has certified bamboo viscose fibre as organic fibre which can degrade under the action of microorganisms and sunshine. Bamboo fibre has high moisture absorption capacity and it is breathable, cool and really soft. In addition, it possesses inherent

antibacterial and anti-UV properties. Bamboo viscose's unique structure aids to quick evaporation of moisture. Bamboo fibre requires less amount of dye for the same shade depth and appears better, softer similar in texture to silk and dries quickly, therefore, the use of bamboo fibre as an alternative of cotton will be better for environment as well as using less dye and consequently less waste would be produced [6]. Bamboo fibre yarns provide the good absorbency, antimicrobial and soft feel properties which desired in home textiles and garments. 100% Bamboo fabric's air permeability is around 200%. The water vapour transmission due to diffusion may also be higher for the bamboo fabrics as the moisture regain of bamboo fibre is higher than that of cotton [7]. Regenerated fibers have a semi-crystalline structure which they have crystalline regions together with more or less amorphous regions. The fibers are of low crystallinity but highly accessible to different media due to their mainly amorphous molecular arrangement and an extensive inner surface. Because of the morphological characteristics, they show stronger swelling effect when compared to the other regenerated cellulose fibers. Cotton structure and viscose structure were shown in Figure 1.1 and Figure 1.2.

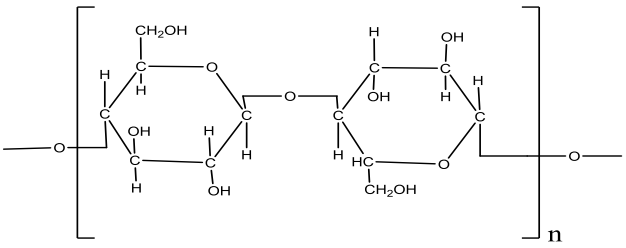

Figure 1.1: Cotton structure.

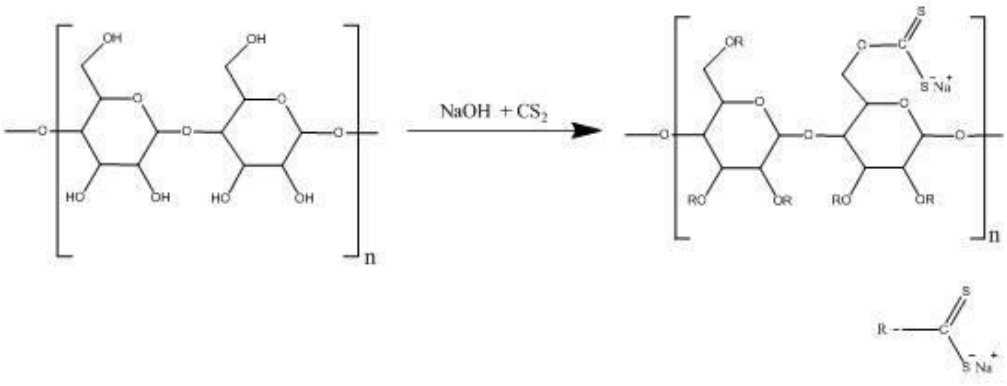

Figure 1.2 : Cellulose is treated with alkali and carbon disulfide to yield viscose.

Beside all these good properties in use, viscose fibers have pilling problem [8]. A pill, known as a bobble, is a small ball of fibers that forms on a piece of cloth, as seen in Figure 1.3. 'Pill' is also a verb for the formation of such balls. Pilling is a surface defect of textiles caused by wear, and is considered unsightly. It happens when washing and wearing of fabrics causes loose fibres to begin to push out from the surface of the cloth, and, over time, abrasion causes the fibres to develop into small spherical bundles, anchored to the surface of the fabric by protruding fibers that haven't broken. The textile industry divides pilling into four stages: fuzz formation, entanglement, growth, and wear-off [9].

Figure 1.3 : Two different pilling examples for viscose fabric.

Average pill size and pill frequency effect the quality of the finished fabrics. The finishing reduces the pill formation by stabilizing the protruded fibers inside the yarn and remove the surface nap because of that it plays a critical role in improving the fabric quality. This can be achieved via heat setting, singeing, brushing, cropping or with chemical treatment [10].

1.2.3 Pilling solving types by chemical treatments

1.2.3.1 Enzyme based product

To decrease the pilling propensity of the fabric, celulases are used. It removes the fuzz or pills on the fiber or fabric surface. Cellulase enzymes are multicomponent enzymes. Commercial cellulases currently used consist of multiple enzyme systems which hydrolyse cellulose in a synergistic way but pilling problem is still in progress in viscose based textile surfaces. Bahtiyari, M.I., et al. obtained, six different commercial cellulases and used them to decrease in pilling tendencies. In the final stage, all experimental processes, pilling values did not increase more than 0.75 point

while bursting strength loss (BSL) has increased (the untreated fabrics's pilling value was 2) and bursting strength losses was determined [11]. Besides enzymes have several disadvantages, If the enzyme which applied on textile material is not suitable, it can damage chemical structure of viscose. When the samples from industry are tested in laboratories, they sometimes encounter mechanical and/or chemical damage problem. Although deactivation step is necessary, sometimes it can be eliminated in some mills. Korlu, A.E., investigated the influence of cellulase waste on the cellulosic fabrics. For this trials viscose and cotton fabrics were used. After biopolishing, viscose and cotton fabrics stayed with cellulase enzyme for 30-60-120-240 min, a day and a week. The effects of enzyme were defined by pilling degree, weight loss, yarn strength, silver nitrate and fehling tests. The results says that; cellulose enzymes were more effective for cotton fabrics. After biopolishing, cotton fabric's pilling degree is 5, but viscose fabric's pilling degree is 2.5. Besides chemical damage occurred on viscose fabrics. Strength losses are 13% for cotton fabric and 23% for viscose fabric. Ezymes are not exact solution as well as it causes to lose existing properties of viscose [12].

1.2.3.2 Silicic acid based products

Silicic acid ester acts as antipilling agent which can comprise with cellulase enzyme. Certain fabric conditioners directed to reduction in fluff formation and pilling in textiles, particularly during a washing or drying process. It also discloses a conditioning substrate containing a conditioner and a conditioning process using the conditioning substrate in a laundry drying process [13]. The major disadvantage of this process is reduction of fiber strength [14].

1.2.3.3 Polyurethane based products

In textile industry polyurethane copolymer are used as an antipilling chemical. Tabasum, S., and Zuber, M., have used polyurethane acrylate copolymer in order to get better rating of pilling in their research. At the end, they got clear separation lines among the five pilling propensity groups and a progressive trend between the no pilling and the most severe pilling samples [15]. Fibers are sticking together and the volume increase of fibers could not be possible by using and vinyl-based polymers as antipilling agent.

Karaboyacı, K., has studied with nano polyurethane to give the fabrics resistance effect to abrasion with padding method. Nano polyurethane has increased the antipilling rating between 0-2 points [16].

1.2.4 Polymers

1.2.4.1 Definition of polymer

A polymer is a large molecule composed of a combination of many subunits which repeat themselves along the long molecule. The small starting molecules are called monomers, and the unit molecules are bonded together in long repeating chains. The length of the polymer chain is specified by the number of repeating units. This chains number is called the degree of polymerization. Most of the monomers are composed of carbon, hydrogen, oxygen, and nitrogen. Few other elements such as fluorine, chlorine, sulfur, etc. may also exist [17].

1.2.4.2 Classification of polymer

Polymers can be classified the in many different ways depending on their various properties. Some of them are given below, polymer classification according to:

a. Natural polymers: comes from natural origin. For example; proteins, starch, cellulose, natural rubber, etc.

b. Synthetic polymers: These are man-made polymers which are synthesized in the laboratories [18].

1.2.4.3 Radicalic polymerisation

Radical polymerisation is a chain reaction which involves three fundamental steps:

-Initiation

-Propagation

-Termination

Also chain transfer may be involved. The reactions of monomers may occur by the absorption of heat or light, but often an initiator is added. The initiator I{ I is a weak organic compound which can be decomposed thermally or by the irradiation to produce free radicals, which are molecules containing atoms with unpaired electrons. After that an addition of the initiator radical to a monomer molecule M follows. This may be represented as indicated in (1.1) (1.2) (1.3). :

$$\mathbf{I - I \rightarrow 2I\cdot} \qquad \mathbf{(1.1)}$$

$$\mathbf{I\cdot + M \rightarrow I - R_1\cdot} \qquad \mathbf{(1.2)}$$

$$\mathbf{I - R_1\cdot + M \rightarrow I - R_{i+1}\cdot} \qquad \mathbf{(1.3)}$$

During propagation, the initiated monomer adds other monomers in rapid succession. This involves the addition of free radicals to the double bond of a monomer, with regeneration of another radical. The active center is thus continuously relocated at the end of the growing polymer chain. Propagation continues until the growing chain radical is deactivated by chain termination or transfer. Termination is a reaction of a polymer chain radical with the initiator radical (1.4) or with an another macro radical (1.5).

$$\mathbf{I - R_i\cdot + I\cdot \rightarrow I - R_i - I} \qquad \mathbf{(1.4)}$$

$$\mathbf{I - R_i\cdot + I - R_j\cdot \rightarrow I - R_i - R_j - I} \qquad \mathbf{(1.5)}$$

In chain transfer reactions a molecule of the chain transfer agent (CTA) is decomposed into a terminating part T and a new radical A· which may initiate a new chain (1.6).

$$\mathbf{I - R_i\cdot + T - A \rightarrow I - R_i - T + A\cdot} \qquad \mathbf{(1.6)}$$

The final result depends on several parameters. Increase of these parameters affects the molecular weight(M_w) of polymer as follows: An increase of temperature causes faster reactions, lower M_w. Higher pressure increases the propagation and inhibits the termination of polymer hence gives higher M_w. Adding more initiator, occurs more radicals, creates more chains, so forms low M_w polymer. More chain transfer agent occurs narrower molecular weight distribution, hereby lower M_w polymer forms. There are many other reactions that may appear, which can be neglected [19].

1.2.4.4 Copolymer

When two or more different types of monomer are bonded in the same polymer chain, it takes the name of copolymer. If exactly three monomers are linked together, it calls terpolymer. Monomers are only occasionally symmetric; the molecular arrangement is the same no matter which end of the monomer molecule. Three type of arrangement can occur in a copolymer; they are head-to-tail, head-to-head, or tail-to-tail. Classification of copolymers based on how units are arranged along the chain.

These classifications include the following:

• Alternating copolymer

• Random copolymer

• Block copolymer

• Graft copolymer.

[20].

1.2.4.5 Hybrid polymer

Day by day the working range of inorganic-organic nanocomposites is growing, because hybrid materials may have combined properties of both the added inorganic materials and the base polymers. Nanocapsules are intriguing materials in hybrides , on account of the ability in making a diverse range of new materials for many applications. A wide variety of colloidal inorganic materials has been used in polymer nanocomposites, including silica, titanium dioxide, copper oxide, magnetic oxide, aluminum hydroxide, silver, clay and carbon black. Silica is the most studied material between them and hybride polymer of silica have excellent physical reinforcements; high thermal resistance, high flexibility, high gas permeability and low surface energy, owing to the including of silica. Nanosilica can improve the strength of polymer due to uniform dispersion, abrasion-resistance, the aging-resistance and the climate-resistance of the polymer, as well. Thus, this material have been used many applications for instance in thermal insulators, bioactive supports, paints, plastics, rubbers, coatings, drug delivery systems and composite materials [21].

1.2.5 Synthesis of nanosilica

Nanoscale materials when compared with bulk materials, show more improved properties. In last years, the demand of nanoscale materials has been increasing, generally in industrial applications [22]. Silica nanoparticles take an important place in researches, due to easy preparation and wide uses in various industrial applications, such as catalysis, pigments, pharmacy, electronic and thin film substrates, electronic and thermal insulators, and humidity sensors. The particle size and size distribution are very important parameters for some of the quality parameters of product [23].Three main types of process methods are suggested for

synthesis of silica particles. Elham Noori et al. prepared silica particles with microemulsions method in the size range of tens to hundreds nanometers. The process works well especially in the range of 30–60 nm, yielding silica spheres with better average monodispersity in comparison with particles synthesized by the Stöber process. Moreover, large amounts of surfactants should be used maybe more by weight than silica while generating inverse micelles. Therefore, Further cleaning requires to remove the remained surfactant from particles. Additionally, it needs a challenge to maintain uniform micelle parts besides soft and sensitive phases. Balthis and Mendenhall reported the production of monodisperse small silica particles through hydrolysis finely divided elemental silicon with water using ammonia as catalyst in the temperature range of 20–90˚C [24]. Meanwhile, important disadvantage of this method is that before reaction, activating of elemental silicon is an essential. Washing with aqueous hydrofluoric acid, pure water, alcohol and ether consecutively to remove silicon dioxide film from the particle surface and to expose a clean silicon surface. To prepare monodisperse silica spheres, Stöber process is more convenient method because, it is simple and most effective system. Normal reactants and controllable reaction condition is necessary for this system so, it is easy to be carried out [25].

