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M.Sc. in Textile Engineering

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UNIVERSITY OF GAZİANTEP

GRADUATE SCHOOL OF

NATURAL & APPLIED SCIENCES

AN INVESTIGATION ABOUT THE APPLICATION OF A SPECIFIC MEDICAL PLANT IN TEXTILE

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An Investigation about the Application of a Specific Medical Plant in Textile

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Supervisor

Assoc. Prof. Dr. Cem GÜNEŞOĞLU

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Gülizar MANTAR

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This is to certify that we have read this thesis and that in our consensus/majority opinion it is fully adequate, in scope and quality, as a thesis for the degree of Master of Science/Doctor of Philosophy.

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I declare that the related thesis was written and all the relevant literature was used in accordance with the academic and ethical rules.

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ABSTRACT

AN INVESTIGATION ABOUT THE APPLICATION OF
A SPECIFIC MEDICAL PLANT IN TEXTILE

MANTAR, Gülizar

M. Sc. in Textile Engineering

Supervisor: Assoc. Prof. Dr. Cem GÜNEŞOĞLU

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Medicinal plants have been utilized from the past to the present mainly for medical and esthetical purposes. The new methods to use active substances in the plants allow the medicinal plants to be used almost in all areas of our lives.

Textile industry is one of the most suitable area in terms of the applicability of medicinal plants. Today, it is possible to add a lot of features to textile surfaces thanks to innovations in finishing processes.

In this study, *Aloe vera* leaf gel was used in order to utilize its antibacterial and antibiotic properties. The gel was applied on the cotton fabrics by microencapsulation method. A serious of tests were applied to fabrics in order to determine their breaking strength, abrasion resistance, water vapor permeability, dry and wet rubbing fastness, color difference, and antibacterial effectiveness.

Keywords: medicinal plant, *Aloe vera* gel, microencapsulation, cotton fabric, antibacterial property.

ÖZET

ÖRNEK BİR TIBBİ BİTKİNİN TEKSTİLDE UYGULANMASI KONULU ARAŞTIRMA

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111 sayfa

Tıbbi bitkiler geçmişten günümüze daha çok medikal ve estetik amaçlar için kullanılan bitkilerdir. Günümüzde bitki ekstraktlarını elde etmek amacıyla ortaya çıkan yeni metodlar tıbbi bitkilerin hayatımızın neredeyse her alanında kullanılmasına olanak sağlamıştır.

Tıbbi bitkilerin uygulanabilirliği açısından tekstil endüstrisi en uygun alanlardan biridir. Bitim işlemlerinde yapılan yenilikler sayesinde tekstil yüzeylerine birçok özellik katmak mümkün hale gelmiştir.

Bu çalışmada, anti bakteriyel ve antibiyotik özelliklerinden faydalanmak amacıyla *Aloe vera* yaprak jeli kullanılmıştır. *Aloe vera* yaprak jeli mikroenkapsülasyon işlemi ile pamuk kumaşlara uygulanmıştır. Kumaş numunelerinin kopma mukavemeti, aşınma dayanımı, su buharı geçirgenliği, kuru ve yaş sürtme haslığı, renk farkı ve antibakteriyel etkinliği gibi özelliklerini belirlemek amacıyla bir dizi test gerçekleştirilmiştir.

Keywords: tıbbi bitki, *Aloe vera* jel, mikroenkapsülasyon, pamuklu kumaş, antibakteriyel etkinlik.

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ABBREVIATIONS

AVG	*aloe vera* gel
AG	arabic gum
SAO	sweet almond oil
BTCA	butanetetracarboxylicacid
SHP	sodiumhypophosphite
STS	sodium thiosulfate
SDS	sodium dodecyl sulfate
UV	ultraviolet
MAP	medicinal and aromatic plant
PTFE	polytetrafluoroethylene
PCM	phase change material
TGA	thermogravimetric analysis
DSC	differential scanning colorimeter
SEM	scanning electron microscope

CHAPTER 1
INTRODUCTION

1.1 Introduction

Health and hygene are the primary requirements of a fabric thus being considered to be an important and inevitable parameter for garments which are in direct contact with human body [1, 2]. Because clothes and other textile goods are usually a great substrate regarding microbial and bacterial growth occurring with appropriate humidity, nutrients and suitable temperature conditions [3, 4]. They are great carriers of numerous kinds of organisms such as pathogenic and odor-generating bacteria, and mold fungus due to the adhesion of those microorganisms to the surface of the fabrics causing health-related difficulties of the wearer. [2, 5, 6]. In order to defend the person through this sort of an infection, the sheet fabric could be treated with a finishing material having antimicrobial property [6].

Antimicrobials are generally defined as the agents which possibly kill microbes (bactericidal) or lessen the development of pathogens (bacteriostatic) [5, 7, 8]. Although the synthetic antimicrobial agents such as Polyhexamethylene biguanide (PHMB), triclosan, metal and the salts of them, nano silver, formaldephenols, quaternary ammonium constituents, and organometallics are very effective against a variety of microbes and allow the long lasting impact on fabrics, some problems occur due to the unwanted side effects, toxicity, actions about non-target microbes and pollution of water and environment [3, 4, 6, 9, 10].

Plant based antimicrobials possess enormous healing potential [11]. They are known for their healing as well as disease-curing qualities for hundreds of years [12, 13]. Botanical researches demonstrate that several plant varieties exhibit antibacterial, antiviral and antifungal houses [10]. To be able to minimize these kinds of risks related to the difficulties with synthetic antimicrobial agencies, antimicrobial textiles

depending on plant components containing active substances are requested increasingly [3, 9, 10].

These active substances are usually phenolic, terpenoids, flavonoids, alkaloids, polypeptide, polyacetylenes etc. acting as antibacterials [2]. They are commonly gained from some sections of herbs including the flowers, barks, roots, leaves, fruits, plant seeds that possess antimicrobial task [1, 10]. The reasonably lower occurrence of side effects of plant based products in comparison with modern manmade pharmaceutical products, together with being low cost, can possibly be utilized as analternative in different areas for textile [3, 9].

The main limitation in the natural antimicrobial finishing materials is non- durability of the finish because they are not able to form any attachment with the textile products. In order to solve or improve this problem microencapsulation technique which is mostly used as a finishing process in textile, may be a good choice. In this method, the microscobic quantity of active substances are entrapped inside lean films of any wall substance such as modified starch, salt alginate, nicotine, gum acacia, etc. and applied on the textile products [6, 14-16].

1.2. Objective of Study

The thesis is focused on to apply extracts / fractions of gel of the *Aloe vera (L.) Burm.* (Syn: *Aloe barbadenis Miller*) plant leaves, antibacterial property of which is proved with various reports in the literature, onto the %100 cotton fabric by the way of microencapsulation. An optimization is also aimed to succeed in extraction, microencapsulation and application of these microparticles on to the textile surfaces together with gaining of antimicrobial property. Thus, it will be possible to produce garments with high value added as a new product for clothing market from the fabrics treated with extract/microcapsule fraction having the highest activity determined with measurement of antibacterial property shown against to the selected microorganisms and evaluation of important textile-related properties like abrasion, water vapor permeability, fastness, tensile strength and color difference.

1.3. Structure of the Thesis

Chapter 2 is about medicinal plants. In this chapter, history of the plants, functional parts and their present use especially in medical area and market situation is explained in details. In chapter 3, *Aloe vera* plant is examined in a detailed manner including its physical and chemical properties, extraction and application methods of the gel which is gained from the plant. Microencapsulation process and techniques are given in chapter 4. All the chemicals used, processes and experimental studies are summarized in chapter 5. Results and discussions take place in the last chapter. (Chapter 6)

CHAPTER 2
MEDICINAL PLANTS

2.1. History of Medicinal Plants

Plants have always been an integral part of traditional medicine throughout human history and they still continue to make important contributions to healthcare and livelihoods [17, 18]. In the past, people have relied upon nature with regards to basic needs to produce various sources that bring about fertilizers, food-stuffs, clothing, transportation, animal shelters, flavors, perfumes, and medications [19, 20]. Man has mainly gathered vegetable and animal sources with regards to antimicrobial qualities and has utilized vegetable ingredients as beneficial agents to treat and heal a variety of illnesses [13, 20-24]. The primary recorded document that is several plant treatments referred to the clay tablets, schedules through Paleolithic age of approximately 50, 000 B. C. that has been seen in the tomb of an Neanderthal person in the Hakkari, far southeast side of Turkey [25].

Because medical plants which are the wealthiest resource of medicines in conventional applications of remedies, have performed an important function in the preservation of human health in the historical civilizations for a long time of time, they still supply to the most of the people around the Earth special chemical materials offering as pattern for the improvement of newer beneficial agencies [13, 19, 20, 26-29].

2.2. Medicinal Plants; Definition, Functional Parts and Applications

Medical plants are the plants and elements of the plants such as vegetable seed, fruits, roots, leaf, bark or floral that are utilized specifically or indirectly mainly for their medicinal as well as aromatic qualities in pharmacy, in perfumery or insectic

repellent agents, fungicidal or other requirements like this, in fresh or dried, crushed, cut or powdered form [17, 20, 30-33]. The main advantages of using plant made remedies are that they are reasonably less hazardous compared to man made alternatives, supplying unique beneficial aspects and cost-effective treatment [11, 34]. The basis for this use is that these vegetation comprise so called effective substances such as alkaloids, glycosides, vital herbal oils and other miscellaneous effective materials which affect physiological operations of dwelling organisms, including humankind [30].