1.2.5.1 Synthesis of nanosilica particles by sol-gel process

It is widely used to produce pure silica particles due to possibility of controlling the particle size, size distribution and morphology through systematic monitoring of reaction parameters. The sol-gel process includes hydrolysis and condensation steps of metal alkoxides ($Si(OR)_4$) such as tetraethylorthosilicate (TEOS,$Si(OC_2H_5)_4$) or inorganic salts for instance; sodium silicate (Na_2SiO_3) in the presence of mineral acid (e.g., HCl) or base (e.g., NH_3) as catalyst. A general flow chart for solgel process for the production of silica is shown in Figure 1.4.

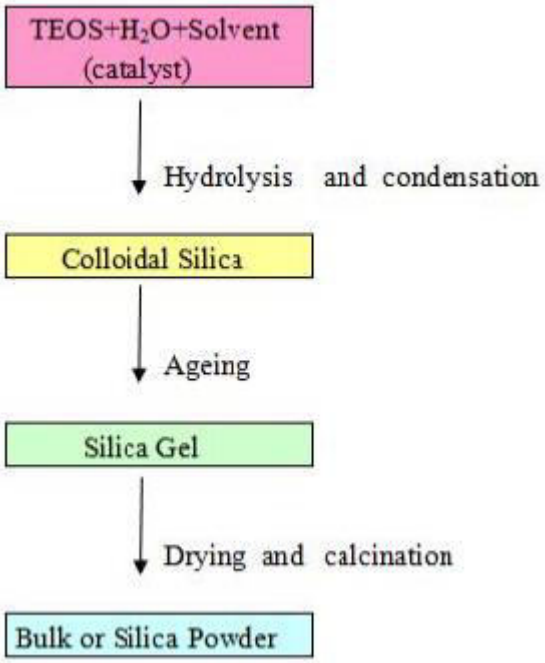

Figure 1.4: Flow chart of a typical sol-gel process

The general reactions of TEOS to form silica particles with sol-gel process can be written as indicated in Figure 1.5.

$$Si(OC_2H_5)_4 + H_2O \xrightarrow{\text{hydrolysis}} Si(OC_2H_5)_3OH + C_2H_5OH$$

$$\equiv Si - O - H + H - O - Si \equiv \xrightarrow{\text{water condensation}} \equiv Si - O - Si \equiv + H_2O$$

$$\equiv Si - OC_2H_5 + H - O - Si \equiv \xrightarrow{\text{alcohol condensation}} \equiv Si - O - Si \equiv + C_2H_5OH.$$

Figure 1.5 : The general reactions of TEOS in the sol-gel process.

After the hydrolysation of TEOS molecules, gives silanol groups. Siloxane bridges (Si–O–Si) that form entire silica structure occurs through the condensation polymerization between the silanol groups or between silanol groups and ethoxy groups. Silica particles forms in two steps: nucleation and growth. And there are two models for growth mechanism of silica, they are monomer addition and controlled aggregation. In monomer addition model; the particle growth occurs after an initial burst of nucleation, owing to the addition of hydrolyzed monomers. Besides, in the aggregation model, nucleation occurs along the reaction and the resulting nuclei which is the primary particles will aggregate together to form dimmer, trimmer, and

larger particles which are the secondary particles. Common points of these models are leading to the formation of either spherical or gel network which depends on the reaction conditions as shown in Figure 1.6 [26].

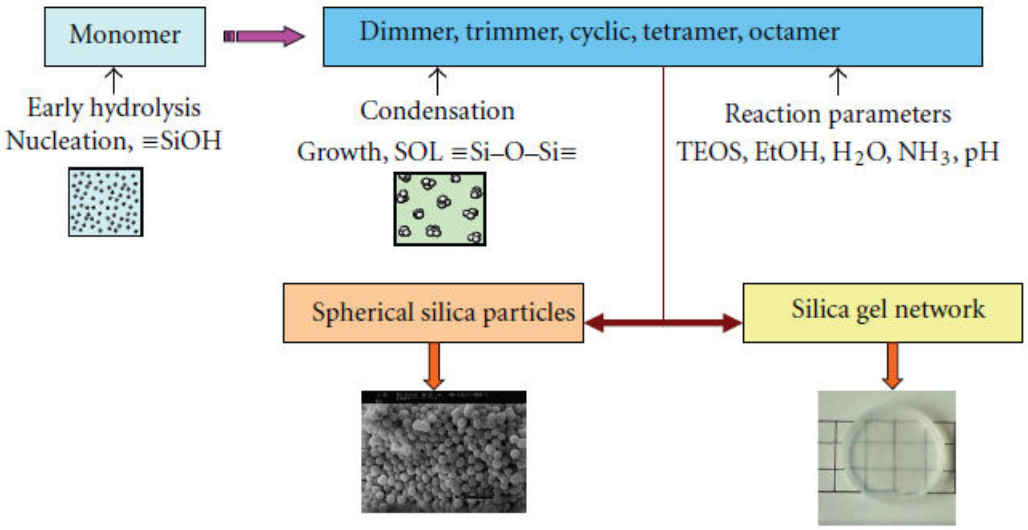

Figure 1.6: Schematic silica formation by sol-gel process.

1.2.5.2 Optimization of reaction parameters

Many researches were performed to find out optimum conditions for sol gel synthesis of stable silica particles. Performed studies and determined parameters are as follows;

Effect of TEOS

Reaction conditions are very important parameters to form homogeneous and monodispersed silica particles. TEOS determines the concentration of nuclei/primary particles which is the source of the monomer in the system. Nucleation occurs in first supersaturated solution and then in induction period it causes the formation of the primary particles. More stable secondary particles can form owing to the aggregation of primary particles. Rahman, I.A., et al. showed, the process continues until all the primary particles consume or until a stable condition was achieved. They analyzed the effect of TEOS concentrations on particle size, yield and size distribution (SD) of the silica particles. Additionally, in Figure 1.7, they found out that, at fixed $[NH_3]$ = 0.08 mole l^{-1} and $[H_2O]$ = 0.04 mole l^{-1}, particle size increase with the increase in [TEOS] until 0.80 mole l^{-1} whereby the size starts to stabilize at~90 nm.

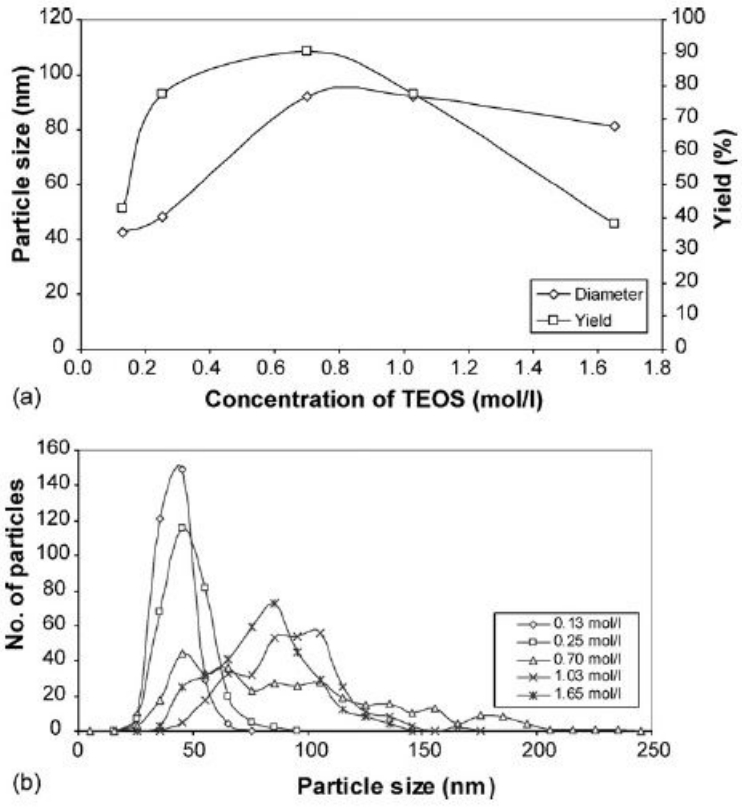

Figure 1.7: Effect of TEOS concentrations on particle size (a) yield and size distribution of the silica particles (b) particle size changes with change of mole of TEOS at $[NH_3]$ = 0.08 mole l^{-1} and $[H_2O]$ = 0.04 mole l^{-1}.

They figure out that; the increasing of particle size is achieved to the increasing of primary particles' concentration at the induction period. Namely [primary particles]∝[TEOS]. Other than this, ammonia becomes the limiting reactant at [TEOS] > 0.80 mole l^{-1} and results in an inefficient hydrolysis and condensation reactions. Consequently, the product yield drops more than 50% due to incomplete reactions, and the particle size remains almost constant. Increasing of TEOS concentration, increases the agregation. Transmission electron microscopy (TEM) images which are given in Figure 1.8 prove these results [22].

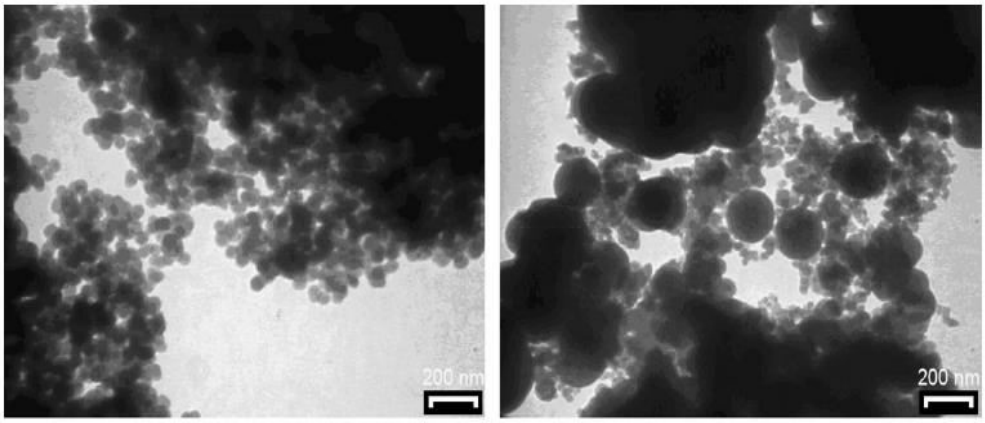

Figure 1.8: TEM images of silica particles synthesized at different concentration of TEOS : (a) 0.13 mole $l^{-1}$and (b) 1.65 mole l^{-1} at fixed conditions.

Effect of the water:TEOS molar ratio

Hydrolysis and condensation kinetics are affected by the quantity of water in the sol-gel solution. Increasing of water content raises hydrolysis and condensation rate. Decreasing of water content because of high dilution with alcohols can lead to a high content of oligomers and reduces the yield of SiO_2. Furthermore oligomers can affect the composition and the homogeneity of the final material. With decreasing of water:TEOS molar ratio, time of gelation decreases and pore size decreases with increasing ethanol: TEOS molar ratio and pH [27].

Effect of pH and catalysts

Hydrolysis and condensation reactions generally controlled by the solution's pH. The hydrolysis kinetic is favoured under acid-catalyzed conditions, differently in alkaline-catalyzed reactions, condensation kinetic is favoured , resulting in a highly condensed species which may agglomerate into fine particles. Si is less electropositive so, change of surface charge can be easily carried out in the case of silicon alkoxides by changing pH using acid or alkaline catalysts to enhance the hydrolysis and condensation reactions. pH value should be preserved between 0 to 2 to stabilize the sols. The resulting surface charges reduce the particle-to-particle interaction and no aggregation or agglomeration takes place. Particle-to-particle distance can be reduced through the solvent evaporation or pH changes. But, growing of particles too much occurs precipitation. At that rate, sol-gel systems based on oxides, the particle-to-particle interactions are strong therefore, the agglomeration is irreversible particularly after drying [27].