They can make improvements in medicinal are by two ways: (1) they may be preffered as a base for the advancement of a drug, an inherent formula for the production of new medicines, or; (2) they may be used as a phytomedicine to treat illness [11]. Medicinal and aromatic plants (MAPs) are generated and provided in a multitude of products, from raw materials to manufactured goods such as pharmaceuticals, herbal remedies, spices, drinks, perfumes, teas, toothpaste, soap, chewing gum, spirits, makeup products, sweets, dietary supplements, varnishes and insecticides, dyes and colorants [20, 22, 33].

From this point of the view, it can be said that *Aloe vera* plant which will be mentioned in the next chapter, is one of the most benefical and most commonly used medicinal plant in the world.

2.3. Extraction Methods of Active Components

Not too long ago, an important raising was seen in studies examining the options of using the some of the aromatic plant extracts or vital herbal oils showing antimicrobial property in controlling pathogenic microorganisms [35]. The use of plant extracts have become popular because a substantial proportion of medicinal plant material is utilized to get plant extracts [24, 34].

The initial step in the processing of the medicinal plants is the seperation of effective ingredients by using various methods from simple classic methods to superior extraction technologies [20, 36]. Usually, plant parts including specific substances are processed by means of drying which is the easiest ways of processing, making it

possible to keep crude medicines. However, the most commonly used technique for this aim is extraction [20, 30]. Extraction requires the decomposition of medicinally effective constituents of plant cells from the non-active or unproductive parts by using special solvents in typical extraction processes [20, 36]. The typical techniques of medicinal plant extraction consist of infusion, maceration, digestion, percolation, hot continuous extraction (Soxhlet), decoction, counter-current extraction, aqueous-alcoholic extraction by fermentation, supercritical fluid extraction, microwave-assisted extraction, phytonic extraction (with hydrofluorocarbon solvents), and ultrasound extraction (sonication) [19, 36].

The best solvent for complete extraction is one that is most selective, has the best convenience of extraction and is suitable for the qualities of the sample to be extracted. The price and accessibility to the solvent are also taken into consideration. Alcohol especially ethanol, though widespread, due to its excellent extractive power it is often the least selective, in that it extracts all soluble constituents [12, 20].

2.4. Present Use of Plants as Antimicrobials

Plants are the oldest supply of pharmacologically effective ingredients, and have offered human kind with many medically beneficial compounds from the hundred of years. Today approximately 70 % of the world's population relays on plant made medicines [23].

Research in plant products as alternative options for antimicrobials have increasingly become more common over the past few years, because of the introduction of multiple-drug resistivity in pathogenic bacteria of human and animal as well as unwanted side effects of specific antibiotics and development and distribute of antibiotic resistance [7, 37-39]. The use of natural goods as alternatives or contrasting to traditional treatment has gained attention due to the belief that herbal products may be safe and have been utilized for several years as traditional remedies [11, 34].

Presently, 80% of the people in developing nations trust mostly plantbased medicines for their health-care requirements [18, 24]. Thinking about the return to nature saying

in today's world, Turkey where involves geographic locations having some endemic kinds, is one of the countries with richest plant variety in the Mediterranean and so has a significant place in medicinal and aromatic plants [25, 40]. Today, the assumption is more than 9,000 plant types of which about 3000 are endemic and 1000 are used as medicine and spice in Turkey [27, 41]. On the other hand, the share of Turkey is quite low in global sales predicted to lie around 65 billion US dollars/year, despite the rich plant diversity in the country [25].

2.5. Medicinal Plants in the World

All around the world, it is predicted that more than 70,000 types of medicinal plants are utilized in folk medicine. In India, about 7,500 types, in China 6,000 species to ten thousand, in Africa, more than 5,000 plant types are used for medicinal requirements. In Europe, approximately 2,000 medicinal and aromatic plant types are used commercially. In Germany, the number is not less than 1,500 and in Spain, about 800 medicinal and aromatic plant species are utilized for similar purposes [42]. By looking at these values it can be said that the number of people using conventional medicines is lower in the developed countries [24].

2.6. Market Situation

Medicinal plants are popular as trade products. Their small size makes their transportation easier to countries with a substandard transport system [43]. Also, the use of herbal crude material is much cheaper than to use chemical alternative materials. As a result, there is a huge desire in botanicals creating an enormous trade, on local, regional, national and international level - for daily use and for industrial applications especially for last three decades [24, 42]. This increased awareness in medicinal plant sector is appearing in not only large but a growing sector [20, 44]. Although there are thousands of medicinal plant types throughout the world, reasonably few are traded in the formal export field [33]. In addition, it is difficult to identify the number of MAP that are commercially traded all over the world. The high amount of the plant material is exported from developing countries, while major markets are in the developed countries [21]. For the last two decades, China, USA

and Singapore are the first three exporting countries in this area sequentially while the range of Turkey is 27 [17].

Medicinal plant markets not only supply an overview of a country's medicinal flora, but they also indicate the issues related with health and illness and the significance of traditional medicine among its occupants [43]. In a near future, biomedical goods will maybe be the major application of antimicrobial textiles since they discover applications for avoidance, as surgical lab apparel, or treatment as wound dressings [3].

CHAPTER 3

THE *ALOE VERA* PLANT

3.1. History of the Plant

The *Aloe vera* plant is known as being one of the oldest medicinal plant in the world and it has been utilized in household medicine, both topically and internally, and also therapeutically for thousands of years [39, 45-49]. The various biblical sources are present related with the use of *Aloe vera* plant in different forms in the literature for over 5000 years, particularly for its wound healing and anti-inflammatory properties [45, 50-53]. Through history, the *Aloe vera* plant has been considered to be a marvelous healing plant [54]. From ancient folklore into the modem time, *Aloe* has carried on to keep forth its many benefits that have been documented by many ancient cultures, containing Egypt, Persia, Greece, India, Arabian, Mexico, Japan, China, the West Indies, Hebrews, Algerians, Moroccans, Tunisians and Africa [47, 54-57]. In each age, on each continent, in each culture, *Aloe vera* has attracted the interest of the most innovative thoughts [58].

Medical utilization of this semi-tropical plant having a long and illustrious background dates back to 2200 B.C. It was firstly recorded in the Sumerian clay tablet that has been found in a Mesopotamian city, Nippur [45, 46, 50, 51, 59]. It has been talked about throughout recorded history and has a high ranking as a multi-purpose natural plant from Cleopatra's beauty regimes, to Alexander's medical therapy to heal his injured soldiers [54, 59, 61-64, 66]. One of the earliest and most outstanding medical report about its use in 1500 B.C in early Egypt, is belong to the famous German Egyptologist and researcher George Ebers [45, 50, 55, 60, 65]. According to this story, after conquering Persia in 333 B.C., Aristotle, adviser of Alexander the Great, told him to get the Socotra island in the Indian Ocean in order to provide *Aloe vera* to cure the injuries of his soldiers [46, 50, 55, 66, 67]. Another saying is that Nefertiti and Cleopatra, the queens of Egyptian, used it as part of their regular beauty regime [51, 68].

3.2. General Information about *Aloe Vera* Plant

Aloe vera have been utilized for hundred of years for its numerous qualities and scientific researches about *Aloe vera* have drawn more interest over the last ten years because of its medicinal, pharmaceutical, health, beauty, skin care and food properties etc. [56, 69-72].

Number of the plant species grown around the world is predicted from 200 to over 500 [39, 47, 50, 54, 59, 73-80]. Among them, only five species, *Aloe arborescens*, *Aloe ferox, Aloe perryi*, *Aloe saponaria*, and *Aloe barbadensis Miller* are basically used for medicinal requirements [50, 59, 77]. The most popular one and the most effective specie of them is *Aloe barbadensis Miller* having widespread use and outstanding healing powers [50, 54, 78, 81]. The name of *Aloe vera* in botany is *Aloe barbadensis* but in some sources it is mentioned as *Aloe vera Linne* [47, 56, 82]. Its name is derived from the Arabic word "*Alloeh*" meaning "polishing bitter material," while '*vera*' refers 'true'in Latin [54, 56]. *Aloe*, native to Africa, is also known as "lily of the desert", the "plant of immortality" and the "medicine plant" [3, 45, 64, 66, 80, 83, 84].

It belongs to the tree lily (*Liliaceae* or *Asphodelaceae)* family having many of other members such as asparagus, leeks, garlic, onion, and turnip [45, 46, 50, 54, 56, 65].
Aloe vera is a sub-tropical or tropical plant with thick, succulent, tapered, green spiny leaves with notched sides having sharp ends. It grows from a short centric stem close to the ground level in the rosette structure [54, 73, 78, 79, 85]. Because the holding of water capacity of leaves is high, it has ability to survive for 7 years or more without water while the majority of other plants can not. When it needs water for survival and growth, it uses the dew gathered on the surface of its leaves [45, 86]. The plant produces flowers in cylindrical or elongated tubular form once a year, generally in the spring. [54, 68, 85]. Color of the flowers is changeable according to the species. It may be yellow, orange or bright scarlet red [54, 68, 73].