Effect of water and ammonia concentration

Hydrolysis is generally a very slow reaction, even though catalysts are used. Ammonium hydroxide acts as a catalyst for hydrolysis and condensation of TEOS in ethanol. Larger particles forms with increasing of the ammonia and water concentrations. Hereby, smaller particles form with increasing of water concentration. Differently , in this research, a reverse effect is observed. Size of silica nanoparticles is decreased while increasing of the ammonia concentration. Studies which are conducted by the researchers reveal different results. Rao Kota Sreenivasan has incorporated this issue in a research. Increasing in water concentration has been yielded smaller particles. On the other hand, in an another trial, larger particles has been obtained at higher water concentrations [23]. Matsoukas, T., et al. showed that; size of silica particles were getting smaller with increasing ammonia concentration as seen in Figure 1.9(A) (Roa, S.K., as referred in 2005). It is reacted in 8 M ethanol, 3 M water, 0.045 M TEOS.

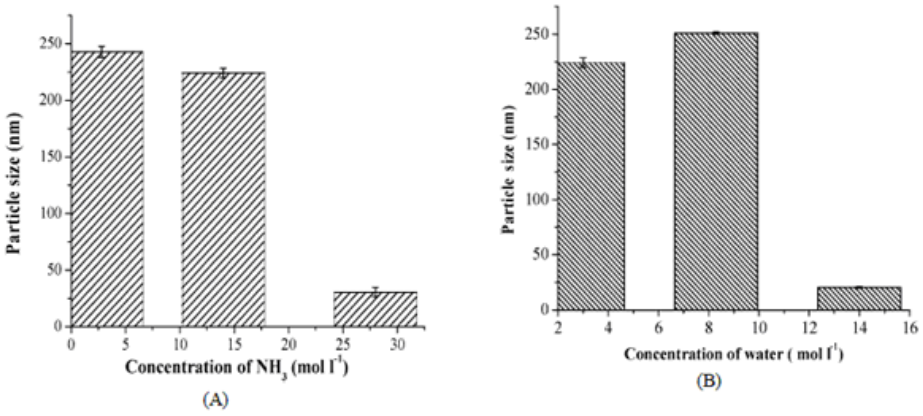

Figure 1.9: (A) Effect of ammonia on particle size; (B) effect of water on particle size.

In the presence of high water concentration (See Figure 1.9 (B), a high nucleation rate occurrs and a lot of subparticles are produced bearing the size 20 nm at 4 M ethanol, 0.045 M TEOS, and 14 M of ammonia concentration [23]. Differently, Park et al. found, mean particle diameter increases with increasing water concentration and ammonia concentration. [H_2O]/[TEOS] ratio and ammonia concentration must be decreased to decrease the particle size. If an acid or a base is used as a catalyst, hydrolysis acts as a very slow reaction. For hydrolysis and condensation of TEOS, ammonia is used as a catalyst. The increasing of water concentration occurs smaller

particles. On the contrary, higher water concentration occurs the large particle size . In high water concentration, a high nucleation rate occurrs so a lot of small sub-particles form. In higher water concentration, hydrogen bond of SiO_2 sub-particles stronger than lower water concentration because of excess water. Thus sub-particles agglomerate each other in the higher water concentration. Small sub-particles have grown into a large particle by agglomeration. Figure 1.10 illustrates the effect of the water concentration on the particle size and its distribution [28].

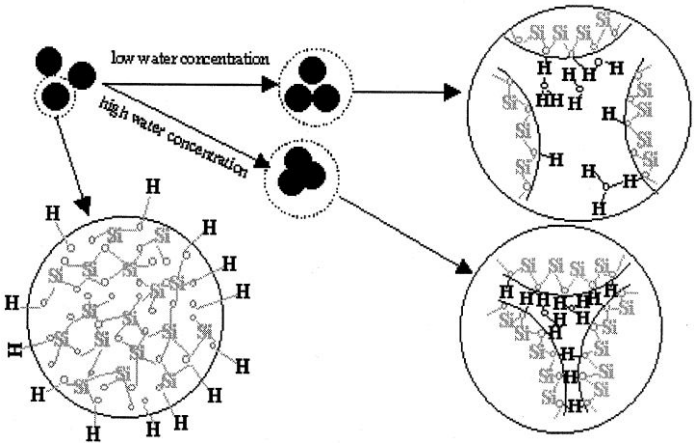

Figure 1.10: Effect of water concentration on particle size and size distribution.

Effect of the ammonia feed rate

The ammonia feed rate into reaction mixture was found to have significant effect on particle size, size distribution and yield. A. Rahman et al. tried to control particle growth through controlling the feed rate of NH_3. They obtained smaller particle size and narrow size distribution by slower feeding of NH_3. Faster feed rate results in faster particle growth besides forming larger particles with wider size distribution. Hereby aggregation occurs quickly in the primary particles. Homogenous,nanoscale, narrow distributed particles were formed at feed rate of 0.05 and 0.03 ml min^{-1}.This feed rate caused slight reduction in the yield, ~25% compared to 0.40 ml min^{-1}, possibly due to incomplete reaction of TEOS. Size of silica prepared at the feed rate of 0.03 ml min^{-1} was 14.7±2.5 nm [22].

Effect of reaction temperature

Particle size generally decreased with an increasing of the reaction temperature (See Table 1.1). With increasing of the temperature nucleation rate is increased and as a result the particle size is reduced because of high nucleation rate. From the temperature of 45 ˚C to 55 ˚C the particle size and standard deviation is decreased.

Besides, when they look 55˚C to 65 ˚C, there is no significant change in the mean particle size, however the standard deviation is reducing [22].

Table 1.1 : Effect of temperature on particle size of silica.

Temperature(˚C)	Particle size(nm)	Standard deviation(nm)
45	92.3	43.6
55	35.2	11.3
65	32.6	7.1

The effect of temperature on size of silica nanoparticles was explored by Zainal et al too. The reaction temperature was varied between 30 °C and 70 °C under the fixed experimental conditions. In these conditions, mean particle size of silica was obtained in the range 29 – 113 nm. With an increasing of the reaction temperature, particle size generally increased. At higher temperature, monodisperse and uniform-sized silica nanoparticles were obtained. From 30 °C to 70 °C nanoparticles also becomes bigger. At 60 °C, produced silica nanoparticles were smooth and spherical. At 70 °C maximum mean particle size of silica nanoparticles was 113.22 nm. High temperature promotes the hydrolysis and rate of condensation reaction. And size of silica nanoparticles becomes larger. Ammonia evaporation causes an increase of particle size at high temperatures. With increasing of temperatures, agglomeration risk is increasing. Zainal et al. chosed the 60 °C chose as the best conditions for the fabrication of the silica nanoparticles [29]. Roa et al. found out overall optimal synthesis conditions to synthesize nanosized particles which have a narrow size distribution (See Table 1.2). In the optimal conditions; minimum particle size was found 30 nm and size distribution was found 5 nm [23].

Table 1.2: Optimal synthesis conditions.

Parameter	Range
R[H_2O/TEOS]	30-55
NH_3 (mole l^{-1})	0.2-0.35
Feed rate (cm^3 min^{-1})	13-17
Temperature(˚C)	55-65

1.2.6 Encapsulation of nanosilica

Polymer coating changes the interfacial properties of modified particles because of this reason surface modification with a polymer shell is more attractive method for inorganic fillers [30]. The thermal and the mechanical properties of the hybrid polymers can be improved by the compatibility of the nanoparticles with the polymer

matrix. Size and shape of the inorganic core determine the physical properties of inorganic core of nanoparticle. Inorganic material surfaces can be functionalized by coating with the polymer chains. Functionalization can be made through covalent bonding (chemically) or physisorption (physically). The incorporation of silica particles into nanocomposite colloids offers a lot of promising perspectives. For preparing the nanocomposite materials, various methods have been developed. For instance; heterophase polymerization which is usually resulting in polymer encapsulation of silica nanoparticles, sol-gel process which is resulting in coating of polymer colloids with silica and and self-assembly. Capsulation of silica nanoparticles with polymer can exhibit unusual, even unique, properties. Encapsulation of silica by polyacrylamide has been achieved in dispersion polymerization and the surface tension of water increased in the presence of silica. Surface tension of water was reduced the from 72 to 50 mN/m by silica/polyacrylamide. Formation silica network in water increased the surface tension and the amide groups of polyacrylamide can be acted as bridges between silica solid particles that facilitating their adsorption at interface. Further, small surface micelles were formed through hydrophobic association between the polymer backbone. Acrylamide molecules first adsorbed as individual molecules due to interaction with OH groups of silica, and then associated into hemimicelles [31]. A graft copolymer based on a component. This component consisting of silica which has been reacted with an unsaturated silane, and sulphonic acid containing polymer component is recomended. Preferably nanosilica and unsaturated alkoxysilane which is ethylenically unsaturated is used. And for copolymerizaton, ethylenically unsaturate AMPS monomer is choosed [32].

1.3. Hyphotesis

Despite all these shortcomings, there is a need in the market to cover a product because available products could not provide the market's need. A new product was intended to develop which could be bonded to viscose by chemically. Target product will not harm the existing properties while providing new features. And it could be applied to fabric easily. Firstly, the structural properties of viscose and cotton were examined to modify viscose with an easy textile finishing to make it similar with cotton. Resistance of viscose to abrasion was aimed to improve while maintaining the high water-holding capacity and flexibility which are the most important

advantages of viscose to cotton. Modified viscose will show anti-friction because of the polymer which is chemically bonded to viscose. It is aimed to give resistance to prevent pilling. From all these researches and prospects. Silane based organic–inorganic nanohybrid polymer was decided to develop. This polymeric nanocomposite possess unique properties, which could be used to improve the antipilling properties of the viscose fabric. Because of the nano hybrid polymer structure it would show better performace than other polymers. These nano structures not only locate the fabric surface but also it would penetrate the inside of fiber thus it would reduce the friction.

2. EXPERIMENTAL

2.1. Materials and Chemicals

Tetraethyl orthosilicate(TEOS), 99%, Zhejaing Feidian Chemical Co., Ltd..

2-Acrylamido-2-methylpropane sulphonic acid (AMPS), industrial grade, Shandong Taihe Water Treatment Technologies Co., Ltd.

Acrylamide, Industrial grade, Zibo Xinye Chemical Co., Ltd..

Ethanol, 99.9%, Merck.

2,2′-Azobis(2-methylpropionitrile) (AIBN), 98%, Sigma Aldrich.

Ammonium hydroxide solution (NH_4OH), 30-33% NH_3 in H_2O, Sigma Aldrich.

Hydrochloric acid(HCl), ACS reagent, 37%, Sigma Aldrich.

Hydrogen fluoride (HF), >99.9%, Sigma Aldrich.

Idrocap 982, Water borne aliphatic polyurethane, Gicap Development Co., Ltd.

Antipill jet conz, Silicic acid salt, Eksoy Chemicals And Dyes.

Biopolish 300, Enzyme, Eksoy Chemicals And Dyes.

Table 2.1 : Fabric Properties which supplied from Akteks Textile

No	Fiber Type		g/m²
1	100% Viscose	White Knitted Fabric	173
2	100% Viscose	White Woven Fabric	116
3	100% Viscose	Khaki Knitted Fabric	175
4	100% Viscose	Khaki Woven Fabric	120
5	100% Viscose	Green Knitted Fabric	160

2.2. Equipments

2.2.1 Gel permeation chromatography(GPC)

Gel permeation chromatography (GPC) analyses were performed with Malvern GPC/SEC system with water as the eluent at a flow rate of 0.7 mL/min. Molecular weights were calculated on the basis of a calibration curve recorded with dextran and polyethylene standards. Gel Permeation Chromatography is used for measurement of

molecular weight, size and structure of polymers. It is an analytical technique which separates dissolved macromolecules by size based on their elution from columns filled with a porous gel. When GPC is coupled with light scattering, viscometer and concentration detectors (known as triple detection), it can measure absolute molecular weight, molecular size and intrinsic viscosity, and generate information on macromolecular structure, conformation, aggregation and branching [33].

2.2.2 Particle size analyser

Malvern Mastersizer 3000E was used for particle size analysis. This instrument uses the technique of laser diffraction to measure the size of particles. It does this by measuring the intensity of light scattered as a laser beam passes through a dispersed particulate sample. This data is then analysed to calculate the size of the particles that created the scattering pattern [34].

2.2.3 Scanning electron microscope- Energy dispersive detector

The Phenom ProX desktop scanning electron microscope (SEM) was used in this thesis. EDS is a technique that analyzes X-rays generated by the bombardment of the sample by an electron beam. With this system sample structures can be physically examined and their elemental composition determined [35].

2.2.4 Thermogravimetric and differential thermal analysis(TG/DTA)

Hitachi STA 7200 system was used for glass transition temperature analysis with a heating rate of 10 ºC min under nitrogen flow. Simultaneous Thermal Analyzer performs complex thermo gravimetry/differential thermal analysis (TG/DTA) of organic materials such as polymers and inorganic materials such as metals and ceramics simultaneously [36].

2.2.5 Fourier transform infrared spectroscopy-Attenuated total reflectance (FTIR-ATR)

IR spectra were recorded on a Perkin Elmer Spectrum One FTIR-ATR Spectrometer. An infrared spectrum represents a fingerprint of a sample with absorption peaks which correspond to the frequencies of vibrations between the bonds of the atoms making up the material [37].

2.2.6 Universal strength tester

James Heal, Titan5 model was used for strength tests of woven fabrics. 5 different observation was done and mean values of observations were calculated for each sample. BS EN ISO 2001 standard was applied.

2.2.7 Burst strength tester

James Heal ,Truburst3 Model 140 was used for strength tests of knitted fabrics. ISO 13938-2 standard was applied.

2.2.8 Abrasion and pilling tester

Pro-Ser Prowhite Martindale was used to analyse the abraisions, pilling and resistance of fabrics toward different surfaces [38].

2.3. Synthesis Methods and Application Processes

2.3.1 Synthesis of nanosilica particles with different mol ratios

Firstly, distilled water and alcohol which were required in the Table 2.2, were weighed and transfered the 250 mL single neck flask and adjusted the ph:8 with NH_4OH. The anions was consisting in a basic medium to prevent the agglomeration of particles, this is the reason of starting with basic medium in the first trials. It was started mixing with a magnetic stirrer at 25˚C. Then, 13.89g TEOS were added by dropwise within 3 hours. It continued to be stirred for 24 hours. Then 55.55g, remaining weight of TEOS, added by dropwise within 5 hours The reactions were terminated when the enough gelling was occured. After that, gels were dried at 80˚C for 24 hours and then calcinated at 600˚C for 2 hours.

2.3.2 Synthesis of nanosilica particles with different catalysts

48.02g distilled water and 15.35g ethanol were weighed and transfered the 250 mL single neck flask and adjusted stated ph with each catalysts as indicated in the Table 2.3. Solution was started mixing with a magnetic stirrer at 25˚C. Then, 13.89g TEOS were added by dropwise within 3 hours. It continued to be stirred for 24 hours. Then 55.55g, remaining weight of TEOS added by dropwise within 5 hours. The reactions were terminated when the enough gelling was occured. After that, gels were dried at 80˚C for 24 hours and then calcinated at 600˚C for 2 hours.

Table 2.2: The weights of H_2O, Si, Ethanol, TEOS, which were used in reactions.

H_2O(g)	Si(g)	Ethanol(g)	TEOS(g)
6	9.36	15.35	69.44
6	9.36	30.7	69.44
6	9.36	46.06	69.44
12	9.36	15.35	69.44
12	9.36	30.7	69.44
12	9.36	46.06	69.44
24.01	9.36	15.35	69.44
24.01	9.36	30.7	69.44
24.01	9.36	46.06	69.44
48.02	9.36	15.35	69.44
48.02	9.36	30.7	69.44
48.02	9.36	46.06	69.44
96.05	9.36	15.35	69.44
96.05	9.36	30.7	69.44
96.05	9.36	46.06	69.44
192.1	9.36	15.35	69.44
192.1	9.36	30.7	69.44
192.1	9.36	46.06	69.44

Table 2.3: Used catalysts and studied pH ranges.

Catalyst	pH
HCl	<2
NH_4OH	7<pH<8.5
NH_4OH	>9
HF	2<pH<7

2.3.3 Synthesis of encapsulated nanosilica particles

AMPS, AAm, AIBN, distilled water and nanosilica (30nm, which synthesized in the first step) as indicated amounts in Table 2.4 were weighed into a 250ml two neck flasks. The polymerization was equipped with a reflux condenser and a mechanical stirrer. While they were stirring, temperature was started to rise, reaction was continued for 4 hours at 60 C. Reactions were terminated by the adding of remained distilled water to make the solution 100%.

Table 2.4: Used w/w % compositions of AMPS, AAm, nanosilica, AIBN and water.

Sample	AMPS(%)	AAm(%)	Nanosilica(%)	AIBN(%)	Water(%)
1	0.0	2.5	1.5	0.02	30
2	0.0	5.0	1.5	0.02	30
3	2.5	0.0	1.5	0.02	30
4	2.5	2.5	1.5	0.02	30
5	2.5	5.0	1.5	0.02	30
6	5.0	0.0	1.5	0.02	30
7	5.0	2.5	1.5	0.02	30
8	5.0	5.0	1.5	0.02	30

2.3.4 Application of hybrid polymer samples to viscose fabrics

20, 40, 60 and 80g, 1 to 8 samples were dissolved in 1 liter distilled water. And solutions were applied on viscose fibers by padding system with catalyst ($K_2S_2O_7$) and all them dried at 100˚C for 5min.

2.3.5 Application of Antipill hp and existing products used in the market to viscose fabrics

20 g and 40g Antipill jet Conz , modified polyurethane and Antipill hp solutions were dissolved in 1 liter distilled water. And they were applied on viscose fabrics by padding system with catalyst ($K_2S_2O_7$) and all them dried at 100˚C for 5min. The reaction accurs between viscose and the samples. Biopolish 300 applied with overflow system, 50˚C for 30 min bath ratio was 1:10 (Bath ratio means the ratio of dry laundry in kilograms to the volume of wash liquor in litres).

3. RESULTS AND DISCUSSION

3.1. Steps of Nanosilica Synthesis by Sol Gel Chemistry

In the first step, nanosilica particles were tried to synthesize via sol gel process. The selected sol gel process was including different stages, they were;

- Achieving hydroxide functionalized product by the hydrolysis reaction,
- Achieving oxidized product by the condensation reaction,
- Occuring gelling as a result of a network solution and viscosity increase,
- Increasing of covalent crosslinks and gelation by aging,
- Drying,

might be listed. Our nanosilica synthesis studies were based on four parameters for condensation and gelling with TEOS.

3.2. Characterization and Discussion of Nanosilica Synthesis

3.2.1 Determination of optimum H_2O:Si and Ethanol:TEOS mole ratios

Studied experiments in literature was took into account while determining the initial prescription H_2O /Si molar ratios. S_N2 reaction occured while condensation reaction of TEOS which is as follows (Figure 3.1) ;

X=R or H ; Y= Si

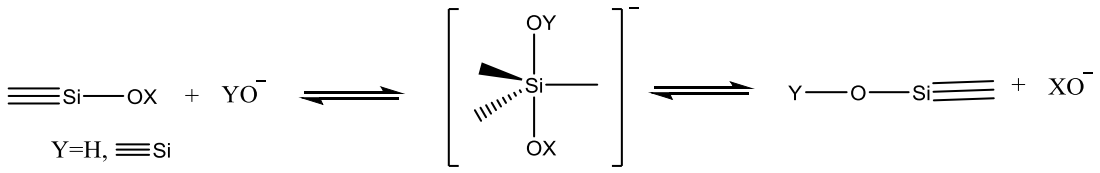

Figure 3.1: Condensation reaction of TEOS.

The reactions were terminated when the enough gelling was occured and the gelling durations were noted in the Table 3.1. At the end of the gelling reactions, particle sizes were measured and againts to H_2O:Si and Ethanol:TEOS molar ratios as indicated in Table 3.1.

Table 3.1: H_2O:Si and Ethanol:TEOS mole ratios vs gelling durations and particle sizes.

H_2O:Si	Ethanol:TEOS	Gelling Duration(h)	Particle Size(nm)
1	1	48-72	65
1	2	72-92	104
1	3	no gelling	---
2	1	24-48	47
2	2	96-120	120
2	3	no gelling	---
4	**1**	**24-36**	**30**
4	2	48-72	32
4	3	no gelling	---
8	1	36-60	154
8	2	72-96	208
8	3	no gelling	---
16	1	48-72	154
16	2	84-120	253
16	3	no gelling	---
32	1	96-120	6000
32	2	no gelling	---
32	3	no gelling	---

When H_2O:TEOS molar ratio was high, hydrolysis was happening faster and hydrolysis yield was increasing. Consequently, participation reactions which were formed by crosslinking was getting slower and duration of gelling reaction was getting longer. While increasing of H_2O:TEOS molar ratio from 1 to 4, the gelation duration was gradually shortened because of the alcohol addition. About H_2O:TEOS molar ratio was 2, reaction released alcohol as a byproduct in consequence of participation reaction. Between 4 to 32, gelation duration was getting longer. Hereby, smallest particle size was obtained 30nm shown in Figure 3.2 when H_2O:TEOS mole ratio was '4' and EtOH:TEOS molar ratio was '1' (optimum mole ratios) which was written in bold in the Table 3.1.

3.2.2 Determination of optimum catalyst

Same observations were repeated for different catalysts using with optimum mole ratios in same conditions to see the effect of pH and catalyst difference on the particle size and gelling duration of the silica . Used catalysts and results were gave in Table 3.2 moreover the obtained data was plotted in Figure 3.3.

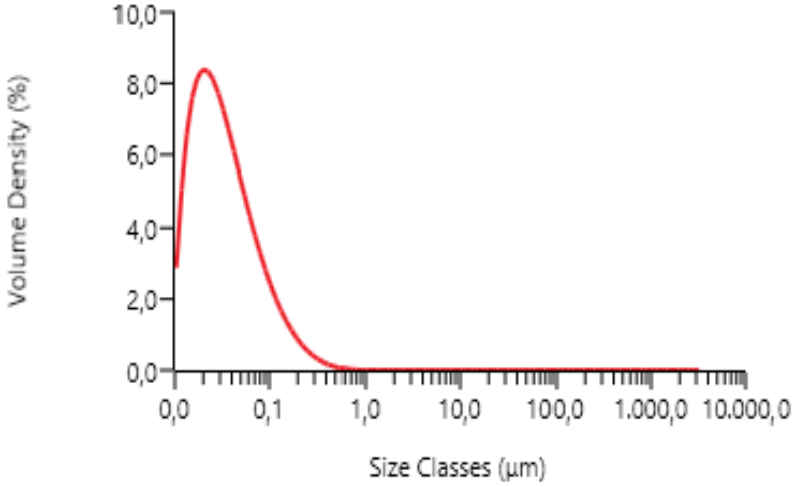

Figure 3.2: Particle size analysis of nanosilica which was synthesized with optimum mole ratios.

Table 3.2: Effect of catalyst and pH changes against to particle size and gelling duration.

Catalyst	pH	Gelling Duration(h)	Particle Size(nm)
HCl	<2	42	52
NH_4OH	7<pH<8.5	48	30
NH_4OH	>9	107	---
HF	2<pH<7	96	25

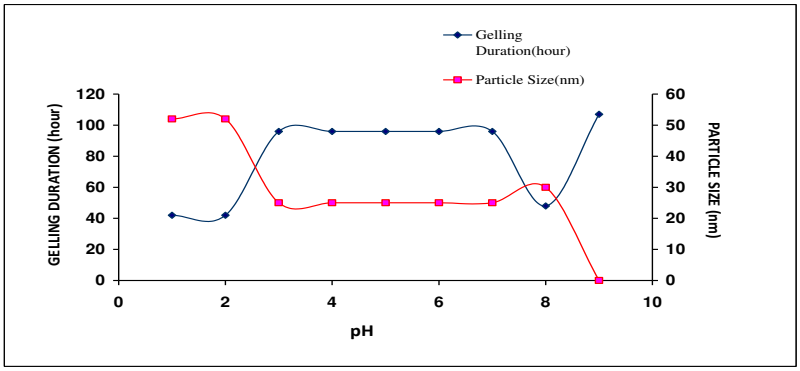

Figure 3.3: Gelling duration and particle size againts to pH.

Particle size and gelling duration were almost constant at 2<pH<7 which was shown in Figure 3.3. Gelling duration and particle size were inversely proportional when pH>9 and pH<2. Smallest particle size obtained with HF, but it had a long gelling duration so, ammonia catalyst was choosen for pH 7 to 8.5. Also, optimum

mole ratios for nanosilica synthesis with ammonia catalyst was obtained when the H_2O:TEOS mole ratio was '4' and EtOH:TEOS molar ratio was '1'.

3.2.3 SEM analysis of synthesized nanosilica

Synthesized nanosilica at optimum conditions was characterized with SEM-EDS which is shown in Figure 3.4. Bigger particle size was determined from the SEM images according to particle size analysis because nanosilica particles was aglomerated while drying for the sample preperation of the SEM.