The *Aloe* plant is grown in warm, dry climate and cannot survive freezing (below -4 C°) temperatures [51, 57, 59]. It needs the soil having a good permeability and moist [51]. It reaches maturity within about four years survive for about 12 years [66, 68].

Each mature plant usually has 12-16 leaves having a lengths starting from 2,5 inches to 3 feet and a base width from three to five inches approximately and has weighing between 2 or 4 pounds. The plants can be collected every 6 to 8 weeks by cutting 3 to 4 leaves of each plant [54, 68]. It is found wild and is grown in countries drawing the boundary with the Mediterranean Sea and in Africa, India, China, and the islands in the Indian Ocean [54, 56, 63, 87].

3.3. Physical Properties

Although it looks like a cactus, *Aloe vera* plant is not a cactus [54, 76, 84]. It is a perennial, stemless or very short stemmed succulent plant with a rubber touch [45, 50, 60, 63, 88]. The *Aloe* plant has large, thick, triangular, fleshy leaves with serrated edges [56, 60, 66]. The leaves that are flat on the upper surface and convex on the under surface grow from the base in the rosette pattern [45, 57]. Generally, yellow flowers occurs on spikes in tubular form once a year and the flowers are not used medicinally [50, 56, 66, 68, 88].

The shell of the *Aloe vera* leaf presents nearly 20–30% by weight of the whole plant leaf, and the pulp which is the important section of the leaf by volume is about 65–80% [47]. The *Aloe vera* cells, apparent at a magnification of nearly 40,000, is surrounded by a cell wall, has a big core and two cell membranes. The mucopolysaccharide produced by the cytoplasm is stored indise the lumen of the cell [89, 90]. When a leaf is cut, three main sections can be seen that are outer shell, middle layer of latex and the inner clear gel [50, 56, 60, 63, 65, 68, 86]. Among these, the inner gel is mainly believed to be the resource of biological effectiveness of the plant [39].

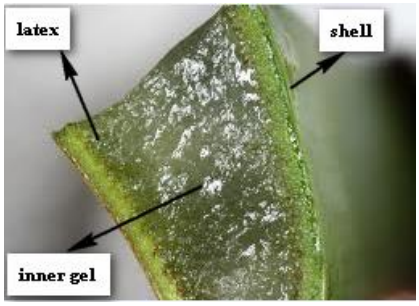


Figure 3.1. Sections of a *Aloe vera* plant leaf

3.3.1. Outer Shell

Outer layer that is the green, external part of the leaves has a tough and leathery surface. Cellulose is part of this rigid structure and the main function of this structure is synthesis of carbohydrates and proteins [56, 68]. The whole leaves are formed by a thick epidermis surrounded with cuticle or shell and inner clear pulp [47, 82]. There are vascular bundles inside the leaf pulp underneath the external thick coating or external cuticle [47, 56, 68, 89]. These vascular bundles comprise of three kinds of tubular constructions: pericyclic plant cells, xylemes, and floemes [68]. Between these three cells, xylem transfers water and nutrients from roots to leaves. The phloem carries starches and other synthesized components to the roots and the big pericyclic plant tissues keep and transfer the *Aloe vera* latex across the edge of the leaf [47, 54].

3.3.2. Middle Layer of Latex

The external leaf, or shell, includes a yellow latex with irritating and laxative qualities [56]. It may acts as a protector of the plant by propelling the animals attempting to consume the plant leaves [46]. This section of the leaves which can also be named as *Aloe* juice or sap has a bitter flavor and annoying acrid odour [60, 68, 91, 92]. The latex or exudate present in the pericyclic cells in the edges of the leaves, is basically utilized for its laxative properties [39, 47, 60, 87]. This incredibly bitter yellow latex flows from the sub epidermal, longitudinal tissues of cut *Aloe* leaves and can be extracted from the vascular bundles at the intersection between the shell and the fillings [34, 52, 78].

3.3.3. Inner Clear Gel

The inner clear gel can be eaisly seen just after the removal of the spiny green shell of the leaves. Beneath the coating of the leave there is a mucilaginous substance including fibres, water and the components to hold the water in the leaf [86, 93]. This transparent mucilaginous, jelly-like substance has a high viscosity. It is colorless, non-bitter part of the plant and acquired from the inner pulp that are large thin-walled parenchyma tissues located at innermost portion of the of the leaf [47,

52, 60, 73, 75, 78, 87, 92, 94]. Unlike latex, the inner leaf, or gel, has a pleasant taste and is relatively reliable [46]. The three structural pieces of the inner leaf pulp are the cell walls, the degenerated organelles and the viscous liquid included inside the cells. They all have different morphology and sugar content [89].

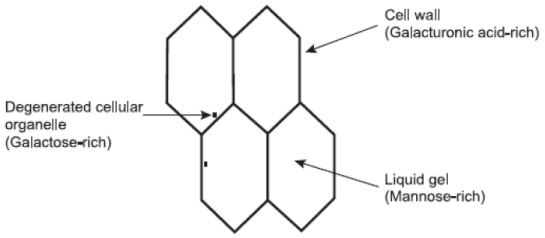


Figure 3.2. *Aloe vera* leaf pulp structure and its components [89]

3.4. Chemical Properties

Although there are numerous components inside the *Aloe vera* gel showing outstanding properties especially in health, amounts of them within the solid part is comparatively low.[45]. This is due to the water content of the plant ranging between 95- 99,5 % [46, 47, 49, 64, 68, 92]. The resudial part is composed of about 70-75 different potentially active components, and over 200 various ingredients. They can be mainly listed as minerals, water-soluble and fat-soluble vitamins, amino acids, enzymes, proteins, steroids, sugars (polysaccharides, monosaccharides) phytosterols, lignin, saponins, salicylic acids, organic acids, lipids, phenolic glycosides, triterpenes [39, 45, 46, 52-54, 56, 59, 60, 64, 68, 73, 76, 80, 87- 89, 91, 93].

It is believed that the most of the medicinal properties of the plant is due to the synergistic effect of these constituents and these properties can be utilized in medical sector [51, 63, 83, 89]. *Aloe vera* is slightly acidic and near to the that of human skin which is about 5,5. Its pH is recorded as 4,4–4,7 or 6 in different sources [45, 47, 68]. If a plant leaf is analyzed in terms of chemical constituents, it can be seen that yellow latex of the leaf includes anthraquinone glycosides such as aloin A and aloin B, and other phenolic substances [52, 56, 60, 68, 85].

In the view of the inner gel, mono- and polysaccharides, glucomannans, amino acids, lipids, sterols and vitamins are present other than a huge amount of water content [56, 75, 92]. Lastly, the outer leaf is composed of cellulose and most of

carbonhydrate (polysaccharides) and glycoprotein (enzymes) components are synthesized in here [54]. Figure 3.3 shows the chemical constituents avaliable in the whole plant leaf.

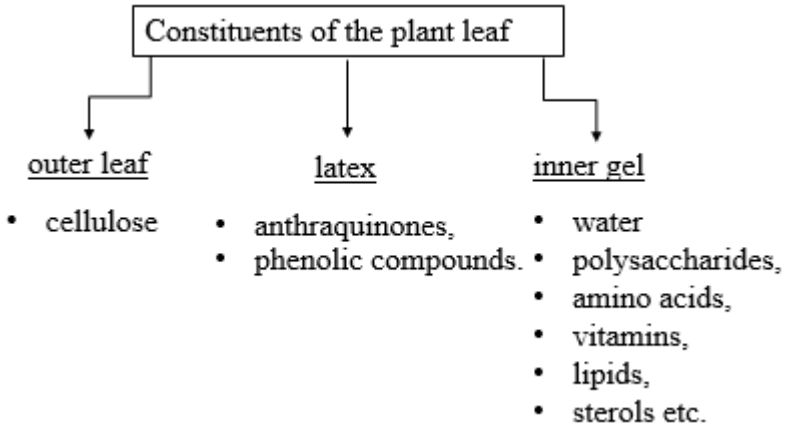


Figure 3.3. Constituents of the plant leaf

The constituents present in the whole leaf of the plant will be examined and explained in details below.

3.4.1. Vitamins

Vitamins are antioxidant supplies and they assist the cell operations and for that reason promote wound healing [56, 59]. There are 12 vitamins in the *Aloe vera* gel [64, 83]. These are vitamin A (beta-carotene), C, E, B1 (thiamine), B2 (riboflavin), B3 (niacin), vitamin B3, B5, B6, choline, folic acid, α-tocopherol [45, 46, 50, 62, 63, 68, 76, 89, 95].

3.4.2. Enzymes

The function of the enzymes is usually the breaking down of the sugars and fats [50, 56, 76]. The enzymes present in the *Aloe vera* plant are mainly aliiase, alkaline phosphatase, amylase, bradykinase, carboxypeptidase, catalase, cellulase, lipase, cyclooxidase, cyclooxygenase, superoxide dismutase and peroxidase [56, 59, 60, 89]. Among these bradykinase is more specific one due to its outstanding properties. It helps to lessen inflamation which is the symptom of the external necrotic event.