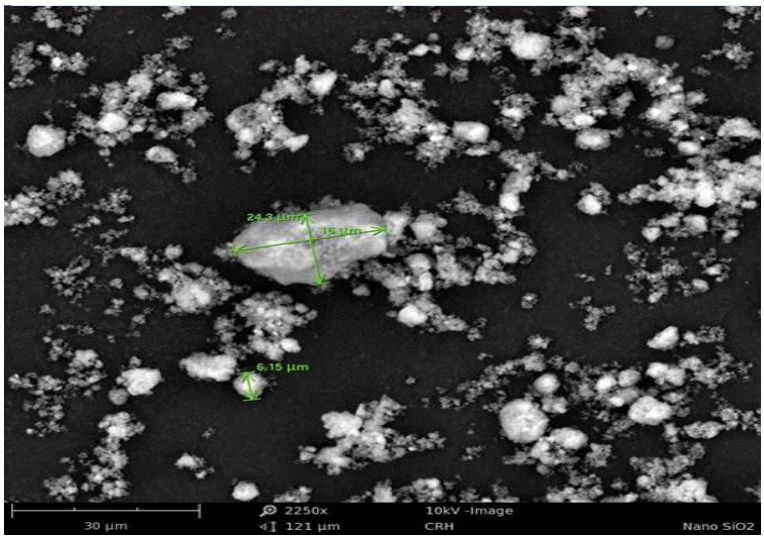

Figure 3.4: SEM images of nanosilica which was synthesized at optimum conditions.

3.3. Characterization and Discussion of Encapsulated Nanosilica

Sulphonic acid containing polymer component AMPS is recomended in literature for silica encapsulation, also acrylamide comonomer was choosen for copolymerization. Therefore encapsulation of nanosilica was performed with poly(AAm), poly(AMPS) and poly(AAm-co-AMPS) as indicated in Figure 3.5, Figure 3.6 and Figure 3.7.

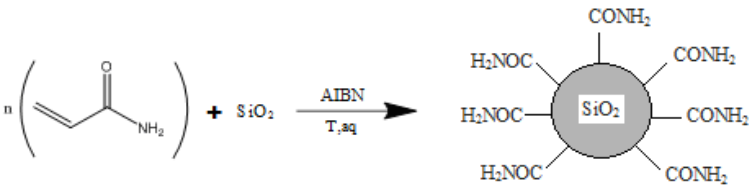

Figure 3.5: Nanosilica encapsulation with poly(AAm).

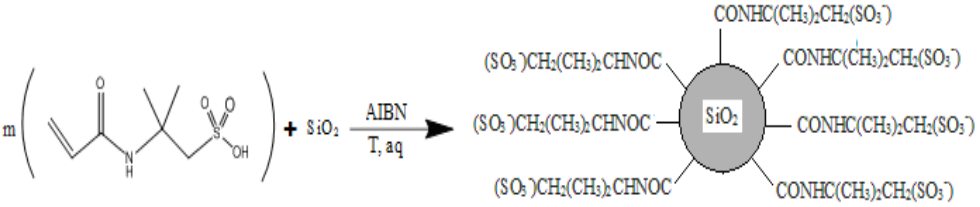

Figure 3.6: Nanosilica encapsulation with poly(AMPS).

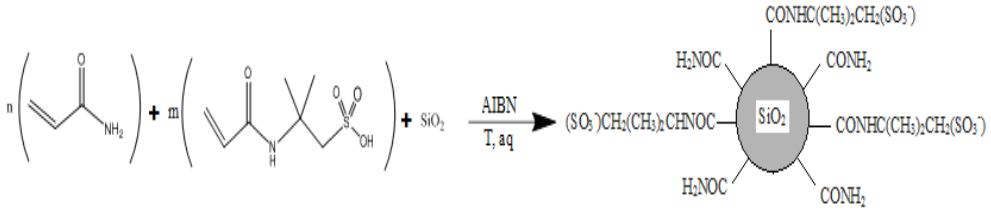

Figure 3.7: Nanosilica encapsulation with poly(AAm-co-AMPS).

3.3.1 Thermal gravimetric analysis of encapsulated nanosilica

Glass transition temperature of samples was measured with TG/DTA. Powdered samples of dried gels were heated in the TG/DTA at 80°C for 20 min to vaporize residual water in the samples, cooled to 25°C at 20°C/min, held at 25°C for 5 min and then heated to 400°C at 10°C/min and Table 3.3 was formed. Tg of poly(AAm-co-AMPS) hybrid samples could not be observed because while glass transition temperature analysis of the first copolymer, the sample poured from the container so analysis failed. These copolymer samples were quite a foaming materials, so could not be tried for other copolymer samples.

Table 3.3: TGA analysis of samples.

Sample	Tg(˚C)
1	165
2	167
3	122
4	--
5	--
6	128
7	--
8	--

3.3.2 SEM-EDX analysis of encapsulated nanosilica

Synthesized polymer Antipill hp gave the best antipilling result so Antipill hp was examined with SEM-EDX and shown in Table 3.8 (a) and (b). Table 3.4 and Table 3.5 showed that, obtained two different points have the three main component of our hybrid polymer (Antipill hp): sulfur, silicon and nitrogen. Antipill hp indicated that

good dispersion was achieved. Ratios of chemical elements in the Antipill hp was calculated from the % ratios of sample 8 (Give in Table 3.6) Analyzed sulfur and nitrogen ratios were similar with calculated ratios but silicon ratio was found half of the calculated ratio.

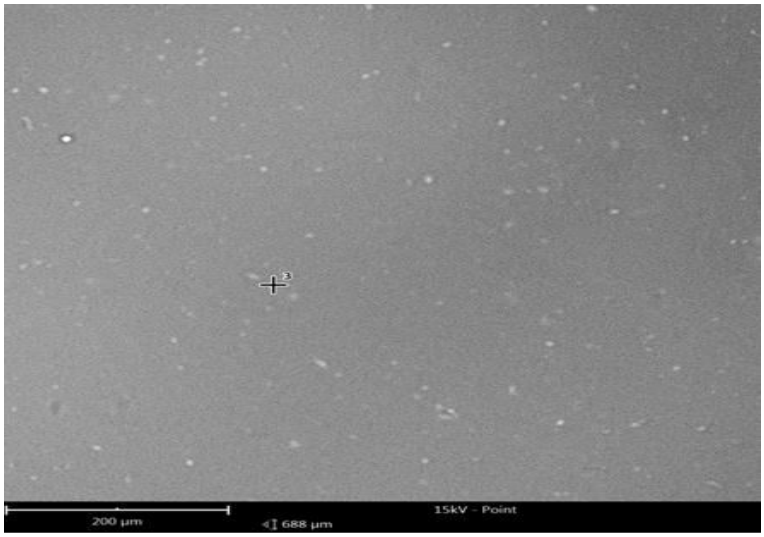

Figure 3.8: SEM image of Antipill hp, poly(AAm-co-AMPS) capsulated nanosilica point 3.

Element Number	Element Symbol	Element Name	Confidence	Concentration	Error
16	S	Sulfur	100.0	8.8	0.7
14	Si	Silicon	100.0	3.8	1.1
7	N	Nitrogen	100.0	13.8	1.7

Table 3.4: % Element concentrations of obtained point in the Figure 3.8.

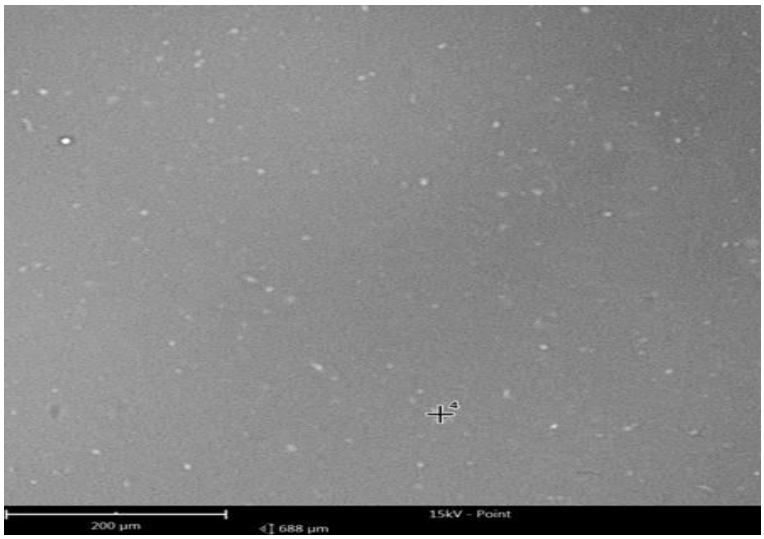

Figure 3.9: SEM image of Antipill hp, poly(AAm-co-AMPS) capsulated nanosilica.

Table 3.5: % Element concentrations of obtained point in the Figure 3.9.

Element Number	Element Symbol	Element Name	Confidence	Concentration	Error
16	S	Sulfur	100.0	8.9	0.9
14	Si	Silicon	100.0	4.7	1.2
7	N	Nitrogen	100.0	14.2	2.2

Table 3.6: % Calculated ratios of elements in the Antipill hp.

Element Number	Element Symbol	Element Name	Concentration
16	S	Sulfur	8.5
14	Si	Silicon	8.0
7	N	Nitrogen	14.5

3.3.3 FT-IR analysis of encapsulated nanosilica

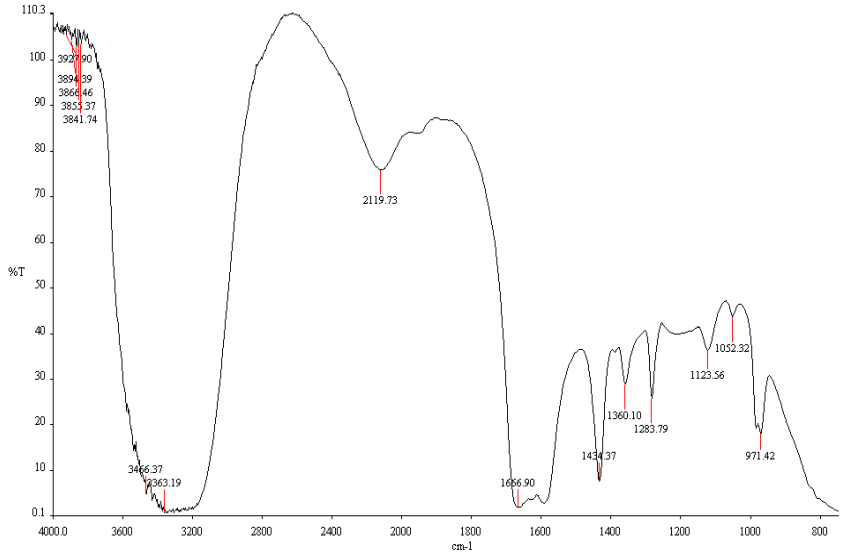

Figure 3.10: IR spectrum of AAm monomer.

The FTIR-ATR spectrum of Acrylamide(AAm) was characterized by the presence of the following vibration frequencies: 1666 cm^{-1}, typical of the C=O stretch; 3363cm^{-1}, NH_2 stretch; 1434 cm^{-1}, characteristic of primary amides; 1052 cm^{-1}, C–N stretch of aliphatic amines; 971 cm^{-1}, stretch of C═CH_2 group (See Figure 3.10).

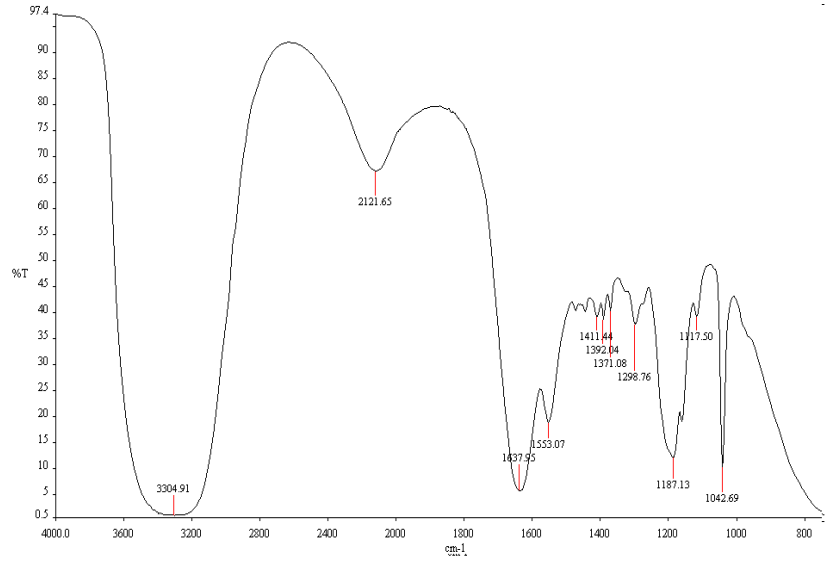

Figure 3.11: IR spectrum of AMPS monomer.