Also, it stimulates the defense mechanisms to improve the presence of macrophages in the area occupied by the exterior attack [56, 60, 68].

3.4.3. Minerals

Minerals are necessary for the correct functioning of several enzyme systems in various metabolic pathways and few are antioxidants [56]. They catalyze cellular operations and help the skin's own protective features [59]. There are 20 minerals present in the *Aloe vera* [64, 71]. Some of them are aluminum, barium, boron, chloride, calcium, chromium, copper, iron, selenium, magnesium, manganese, phosphorous, potassium, sodium and zinc [45, 46, 50, 56, 59, 62, 68, 76, 89, 95].

3.4.4. Amino acids

Amino acids are the proteins that are responsible of development of the tissues. Morever, they are known as energy suppliers, stimulators of collagenogenesis and catalysts [50, 59, 76]. The *Aloe vera* plant has 18-20 of amino acids of 22 which people needed [45, 83]. In the nature, there are essential and non-essential amino acids and *Aloe vera* plant includes 7 of 8 essential amino acids [59]. Lysine, histidine, arginine, aspartic acid, threonine, serine, hydroxyproline, proline, glycine, alanine, cystine, valine, methionine, isoleucine, leucine, glutamic acid, phenylalanine, tryptophane, tyrosine are the amino acids available in the plant [59, 68, 89].

3.4.5. Sugars

Sugars are available in the parenchymatous cells or mucilage layer of the leaves [56, 82, 89]. Both mono- and polyshaccarides are present in the plant. Glucose and mannose are the most important types of the monoshaccarides while glucomannans, pectin, cellulose and acemannans are the mostly known polyshaccarides (complex molecules) in the plant [47, 56, 59, 68, 75, 89, 90]. Polysaccharides are long-chain biopolymers which may be developped from different sugar products such as mannose and glucose. In fact, most of the medicinal qualities of *Aloe vera* plant have been mainly linked to the polysaccharides [39, 48, 79, 89, 96]. The antiviral property,

stimulating the immune system, promoting and accelerating wound healing are some of the medicinal properties of these sugars [50, 59, 75, 76]. They have high molecular mass, therefore they can not move through the barrier layer of the epidermis supplying the skin humid. That is why the polysccharides having water retention capacity, are preffered for wound healing and in cosmetic area [59, 68]. In *Aloe vera* gel, these polysaccharides are in the form of acetylated polymannans or acemannans which is the most significant one due to its excellent medicinal qualities [56, 59]. So it can be said that, the higher the acemannan content, the greater bioactivity and useful impact on skin care and wound curing [96].

3.4.6. Anthraquinones

The anthraquinones are present within the yellow sap including a mixture of anthracene components, of the plant leaves [47, 97]. It is located in the lining or under the epidermis of the leaf and the bitter taste is directly related with these phenolic components [56, 68]. Anthraquinones are referred to as laxatives and considered to be a pain-related element [56, 93]. They are soluble in alcohol, acetone, etc. but poorly or insoluble in water. Therefore, an ethanolic extract should be recommended for a considerable antibacterial result [49]. Aloe emodin is the most important one among the 12 anthraquinones present in *Aloe vera* plant [56, 97]. Others can be listed as aloin A and B (barbaloin), aloeitic acid, anthranol, crysophanic acid, an ester of cinnamic acid, and resistannol etc [56, 68, 79, 89]. Anthraquinones contain analgesic, antibacterial, antiviral, antibiotic and antifungal properties. Morever, they are used as purging and cleansing agents. Hovewer, it may take between 6-24 hours to see their results on the human body [50, 56, 68, 76].

3.4.7. Others

3.4.7.1. Fatty Acids

The plant has 4 plant steroids that are cholesterol, campesterol, β-sisosterol and lupeol. These phytosterols show anti-inflammatory and anti-microbial effect. Also they promote cell division. Among them, lupeol also has antiseptic and analgesic properties [54, 56, 59, 89].

3.4.7.2. Hormones

The growth elements auxins and gibberellins contained in the *Aloe vera* plant have an influence on wound curing and antiinflammation. They accelerate and therefore promote wound healing by stimulating cell division [50, 56, 59, 76].

3.4.7.3. Salicylic acid

Salicylic acid is the basic element of aspirin and its salts. In *Aloe vera*, salicylic acid serves as an antiseptic, analgesic, anti-bacterial, and an anti-inflammatory agent [54, 56, 59, 68].

3.4.7.4. Lignin and Saponins

Lignin, an inactive component, helps the other ingredients to penetrate into the skin. Saponins are the soapy materials and represent about 3% of the solid part of the gel. They have detoxification, antiseptic and antimicrobial qualities [50, 56, 68, 76, 89, 93].

3.5. Medicinal Properties

3.5.1. Mechanism of Action

Aloe vera plant has been utilized as a coventional drug for ages [39, 98]. Due to its numerous components it has several prospective biological activities [47]. It is predicted that, the medicinal properties of *Aloe vera* is not due to the multiplicity of these component, it is due to the synergistic effect of these components [51, 63, 89]. *Aloe vera* works as a type of conductor creating songs with an orchestra composed of several biological effective substances [54, 90]. Here, polysaccharides are accepted as conductor while the other components symbolize the orchestra [82]. *Aloe vera* has can be used both topically and orally. In both cases it has various pleasant effects on the human body. However, oral therapy is considered to be more efficient than topical therapy due to acting on the damaged tissue by the way of arteries [54].

3.5.2. Biological Activities of the Plant

The most commonly known and utilized medicinal properties of the plant can be summarized as moisturizing, anti-inflammatory effect, immunomodulatory effect, wound and burn healing action, antiseptic, anti-viral, antipyretic, anti-allergic, anti-biotic, anti-bacterial, antifungal, detoxification, ulcer, anti-tumor, breaking down dead cells such as acne, reducing high cholesterol, skin restoration, sleeping disorders, kidney illnesses, analgesic effect, anti-arthritic activity, restoration of radiation damage, anti-neoplastic activities, activation of hematopoiesis and anti-oxidant effects, anti-rheumatoid, anti-cancer, a relaxing effect on nervous system, gastroprotective qualities, and phototoxicity [26, 34, 45-47, 50, 51, 54, 59, 61, 62, 66, 68, 69, 71-73, 79, 80, 84, 89, 90, 96, 98-101].

Some of these medicinal properties of the plant will be described below.

3.5.2.1. Effect on Skin

Some components of *Aloe vera* gel such as amino acids and polysaccharides show skin moisturizing, softening and nourishing effect [59, 80]. Because the polysaccharides have water holding ability, they assist forming a barrier in order to prevent moisture loss from the skin. Furthermore, amino acids make softer hardened skin tissues and zinc functions as an constringent to tighten pores [56, 70, 86]. *Aloe vera* gel can move through nearly 4 times faster than water and therefore moisturizes and penetrates to the 7 layers deep of the body cells including three epidermal layers of the skin [45, 54, 70, 102]. Just after applying the gel on skin surface a tightening effect will be felt and skin will become less wrinkled as you repeat application [56, 95]. It fastens skin repair or calming and renewal of new skin cells by promoting fibroblast creation and increasing collagen [80, 86, 96, 103]. *Aloe* also help to heal some dermatological issues like dermatitis, eczema, acne, allergy, sunburns, radiation burns or damages, irritation and so on. Although, people prefer to use aloe vera topically in general, it can also boost skin health when applied internally [56, 54, 67, 73, 80, 92].

3.5.2.2. Wound Healing Properties

Actually, this property is a direct consequence of the moisturizing effect of the gel. We know that, healing qualities of *Aloe* have been used more than thousand of years [54, 104]. *Aloe vera* gel is incredibly powerful in supporting wound healing and pain relief [61, 73, 80]. Water holding capability of the gel maintains the wound moist and this reduces the wound healing time to one third of typical period when *Aloe vera* is preffered rather than other medicines. [45, 63, 89, 92].

Varios mechanisms have been predicted for the wound treatment ability of *Aloe vera* gel [89]. At this point of the view, polysaccharides are ascribed for wound-healing activity due to their restoration capacity of destroyed tissues. Another components responsible for wound healing are the hormones present in the gel such as gibberellins, auxin and mannose phosphate. Hormones help the tissues in the wound area to connect each other. So, they promote the healing by increasing protein synthesis together with cell duplication. [54, 63, 68, 56, 90, 102]. One more theory is related with the phytosterols such as lupeol and b-sitosterol in *Aloe vera*. They are considered to accelerate angiogenesis which is a significant process for the effective treatment of wounds [59, 81]. They play an important role in reduction of wound diameter and also result in better vascularity and healthier granulation tissue [92, 105]. Besides these components, minerals, vitamin C, E and amino acids available in the gel are also eefective in wound curing [72, 95].

3.5.2.3. Anti-inflammatory effect

Inflammation occurs after destroying stimulus of a living cell or it is such a protection of the body against a painful event [54]. It is well known that topical application of *Aloe vera* gel is lessen inflammation [80, 89]. This is attributed to lowering of oxidation of the arachidonic acid pathway via cyclooxygenase leading to a reduction in prostaglandin synthesis [56, 59, 60, 70]. There are various components in the plant *Aloe vera* having anti-inflammatory qualities. For example, acemannan which is the basic sugar in the gel, has been predicted to boots anti-inflammatory action [103, 104]. The anti-inflammatory action of *Aloe* is also associated with its three plant steroids: lupeol, beta-sistosterol, and campesterol [54, 68, 90]. Another

substances showing anti-inflammatory activity are amino acids like tryptophane and phenylaianine [54, 90]. This effect may be due to the salicylates. Because they prevent the activation of bradykinin accelerating inflammation and inhibit histamine production [69, 70, 73]. In addition, gibberellin may be responsible for anti-inflammatory effect [93]. And finally, the anthraquinones particularly Aloe emodin in the yellow sap are possible anti-inflammatory agents by destruction of fibroblasts [79, 90, 93].

3.5.2.4. Immunomodulatory Effect

Aloe vera has been proven to develop immune mechanism by initiating the particular tissues of the system called as macrophages [60, 89, 95, 102]. According to the most of the documents, polysaccharides particularly acemannan in the gel shows potent immunomodulatory activity supplying a general immune system support [47, 54, 67, 68, 80]. This strong effect of acemannan on the immune system is due to trace minerals which are rhodium and iridium. They initiate and accelerate the macrophages (white blood cells), monocytes, antibodies and T-cells and they also increase the size of the thymus gland by 40 % generated by T-cells [70, 73, 102, 104].

3.5.2.5. Antibacterial, Antifungal, Antiviral and Antibiotic Effects

Aloe vera is good antibacterial, antiviral and antifungal agent when applied both orally and topically [73, 80]. There are various components present in the gel influencing antibacterial propety. Among them, aloe emodin (purified from barbaloin) and aloesin which are the main anthraquinones in the gel have been asociated with an excellent antibacterial quality against scientifically isolated bacterial pathogens. Aloe emodin works as an antibacterial and antiviral agent against and it inactivtes several viruses such as herpes simplex virus type I and type II, varicella zoster and influenza, pseudorabies virus by preventing nucleic acid biosynthesis while barbaloin and aloetic acid function as remarkable antibiotic agents. [39, 47, 56, 57, 68, 70, 72, 79, 84, 89].

Morever, saponins and other antiseptic agents in the gel such as lupeol which is a natural salicylic acid, urea nitrogen, cinnamic acid, phenol, and sulfur also show anti-

microbial property [54, 68, 98]. Another compounds that have a direct effect on antibacterial activity of the plant are polysaccharides (particularly acemannan) working by accelerating phagocytic leukocytes [98]. Here are the bacterias including Gram negative and Gram positive that are affected from the *Aloe vera* gel constituents. They are Escherichia coli, Klebsiella pneumoniae, Pseudomonas aeruginosa, Mycobacterium tuberculosis, Citrobacter, Salmonella species, Staphylococcus aureus, Streptococcus agalactiae, Streptococcus faecalis, Serratia marcescens, Shigella flexneri and Streptococcus progenes. Candida albicans, Trichophyton species are some of fungi influenced by *Aloe vera* plant [70, 73, 89, 98, 99].

3.5.2.6. Anticancer - Antitumor Activity

The anti-cancer property of *Aloe vera* is mainly attributed with polysaccharides and lectins. Polysaccharides supply this property by accelerating immune system [62, 73, 89, 99]. As you produce more nitric oxide (to a point), the possibility of having cancer will probably lesser. This increasing of nitric acid is achieved by acemannan which is one of the most important polysaccharides in the plant [80]. The organic germanium which is an excellent mineral in the plant supports for de-activating of cancer cells by delivering signals to the DNA of cancer cells to inhibit dublicating [95]. In some papers, anticancer effect is said to have a connection with vitamin B12 [68]. Polysaccharides and lectin-like substances also have been considered to show cytotoxic activity against tumor and abnormal tissues by reducing tumor problem, tumor shrinkage, tumor necrosis and extended survival rates. Additionally, aloe-emodin which is the main anthraquinone is the capable of to directly destroying tumor cells. [47, 60, 62, 68, 89]. To sum up, the gel supports the accelerating of the defense mechanism of the body while damaging cancer and tumors cells [80].

3.5.2.7. Laxative Effect

Anthraquinones present in the latex part of the plant is considered to have laxative property. Normally, it is due to the increase in enteric water content by means of active Na^+ transportation or water release caused by a prostaglandin-based system [56, 60, 92].

3.6. Side Effects

No significant adverse effects belonging to *Aloe vera* have been documented but existing toxicity and side effects are reversible, very well tolerated, reasonably uncommon and not usually serious [57, 76, 99]. Redness, burning, biting feelings and almost never generalized dermatitis are maybe observed in vulnerable people when it is applied topically. In fact, existing allergic reactions are mainly the result of anthraquinones, such as aloin and barbaloin. However, in oral use it may lead to stomach crampings, diarrhea, reddish urine, hepatitis, worsening of costiveness [56, 84].

3.7. Uses of *Aloe Vera*

Aloe Vera is one of the most functional plant all over the world and its possibility of usage both externally and internally allows this plant to be included in almost every area in the human life. Some of the products used in our daily lives can be listed as skin moisturizers, face and hand lotions, cleansers, soaps, sunproofing lotions, shampoos and hair tonics, shaving products, shower products, makeup and aroma products, and baby lotions and wipes. Beside these, topical preparations of *Aloe vera* also include frostbite treatment, radiation burns, dermatitis, sunburns, ulcers, psoriasis, wounds healing, acne and skin diseases etc. [45, 47, 54, 57, 60, 73, 89].

3.8. Application of *Aloe vera* in Textile Fabrics

The antibacterial and antifungal properties of *Aloe vera* enable it to be used in medical textiles including bandages, wound dressing, sutures, bioactive textiles, etc. [3, 83]. In addition, the benefits of *Aloe vera* has been met with the garment owing to the innovative developments in the textile area. Such that, these garments can prevent aging of the skin, regenerates skin tissue, and protects the skin from microbial diseases. This type of fabrics are generally preffered in production of inner garments because they are close to the skin [64]. Microencapsulation technology which will be mentioned in chapter 4 is one of the novel method utilized for this purpose.

CHAPTER 4

MICROENCAPSULATION

Textile finishing lies to a very large extent. So, textile materials are continiously searching for new and innovative application areas to be able to find out new markets and new ideas for textile industry [106, 107].

From this point of the view, microencapsulation is a very good choise of creating functional products in various sectors [2]. Actually, this method have been utilized for years for different purposes such as carbonless copying paper, liquid crystals, adhesives, makeup products, insec repellents, pesticides, colorants, vitamins, antimicrobial agents, phase-change materials, medical apparatus, drugs, pharmaceutics and so many more products concerning food and textile industry. Applications of microencapsulation in textiles include medical applications, linings, carpets and tapestry, blankets, ribbons, scarves, handkerchiefs, curtains, clothes, furnishings etc. [16, 107-109].

In the last decade, microencapsulation has become very popular due to its rapidly growing technology and increased feasibility in textiles by the application of new components like fragrances, dyes, antimicrobials, and PCMs (phase change materials) [1, 14]. In addition, it is more benefical compared to traditional techniques by means of of economy, energy saving, being ecofriendly and controlled release of ingredients [2, 15]. It also enhance solubility, dispersibility and flowability together with allowing mixing of discordant components [8, 110].

4.1. Definition

Microencapsulation is kind of process in which microscopic amount of particles or droplets are surrounded or coated with a continuous film of a polymeric substance in

order to give small capsules many useful properties. In other words, a microcapsule is a diminutive vessel encircled with a homogeneous shell [2, 14, 15, 106, 109-112].

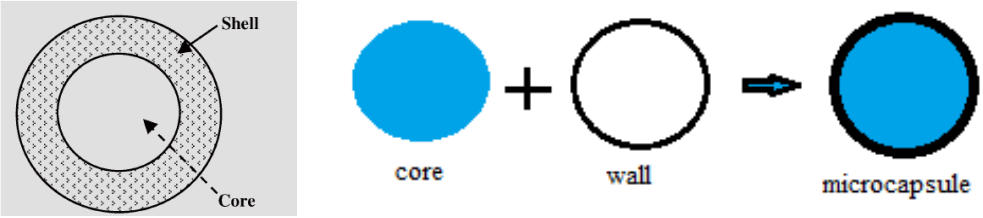


Figure 4.1. Structure and formation of a microcapsule [110]

Generally, capsules consist of two different layers. The substance inside the capsule is named as internal phase, core material, payloads or filler while the outer layer of the capsule shell is called as coating, wall or membrane. Dimensions of microcapsules range from a few micrometers to a few millimeters. The factors influencing microencapsulation can be listed as rate of solvent removal, polymer concentration, solubility of polymer in solvent, and solubility of organic solvent in water etc.

Aim of the shell is to provide a means of packaging, separating and storing core materials by guarding or keeping the core against external damages, vaporization, and contamination. Actually, this technique does not suggest a complete isolation of the core. It only supply a controlled release of the core material thus enhance the durability. There are numerous ways of releasing core substance from a microcapsule. It may be due to the mechanical breaking of the capsule coating, dissolution or melting of the wall, and diffusion from the wall, abrasion, deformation, and rubbing, biodegradation, and pH etc.