In the FTIR-ATR spectrum of 2-Acrylamido-2-methylpropane sulphonic acid; 3304 cm^{-1}, N–H stretch of 2° amines; 1637 cm^{-1}, C=O stretching vibration of -CONH groups; and 1187 cm^{-1}, stretching vibration of -SO_3H group were found (See Figure 3.11).

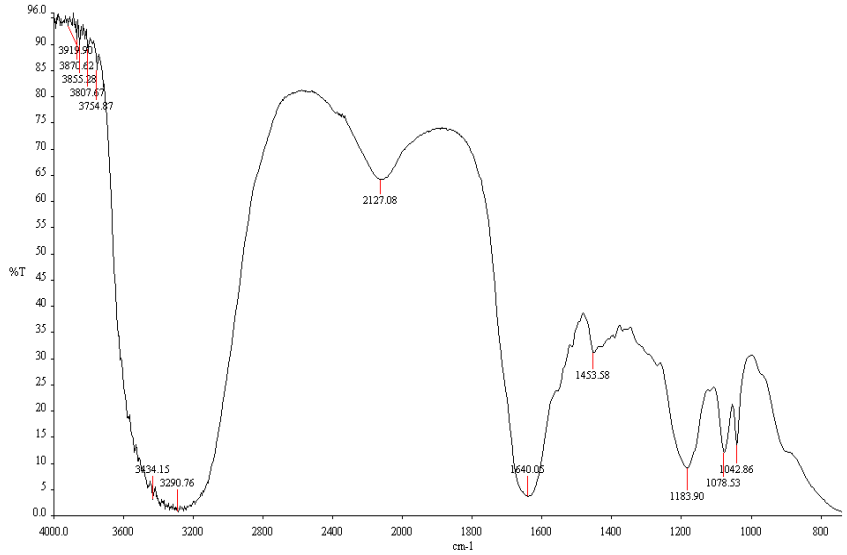

Figure 3.12: IR spectrum of poly(AAm-co-AMPS) capsulated nanosilica (Antipill hp).

In the FTIR-ATR spectrum of copolymer, the following bands were obtained. The characteristic bands of NH_2 was shown at 3290 cm^{-1}; amino and amidic carbonyl groups were shown stretching vibration peaks at 3434 cm^{-1} and 1640 cm^{-1} for respectively. 1453 cm^{-1} was C–H band; sulphonic acid (SO_2) symmetric band was found at the 1078 cm^{-1} and 1183 cm^{-1} was C–N stretch band. The peaks situated

around 1042 cm^{-1} and 1183 cm^{-1} are the characteristic peaks of the asymmetric and symmetric bands of sulphonate groups in AMPS unit. Furthermore stretching vibration peak of C═CH_2 group was disappeared (See Figure 3.12 and Figure 3.13)

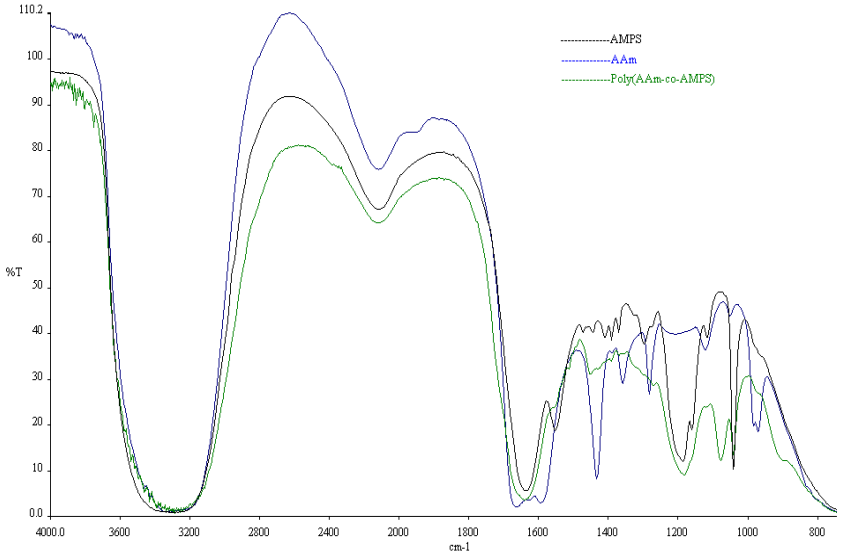

Figure 3.13: IR spectrum of monomers and Antipill hp in the same graph.

3.3.4 Gel permeation chromatography analysis of poly(AAm-co-AMPS)

Antipill hp, GPC analysis could not be done because of the silica remaining risk in the columns. Therefore Antipill hp was prepared without nanosilica for once with the same polymerization method and condition to measure the molecular weight of polymer (See Figure 3.14). And 0.05g synthesized polymer dissolved in 100ml distilled water and mixed with the magnetic stirrer for 12 hours and then analysed with the GPC/SEC system when water as the eluent at a flow rate of 0.7 mL/min. Dedector response againts to retention volume was shown in Figure 3.15 and calculated results of poly(AAm-co-AMPS) were shown in Table 3.7. High molecular weight polymer was obtained because free radical polymerization was occured and monomers created a long chain by attaching each other freely, there was not any restriction for polymerizing area such as silica.

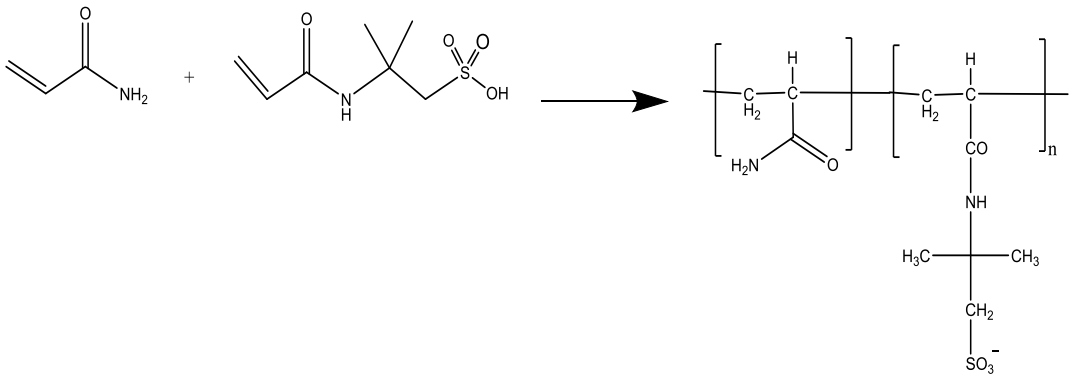

Figure 3.14: Radical copolymerization of acrylamide and 2-acrylamido-2-methyl-1- propane sulphonic acid.

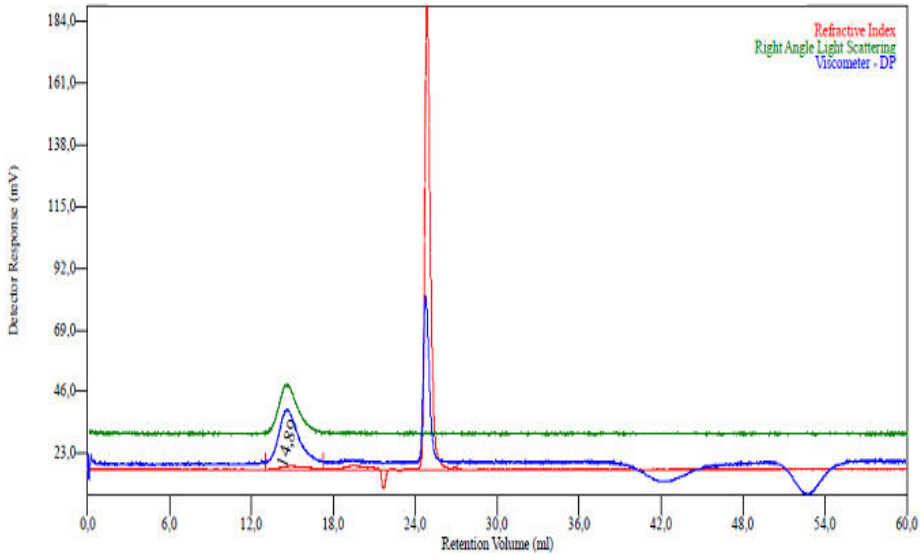

Figure 3.15: GPC-Dedector response againts to retention volume of poly(AAm-co-AMPS).

Table 3.7: GPC Multidedectors results of poly(AAm-co-AMPS).

Multidedectors Results	
Mn(Daltons)	7.241×10^6
Mw(Daltons)	1.147×10^7
Mz(Daltons)	1.659×10^6
Mw/Mn	1.584

3.4. Characterization and Discussion of Encapsulated Nanosilica Applied Viscose Fabrics

The reaction accurs between viscose and the sample which was shown in Figure 3.16 by heat and catalyst ($K_2S_2O_7$) effect.

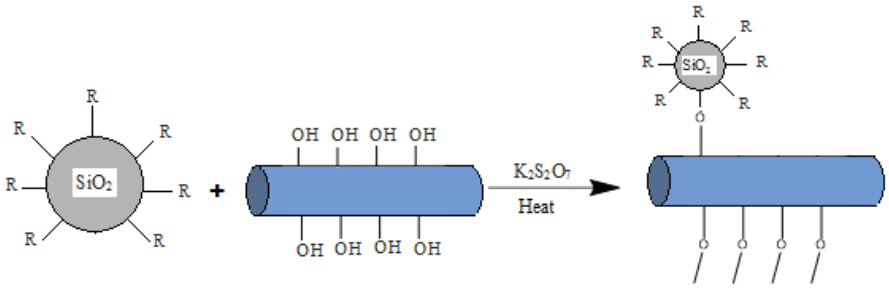

Figure 3.16: Nanosilica capsule and viscose fabric reaction

3.4.1 Antipilling analysis of viscose fabrics

40g/l sample applied knitted and woven viscose fabric were analyzed for pilling property. BS EN ISO 2001 standard was applied and treated viscose fabric samples were rotated 2000 tours with Martindale 06A . Results were shown in Table 3.8 and photos shown in Figure 3.17. Best antipilling results were obtained in sample 8. 5% (w/w) AMPS and 5%(w/w) AAm were selected as the optimum ratios of monomers and following studies were made with these ratios. Sample 8 was called with the name Antipill hp.

Table 3.8: Pilling ratings of viscose fabrics.