Various substances can be encapsulated in solid, liquid, and/or gaseous phase. They may be active agents, catalysts, vitamins, drugs, bioactive materials, proteins, deodorants and phase change materials (PCMs) while polysaccharides, celluloses, gums (arabic gum, sodium alginate), lipids (waxes), proteins (gelatin, albumin) and synthetic polymers are the basic wall materials that are used frequently. Microcapsules can be applied to textile materials by impregnation, spraying, exhaustion etc. [1, 2, 8, 14-16, 106, 109-111, 113-116].

4.2. Morphology of Microcapsules

The morphology of microcapsules are generally based on core substance and forming process of wall material. Microcapsules may be in the form of regular or irregular design. They are commonly classified as mononuclear, polynuclear, and matrix types.

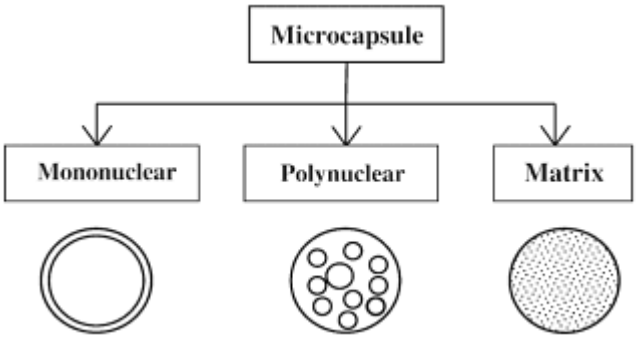


Figure 4.2. Types of microcapsules [8, 110]

Mononuclear microcapsules is composed of a single wall around the capsule. Polynuclear capsules have several cores surrounded with the wall and finally, the matrix type microcapsule has the active substance incorporated within into the coating material. Beside these main structures, mononuclear microcapsules can also have multiple walls, or they may be in the form of microcapsule groups [8, 16, 110, 115].

4.3. Types of Microencapsulation Process

There are various ways of producing microcapsules. It consists of mainly chemical, physicochemical and mechanical methods. Chemical methods include interfacial polymerization, and in-situ polymerization. Physico-chemical methods are solvent evaporation, coacervation (phase separation), encapsulation by supercritical fluid, phase inversion and hot melt. Physico-mechanical methods can be listed as pan coating, spray-drying, spinning disk, vibrational nozzle, air-suspension coating and co-extrusion [16, 109-111, 114].

In this study coacervation method has been used. Therefore it will be explained in details below.

4.3.1. Coacervation Method

One of the most frequently used and most common ways of encapsulation of active materials is the coacervation method. It is a physicochemical process and also called as phase separation. Two kinds of coacervation techniques are utilized in microencapsulation. They are named as simple and complex coacervation. In coacervation method, pH, ionic strength, temperature, molecular weight, and concentration plays an important role. In both method of coacervation, the completely dissolved polymer is become dissolvated gradually by changing temperature, by electrolytes action or by addition of a water miscible nonsolvent. In simple cocervation, only one polymer is used while in complex coacervation two oppositely charged polymers are utilized in combination.

In this study, both simple and complex coacervation methods have been performed to form microcapsules. There are basically three steps of making microcapsules with simple coacervation method. The first step is the preparation of emulsion or dispersion with core material. The next is the encapsulation of the core and final step is making durable of encapsulated substance. These steps differ for complex coacervation. It starts with preparation of three immiscible phases and continues with deposition of the shell materail. The last step is rigidization of the wall. [8, 16, 109-111, 114].

4.4. Microfluidic Processing

Microfluidization is a kind of system or technology processing fluids at microscopic scale via the channels having dimensions ranging between 10-100 micrometres with a low degree of size distribution [117-119]. It has been used particularly for size reduction, dispersion, and particle deagglomeration [120].

In this process, the processing fluid is accelerated by pumping it with high velocity through an interaction chamber via the small channels under high pressures (up to 40,000 psi or 2750 bar). At this point, the speed of the fluid can reach to about 400-500 m/s. This results a uniform high shear stress subjected to the fluid in the chamber causing a high reduction in particle size. [120-122]. Microfluidic devices provide

repeatability of the process. The number of repeats and the value of pressure are changable according to the material and/or user [123, 124]

In this study, an air-driven microfluidizer of model M-110 L was used as shown in Figure 4.3. It consists of a pneumatic pump, a filter, and an interaction chamber.

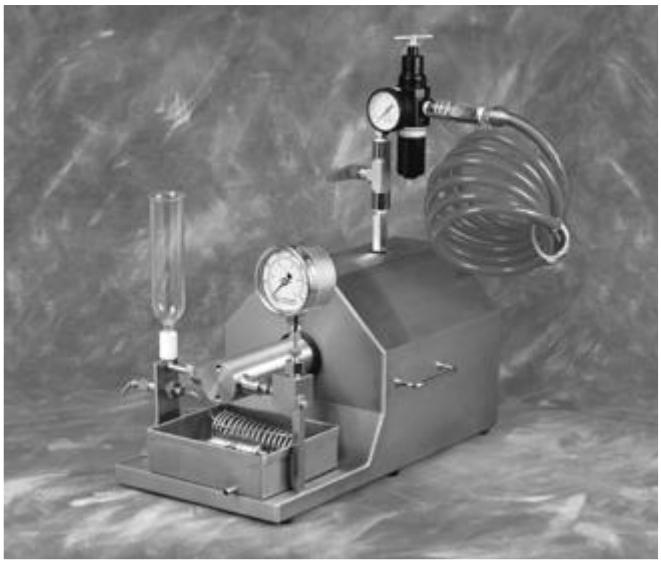


Figure 4.3. Microfluidizer (model M-110L) [122]

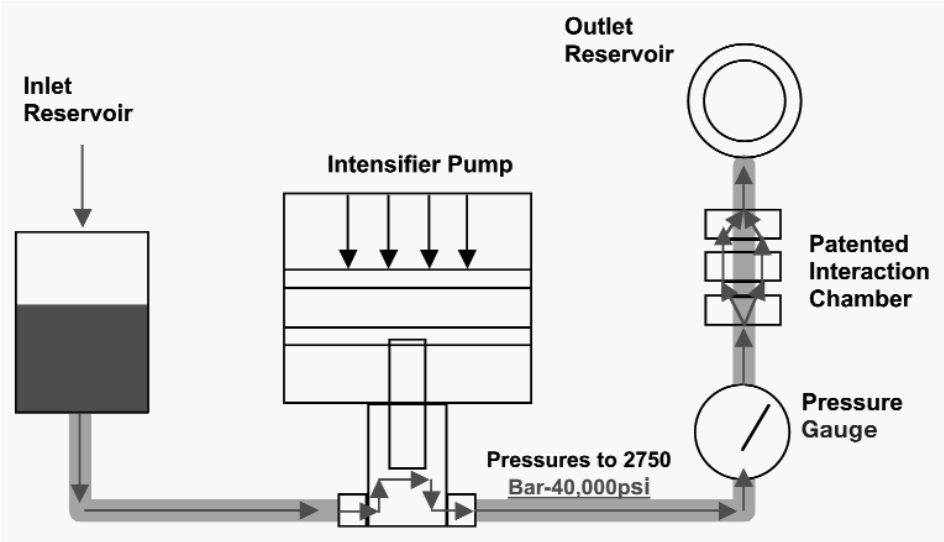
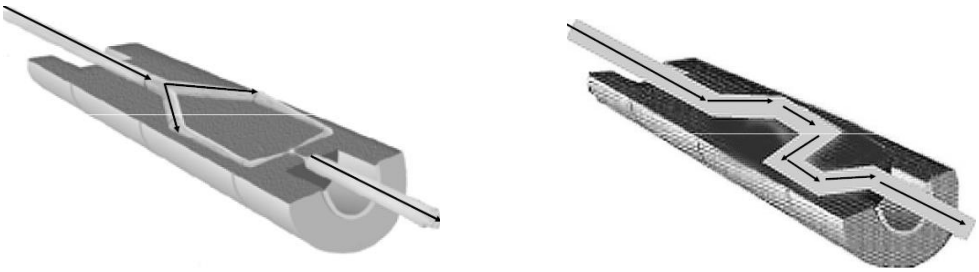


Figure 4.4. Shematic representation of microfluidic system [121, 125]

The interaction chamber has various types but mainly Y-type and Z-type chambers are used. In Y-type chamber, the flow of the product is seperated into 2 channels that merges together while in Z-type chambers, product flow follows a zigzag path

through the interaction chamber [121]. Z-type was equipped with the microfluidic device utilized in our study.



(a) (b)

Figure 4.5. a) Y-type and b) Z-type interaction chamber [125]

In literature, there are numerous examples of studies in which microflidization has been utilized. Jafari and his friends have tried to produce a nano-emulsion including fish oil and d-limonene through microfluidization [123]. Another study is about the cancer treatment via the cell-based drug delivery by using cell-containing hydrogel particles. The microfuidic approaches were examined in order to synthesize these hydrogel particles [118].

4.5. Advantages of Microencapsulation

Microencapsulation process provides various benefits for our lives. The most significant advantages are:

- Controlled and targeted release of active ingredients,
- Allowing mixing of incompatible materials,
- Protection against heat, UV, oxidation, acids, and bases,
- Prolonged shelf life due to inhibiting degradative reactions like oxidation, and dehydration,
- Masking capability of odours and/or taste,
- Enhancing solubility, dispersibility and flowability,
- Handling liquids as solids [110, 115, 119].

4.6. Previous Studies about Microencapsulation

A study related with microencapsulation process was performed by Chinta and his friends in 2013. They have made microencapsulation using neem oil, clove oil, Aloe vera powder, and Benzalkonium Chloride (BKC) as active substances. They have then applied these microcapsule solutions to woven cotton and P/C blend fabric, and nonwoven polypropylene fabric in order to give antimicrobial property and compare their physical and performance properties [15].

Another study is about imparting antimicrobial property to the cotton fabric by coacervation method using extracts of neem and Mexican daisy as core material and acacia as wall material. Extracts were also applied directly to the fabric in order to make a comparison in terms of durability after washing cycles. It has been proven that the extracts in microencapsulated form have a good durability against microbes even after 15 washing cycles [1].

One more study about microencapsulation was performed by Sathianarayanan and his friends. Extracts gained from tulsi leaf and pomegranate were applied to cotton fabric by several methods including microencapsulation, resin crosslinking, direct application, and their combinations in order to give antibacterial property to the fabric. The samples show good antibacterial property until 15 washing cycles except the samples which have been directly treated with the extracts [2].

Devi and his friends carried out a complex coacervation of olive oil with gelatin A and sodium alginate in order to get maximum coacervate yield. The maximum yield was obtained at pH= 3,5-3,8 and at 3,5:1 ratios between gelatin A and sodium alginate. Glutereldehyde was used as crosslinking agent. The aim is to observe release behaviour of core material under different conditions [126].

The microencapsulation process was utilized to eliminate some difficulties related with propolis extract which has antioxidant and antimicrobial properties while it is alcohol soluble and has strong flavour. For this purpose a complex coacervation process was performed by using isolated soy protein and pectin in wall material [127].

Another utilization of microencapsulation includes application and improvement the oxidative stability of fish oil as core material. Simple coacervation technique was used in this study followed by spray drying. Malt dextrin was added into the hydroxypropyl methylcellulose (HPMC) and fish oil solution. The optimum HPMC concentration, dextrose equivalent value of malt dextrin, and percentage of fish oil in microcapsules were found not to be more than 5%, 20, and 40% respectively [128].

In a study about microencapsulation, soy glycinin was used as wall material in order to encapsulate hexadecane which is a dispersed oil phase. The aim is to observe capability of an oil to be encapsulated by protein. The optimum conditions (pH and temperature) were decided for this purpose to support wall formation around the core [129].

Using lemongrass oil having antimicrobial activity as core material is another microencapsulation study by simple coacervation technique. In this study Poly(vinyl alcohol) was preferred as wall material while glutaraldehyde was used as crosslinking agent. The effects of process parameters on microcapsules and antimicrobial properties of the oil were measured [130].

Similarly, Zanthoxylum limonella oil was tried to encapsulate by coacervation method. Gelatin was used as wall forming material and glutaraldehyde was crosslinking agent. The effect of process parameters like concentration of gelatin, oil-loading, and amount of crosslinking on release rate of microcapsules were evaluated [131].

A study belonging to Türkoğlu and his friends was performed by using complex coacervation method. They aimed to produce microcapsules including α-Tocopherol and apply them to cotton fabric by padding method. The final product was examined in terms of its antioxidant propety. [132]

CHAPTER 5
MATERIALS AND METHODS

5.1. Fabric Samples

100% cotton woven gabardine and denim fabrics (3/1 Z, 373 g/m2, 30 x 22 ends x picks) were used in this study. The fabrics were supplied in ready-to-finishing conditions by Gap Güneydoğu Tekstil A.Ş. / Malatya.

5.2. *Aloe Vera* Gel

Fresh clean whole *Aloe barbadensis Miller* leaves were supplied by Batı Akdeniz Agricultural Research Institute (BATEM) / Antalya.

Figure 5.1. Top and side views of *Aloe vera* plant

5.2.1. Discarding/Gaining of the Gel from the Leaves

Gaining of the gel from the plant leaves start with the disinfection of the leaves with 1% of sodium hypochlorite solution and then rinsing. After the outer green rind was discarded, the gel including both latex and inner colorless gel was poured out into separate containers and thoroughly homogenized with a hand blender. Because both have a similar antibacterial effectiveness which has been proved with antibacterial assessment test, in the next steps of the study both latex and inner gel will be used together. One single leaf was found to give gel of approximately 60% of its weight.

Figure 5.2. Discarding of *Aloe vera* leaf gel

5.2.2. Extraction of Aloe vera Gel

Rotavapour machine is one of the most commonly used instrument for extraction. The machine has a flask containing both solvent and extraction material. The flask was taken and replaced in a rotary vaporization machine full with water at 25 ºC and then started to rotate at 175 rpm, at constant pressure and under a vacuum value increased gradually from 50 to 90 mbar.

The purpose was to provide a gentle removal of the solvent from samples by evaporation in order to get pure extract left in the collector flask. *Aloe vera* gel was observed not to be very suitable for this extraction method because a color change has occurred during extraction process. Therefore, liyophilization technique has been tried as an alternative.

Figure 5.3. Rotovapour machine

5.2.3. Liyophilization of *Aloe vera* Gel

Liyophilization is a kind process that allow the water inherent in a sample to be evaporated without heating. Evaporation is due to a vacuum application at high pressure and at low temperature. *Aloe vera* gel samples were replaced in the machine and the process takes place at -50 ºC with a vacuum value of 98mT for three days. Remaining material was in the powder form.

Figure 5.4. Lyophization machine and *Aloe vera* gel before and after process

5.3. Chemical Materials

The chemical materials used throughout this study can be listed as;

- Arabic Gum (CAS Number: 9000-01-5)
- Sodium Sulfate (Na_2SO_4) (CAS Number: 7757-82-6)
- Formaldehyde (CAS Number: 50-00-0)
- Glycerin (CAS Number: 56-81-5)
- Sodium hydroxide (NaOH) (CAS Number: 1370-73-2)
- Butanetetracarboxylic acid (BTCA) (CAS Number: 1703-58-8)
- Sodium hypophosphite (SHP) (CAS Number: 7681-53-0)
- Sodium dodecyl sulfate (SDS) (CAS Number: 151-21-3)
- Sodium thiosulfate (STS) (CAS Number: 7772-98-7)
- Sweet almond oil (CAS Number: 8007-69-0)
- Sodium perborate trihydrate (CAS Number: 10486-00-7)
- ECE phosphate (reference detergent)

5.4. Microencapsulation and Application

Microencapsulation is one of the novel techniques of textile finishing and includes covering the tiny droplets of liquids or solid particles with continuous polymeric film to form so-called microcapsules. The fabrics treated with those microcapsules are reported to give increased durability. In this study microencapsulation process was performed with both simple and complex coacervation techniques.

5.4.1. Microencapsulation with Simple Coacervation

A standard microencapsulation process with simple coacervation technique and its application starts with the usage of *Aloe vera* gel as core material and gum acacia as wall material. 35 grams of acacia powder was allowed to swell in 350 mL of hot distilled water and stirred at 1000 rpm for 30 minutes maintaining the temperature between 55 ºC and 60 ºC. 50 ml of core material was slowly added and stirring was continued for 25 minutes with magnetic stirrer and for 5 minutes with ultrasonic stirrer. 6 mL of 20% sodium sulfate was added within 5 min intervals. Finally, 30 mL of formaldehyde was added and the solution was stirred for 30 min and freezed in the refrigerator to develop microcapsules. The microcapsules were later observed under optical microscope. The samples were immersed in the microcapsule solution, padded through pneumatic padding mangle at 3 psi pressure and 2 m/s roller speed and then dried at 100°C for 5 minutes.

5.4.1.1. Dilution of Microcapsule Solution with Water

In this method, a standard microcapsule solution was diluted with distilled water at different ratios (20, 40, 50, and 100g/L) and the samples were treated with them for comparison in terms of antibacterial effectiveness and textile performances. Because there is no a significant difference between whole gel (latex and inner gel) and inner gel of *Aloe vera* plant leaves for antibacterial effectiveness, whole gel was preferred for microencapsulation for the following steps of the study.

5.4.1.2. Dilution of *Aloe Vera* Gel with Water

The microencapsulation and application techniques were repeated as in section 5.4.1 when crude *Aloe vera* gel was mixed with distilled water at 1:1, 1:50 and 1:100 weight ratios and used as core material in order to compare antibacterial effectiveness with crude gel. Because optimum results were observed with 1:100 ratio, it was determined to be used as core material throughout the study without exceptions.

5.4.1.3. Microencapsulation of *Aloe Vera* Gel in Powder Form using Different Solvents

An additional work was also performed after liyophilization of the gel. Solutions of the gel which is in powder form were prepared with distilled water, ethanol, methanol and acetone. Then, microencapsulation process was performed as in the section 5.4.1 using these solutions as core materials. Antibacterial activity of these microcapsule solutions and the fabric samples treated with these microcapsule solutions were assessed.