Sample	Viscose 1 Rating	Viscose 2 Rating
Control	1-2	1-2
1	2-3	2-3
2	3-4	3-4
3	2-3	2-3
4	3	3
5	3-4	3-4
6	3-4	3-4
7	3-4	3-4
8	4	4-5

Control : Untreated fabric
Viscose 1 : Khaki Knitted Fabric
Viscose 2 : Khaki Woven Fabric

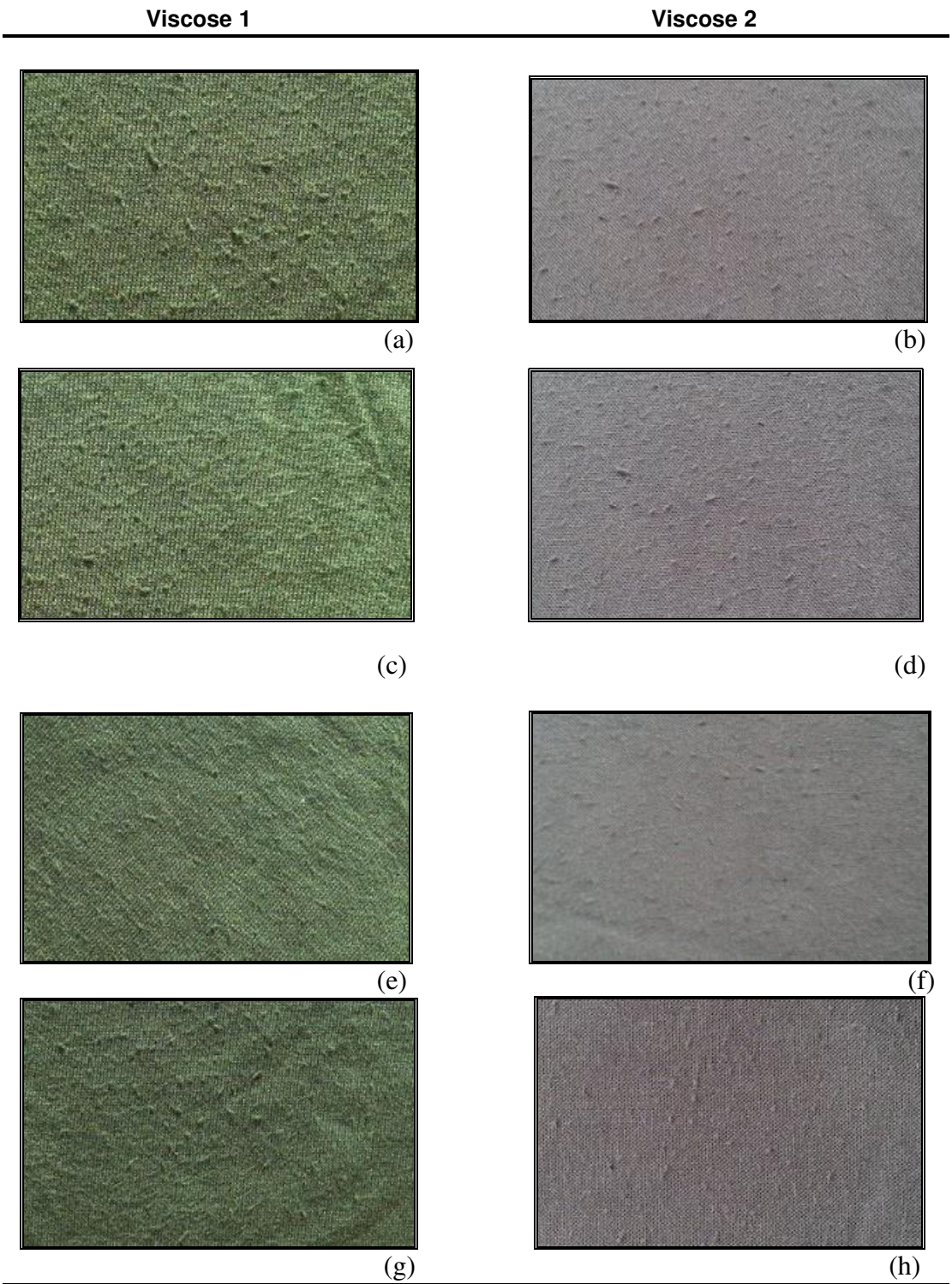

Figure 3.17: Viscose fabric photos after the 2000 rotation with Martindale which were given sample numbers in table 3.8.

(a) Viscose 1- control (b) Viscose 2-control (c) Viscose 1-sample 1 (d)Viscose 2-sample 1 (e) Viscose 1-sample 2 (f) Viscose 2-sample 2 (g) Viscose 1-sample 3 (h) Viscose 2-sample 3.

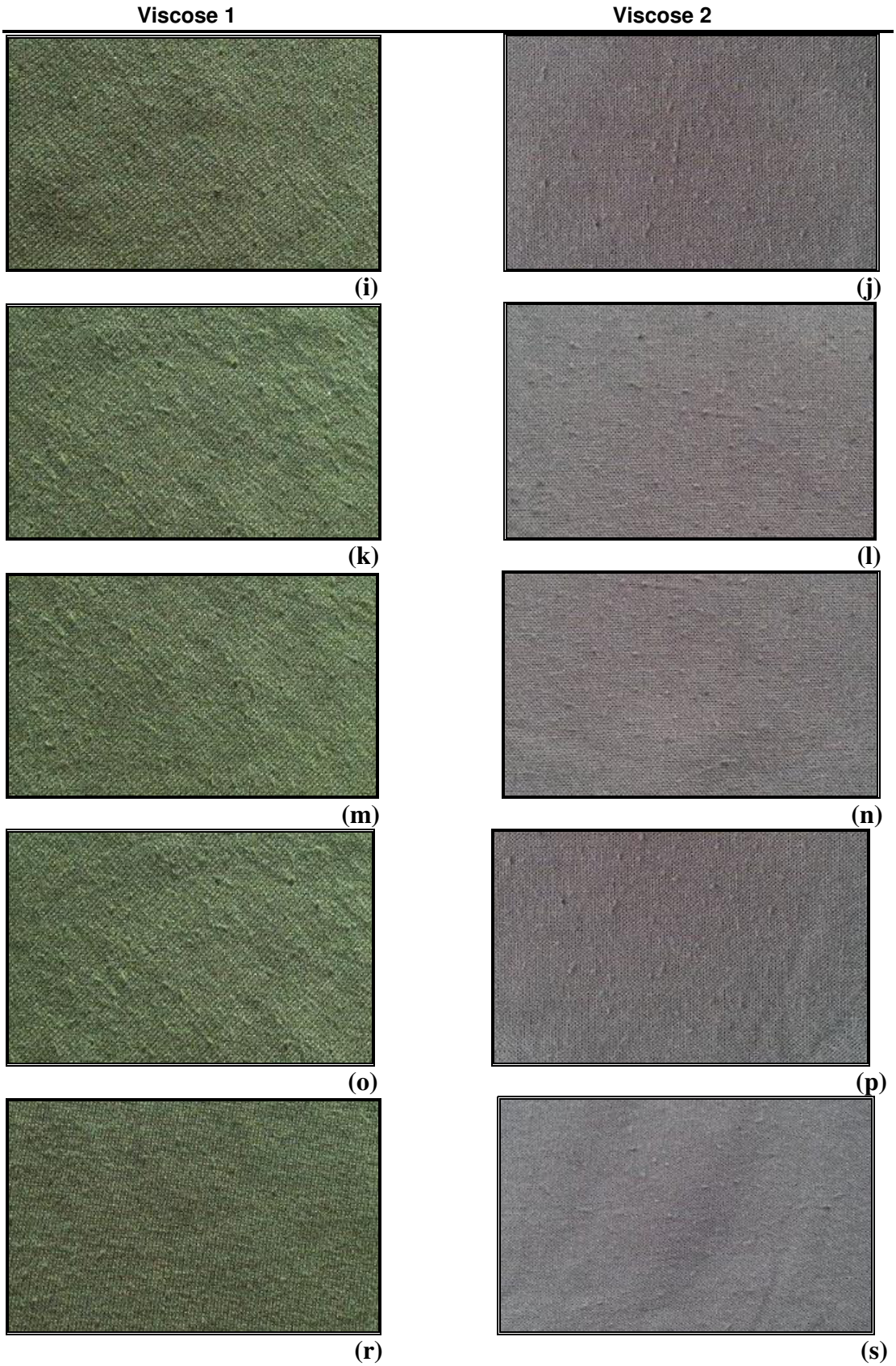

Figure 3.18: Viscose fabric photos after the 2000 rotation with Martindale which were given sample numbers in table 3.8 (more).

(i) Viscose 1-sample 4 (j) Viscose 2-sample 4 (k) Viscose 1- sample 5 (l) Viscose 2- sample 5 (m) Viscose 1- sample 6 (n) Viscose 2- sample 6 (o) Viscose 1-sample 7 (p) Viscose 2- sample 7 (r) Viscose 1-sample 8 (s) Viscose 2-sample 8

3.4.2 SEM analysis of synthesized nanosilica

40g/l Antipill hp applied white knitted viscose fabric and untreated fabric were examined with SEM-EDX. Difference of untreated and treated fabrics was shown Figure 3.19 and Figure 3.20, particles were visible on the fibres in Figure 3.21, Figure 3.22 and Figure 3.23 images which were magnified, fibers could be seen more clear. Particles surface distribution were seen in Figure 3.20 particles was measured in Figure 3.23, 2.7 micrometers and 457 nanometers were obtained.

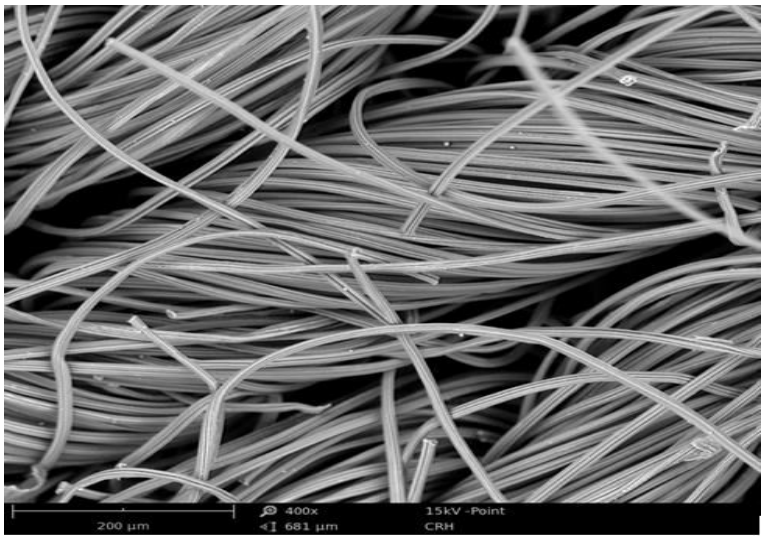

Figure 3.19: SEM image of untreated white knitted viscose fabric.

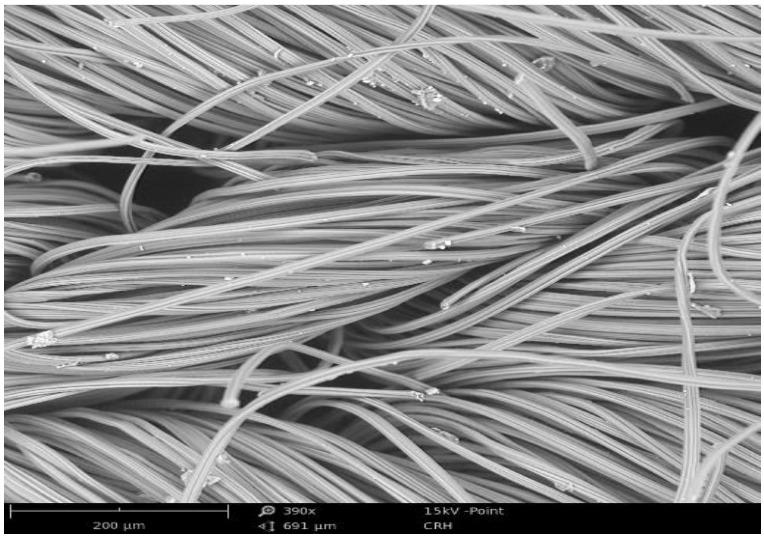

Figure 3.20: SEM image of 40g/l Antipill hp applied white knitted viscose fabric.

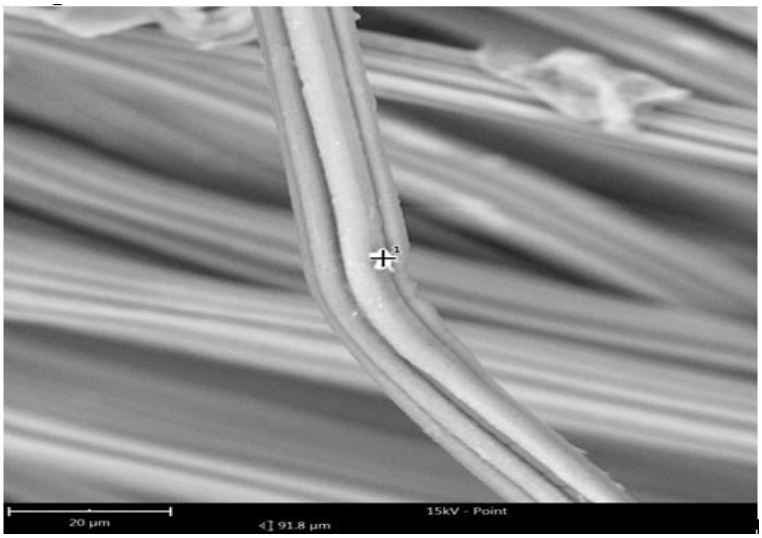

Figure 3.21: SEM image of untreated white knitted viscose fabric.

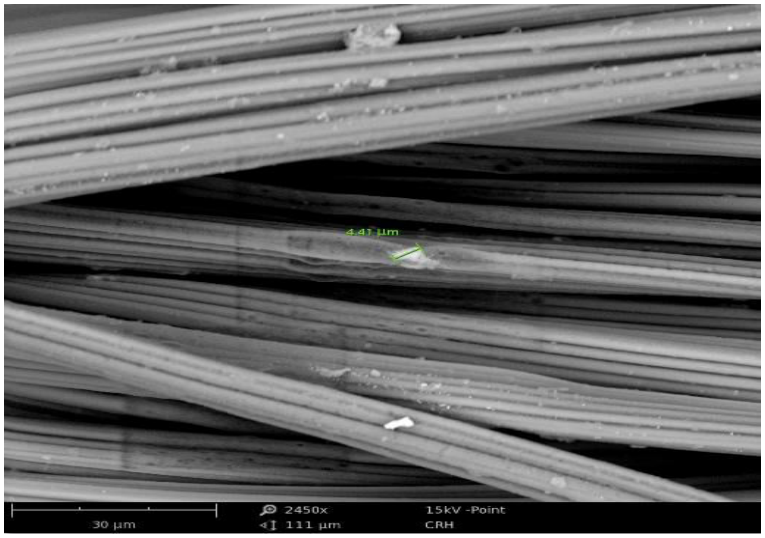

Figure 3.22: SEM image of 40g/l Antipill hp applied white knitted viscose fabric.

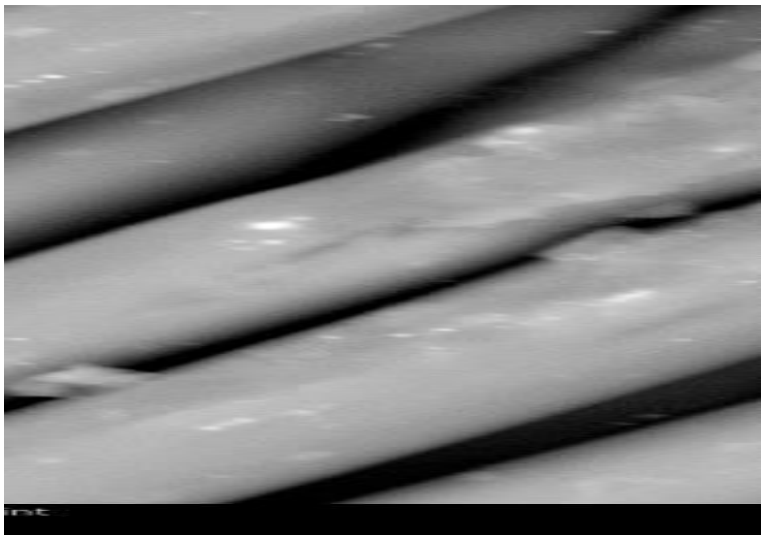

Figure 3.23: SEM image of 40g/l Antipill hp applied white knitted viscose fabric.

3.4.3 Strength Analysis of Viscose Fabrics

Similar results were obtained for burst strengths and tensile strengths of untreated(control) and treated fabric. So results in Table 3.9 and Table 3.10 showed that; Antipill hp did not reduced the strenght of fabrics. On the contrary, Figure 3.24 showed, Antipill hp provided a slight increase in tensile strength of fabric.

Table 3.9: Tru-bursting strengths of Antipill hp applied white knitted fabric**.**

Concentration	Pressure (kPa)	Distension (mm)	Time (s)
Control	486	10.5	22.5
20 g/l Antipill hp	487	10.4	20.8
40 g/l Antipill hp	485	10.6	21.7
60 g/l Antipill hp	485	10.5	22.0
80 g/l Antipill hp	482	10.3	21.7

Table 3.10: Tensile strength of Antipill hp applied white woven fabric.
(Standard : ISO 13934-1 200-100mm min)

Concentration	Force (N)	Elongation (%)
Control	963	37.00
20 g/l Antipill hp	1005	37.70
40 g/l Antipill hp	1162	40.02
60 g/l Antipill hp	1139	46.31
80 g/l Antipill hp	1071	44.19

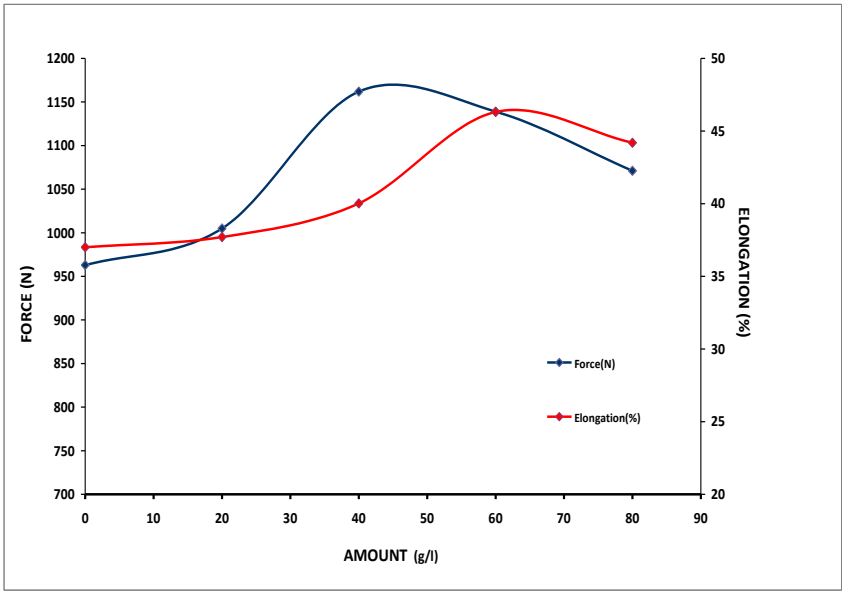

Figure 3.24: Force and Elongation changes of tensile strength of viscose fabric againts to applied amount of sample.

3.5. Comparison of Antipill hp with the existing products used in the market

After treatment the green knitted viscose fabric was analyzed for pilling property. BS EN ISO 2001 standard was applied and treated viscose fabric samples were rotated 2000 tours with Martindale 06A . Results were shown in Table 3.11 and photos of fabrics shown in Figure 3.25. Best antipilling results were obtained in Antipill hp and Antipill jet conz. But tensile strenghts of existing products reduced after the treatment especially Antipill jet conz (See Table 3.12). Only Antipill hp maintained the tensile strength of untreated fabric.

Table 3.11: Pilling ratings of green knitted viscose fabric.

Sample Numbers	Sample	Rating
	Control	1-2
1	0.5% Biopolish 300	2-3
2	1% Biopolish 300	2-3
3	20g/l Antipill jet Conz	4
4	40g/l Antipill jet Conz	4-5
5	20g/l Idrocap 982	3
6	40g/l Idrocap 982	3-4
7	20g/l Antipill hp	4
8	40g/l Antipill hp	4-5

Table 3.12: Tensile strength comparison of khaki woven fabric for different products (Standard : ISO 13934-1 200-100mm min).

Sample	Max Force(N)	Elongation(%)
Control	305.36	17.50
0.5% Biopolish 300	286.88	19.77
1% Biopolish 300	267.55	21.87
20g/l Antipill jet conz	278.90	15.49
40g/l Antipill jet conz	230.36	15.13
20g/l Idrocap 982	294.91	16.15
40g/l Idrocap 982	288.12	15.12
20g/l Antipill hp	306.21	18.18
40g/l Antipill hp	308.30	18.24

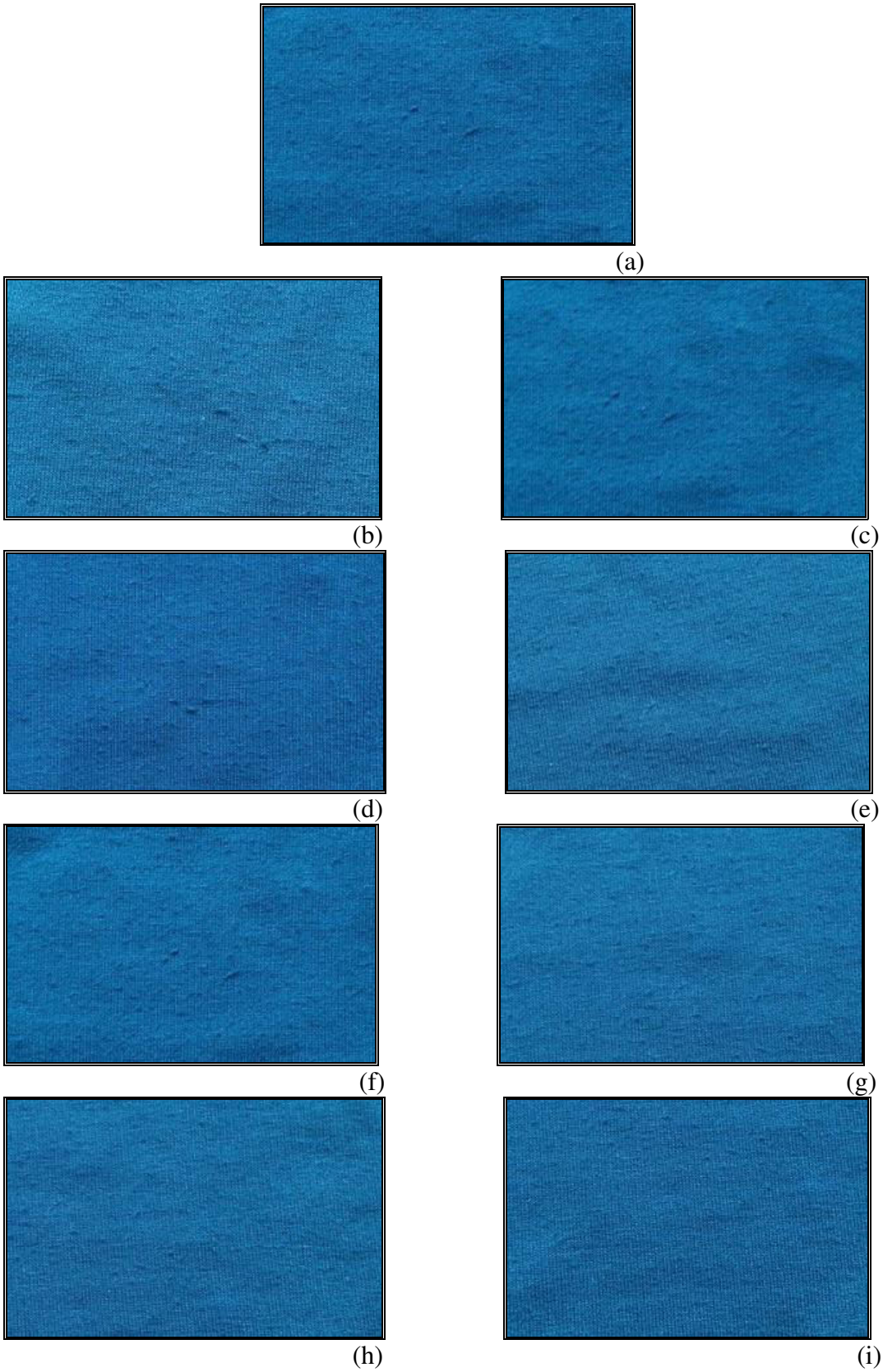

Figure 3.25: Photos of viscose fabrics after the 2000 rotation which are indicated in table 3.11. (a)Control (b)sample 1 (c)sample 2 (d)sample 3 (e)sample 4 (f)sample 5 (g)sample 6 (h)sample (i)sample 8

4. CONCLUSION

This work is aimed at developing and investigating silane based organic–inorganic hybrid. Poly[acrylamide-co-(2-Acrylamido-2-methylpropane sulphonic acid)] nanocomposite possessing unique properties, which can be used to improve the antipilling properties of the viscose fabric. A series of nanosilica synthesized with different mole ratios and catalysts. All the samples were analysed with particle size analyser to obtain minimum particle size and optimum ratios. With these ratios ~30nm silica synthesized. SEM analysis showed that, synthesized silica agglomerated while drying. Then silica encapsulated with different weight ratios of AAm and/or AMPS monomers. To determine the optimum ratios of monomers, samples of encapsulated nanosilica were applied to viscose fabrics and pilling ratings were identified. Antipill hp gave the best results and studies continued with this sample. SEM and FTIR-ATR characterizations proved that; silica could be capsulated with poly[acrylamide-co-(2-Acrylamido-2-methylpropane sulphonic acid)] but particle sizes detected from SEM images above the particle size observation from particle size analyser because of the silica agglomeration while sample preparation of SEM. Antipill hp applied viscose fabrics were compared with existing products and results showed that; unlike its competitors it did not not make the loss of strength, and showed a very good antipilling effect. As we aimed, it improved the weakness of viscose without damaging the existing features.

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