5.4.1.4. Change in Core/Wall Ratio and Addition of Glycerin

Another microencapsulation process was performed when the ratio between core and wall material were 1:1 and 1:5 in which wall material includes only arabic gum. In the preparation of 10% arabic gum solution glycerin was also used at 100% and 1:1 weight ratio with distiled water. As it was stated before, 1:100 diluted *Aloe vera* gel was used as core material. Four different microcapsule solutions were obtained with these variations. They will be mentioned as AA, BB, CC, and DD in the following parts of the thesis. The same recipes were named as AA_m, BB_m, CC_m, and DD_m when they are produced with microfluidic device. Table 5.1 shows the content of the microcapsule solutions produced with two methods. The core and wall materials and the ratio between core and wall material were also given in Table 5.1.

Table 5.1. The microcapsule solutions produced with simple coacervation and microfluidic device.

Method	Sample code	Core	Wall	Core:wall
Simple coacervation	AA	1:100 AVG:water	Arabic gum in water	1:1
	BB	1:100 AVG:water	Arabic gum in water	1:5
	CC	1:100 AVG:water	Arabic gum in 1:1 water:glycerin	1:1
	DD	1:100 AVG:water	Arabic gum in 1:1 water:glycerin	1:5
Microfluidic	AAm	1:100 AVG:water	Arabic gum in water	1:1
	BBm	1:100 AVG:water	Arabic gum in water	1:5
	CCm	1:100 AVG:water	Arabic gum in 1:1 water:glycerin	1:1
	DDm	1:100 AVG:water	Arabic gum in 1:1 water:glycerin	1:5

5.4.1.5. Addition of Sodium Sulfate and Ethanol Gradually

In this stage of the study, a gradual addition of sodium sulfate and ethanol (96%) into the microcapsule solution was performed. After addition of 50 ml of core material (1:10 diluted *Aloe vera* gel) into 50 ml of wall material (10% Arabic gum solution) at 45-50 °C within 30 min, 10 ml of 20% sodium sulfate and 10 ml of 96% ethanol were added drop by drop within 2 hours. Then, the temperature was reduced to 10-15 °C during stirring at maximum speed. Together with the dropwise addition of 37 % formaldehyde into the solution, it was heated again to the beginning temperature. It was allowed to be stirred at maximum speed for 4-6 hours and then cooled in the refrigerator in order to observe microcapsules under optical microscope. Purpose of this process is to develop more homogeneous microcapsules.

5.4.1.6. Using Crosslinking Agent

5.4.1.6.1. Recipe 1

In order to improve washing durability of the fabrics treated with *Aloe vera* microcapsules prepared according to *AA, BB, CC,* and *DD* methods given in Table 5.1, BTCA (Butanetetracarboxylicacid) and SHP (Sodiumhypophosphite) were used as crosslinking agent and catalyzer respectively. The process tooks place just before

the application of microcapsule solutions to the fabric samples. The amount of BTCA was calculated as 5% of sample weight. After preparing BTCA and SHP solution at 1:1 ratio, they were simply mixed with microcapsule solutions and then applied to the samples. The samples were only dried at 85 °C for 3 min for wet condenzation and also cured at 120 °C for 8 min for dry condenzation.

Another study was performed by changing curing temperature and amount of crosslinking agent. At this time, *BB* type microcapsule solution was prepared only. BTCA and SHP were mixed at 1:1 weight ratio to prepare solution and the amount of BTCA was equal to the 5% of total bath volume. After addition of BTCA-SHP solution to the microcapsule solution, it was applied to the fabric samples. The samples were dried at 85 °C for 5 min and packaged for wet condenzation while they were also subjected to curing at 150 °C for 2 min for dry condenzation.Same processes were applied for 100 gr/L of the microcapsule solutions with distiled water.

5.4.1.6.2. Recipe 2

In this case, *BB* type microcapsule solution which has been described in Table 5.1. was prepared by using BTCA within the wall material together with Arabic gum at 1:1 weight ratio. SHP was added just before the application of microcapsule solution to the fabric sample. The amount of SHP was equal to the 5% of bath volume. For wet condenzation, the samples were packaged for 24 hr after drying at 85 °C for 5 min. A condenzation process also tooks place for dry condenzation at 150 °C for 2 min without packaging. Same processes were applied for 100 gr/L of the microcapsule solution with distiled water.

5.4.1.6.3. Recipe 3

Another way of improvement of washing durability is the treatment of the samples with crosslinking agents before the application of microcapsule solution. 40 and 80 gr/L solutions of BTCA and SHP (at 1:1 weight ratio) were prepared and applied to the samples like in the section 5.4.1. Then, they were dried at 80°C for 5 min and condenzated at 160 °C for 3 min. The microcapsule solution which has been

prepared according to a desired recipe was applied to the same samples with the same application, drying and curing conditions.

5.4.1.6.4. Recipe 4

40 gr/L of 100 ml BTCA and SHP mixture at 1:1 weight ratio was applied to the fabric samples and the samples were dried at 80 °C for 5 min. The microcapsule solutions prepared according to the recipes 9 and 19 were heated to 50 °C and the samples treated with crosslinking agents were put in the microcapsule solutions and stirred with magnetic stirrer for 1 hour. The samples were cured 150 °C for 3 min.

5.4.1.7. Effect of pH on Microcapsules

pH is one of the most important parameter for microencapsulation process because it has ability to make phase seperation which is the most indispensable and essential stage of the process. Arabic gum was used as wall material throughout the study, as it is known. It is well-known that arabic gum shows the highest swelling degree at pH between 9.2-9.3 and its solubility is directly proportional with temperature raising.

5.4.1.7.1. Recipe 5

After addition of 40 ml of core material (1:100 diluted *Aloe vera* with distilled water) to the 200 ml of 10% arabic gum solution gradually on the magnetic stirrer at 40-45°C, pH was measured and found to be approximately 6. In order to increase pH to about 9.3 NaOH solution was prepared at pH=13-14 and it was added drop by drop until the desired value is achieved. Then, 20 ml of formaldehyde was added and the solution was stirred for 20 min and cooled.

5.4.1.7.2. Recipe 6

100 ml of 10% arabic gum solution was prepared at 40-45°C. 50 ml of 1:100 diluted *Aloe vera* with distilled water was added gradually to the wall material on the magnetic stirrer. After 15 min of stirring pH was increased to about 9 with NaOH. 10 ml of 96% ethanol and 10 ml of 20% sodium sulphate solution were added drop by

drop at the same temperature. Finally, 5 ml of 37 % formaldehyde was added and freezed.

5.4.1.7.3. Recipe 7

100 ml of 10% arabic gum solution was prepared at 50-55°C. 50 ml of 1:100 diluted *Aloe vera* with distilled water was added and stirred for 25 min with magnetic stirrer and for 5 min with ultrasonic stirrer. 35 ml of 40% sodium sulfate solution was added to the mixture and stirred for 10 min with magnetic stirrer. pH of the solution was raised to 9.2 with NaOH. Then, 10 ml of 96% ethanol was added gradually and the solution was stirred with ultrasonic stirrer for 5 min. After addition of 5 ml of formaldehyde, the solution was allowed for stirring for about 4 hours.

5.4.1.7.4. Recipe 8

100 ml of 10% arabic gum solution was prepared at 40-45°C with magnetic stirrer. 20 ml of 100% *Aloe vera* gel was added to the solution and stirred for 30 min at maximum speed. pH of the solution was raised to 9.2 with NaOH. 10 ml of 96% ethanol and 10 ml of 20% sodium sulfate solution were added drop by drop within 2 hours. Finally, 5 ml of 37% formaldehyde solution was added gradually and stirred for 20 min. The solution was allowed for cooling in refrigerator.

5.4.1.7.5. Recipe 9

50 ml of 10% arabic gum solution was prepared at 40-45°C and stirred for 1 hour at maximum speed with magnetic stirrer. 50 ml of 1:10 diluted *Aloe vera* with distilled water was added and stirred for 30 min under the same conditions. 10 ml of 96% ethanol and 10 ml of 20% sodium sulfate solution were added drop by drop within 2 hours. The microcapsule solution was cooled to 10-15 °C before adding 10 ml of 37% formaldehyde within 30 min. Then, temperature was raised to 40-45 °C again. The microcapsule solution was kept at that temperature and stirred at maximum speed for 4-6 hours. Then, it was allowed for cooling in the refrigerator.

5.4.1.8. Microencapsulation with Ultrasonic Stirrer

100 ml of 10% arabic gum solution was prepared by using ultrasonic stirrer for 20 min at frequency of 5. 20 ml of 100% *Aloe vera* gel was added to the wall material and stirred for 20 min under the same conditions.10 ml of 20% sodium sulfate solution, 10 ml of 96% ethanol, and 5ml of formaldehyde were added to the solution with 5 min intervals and the solution was cooled in refrigerator.

5.4.1.9. Using Sweet Almond Oil: *Aloe vera* Gel as Core

As it is known, almost 99% of *Aloe vera* gel is composed of water. Similarly, the wall material used in the study is prepared with water. This is like a microencapsulation of a water-in-water emulsion. Because, it is more common and easier to encapsulate water-in-oil and/or oil-in-water emulsions, *Aloe vera* gel was mixed with sweet almond oil with the help of an emulsifier.

5.4.1.9.1. Recipe 10

100 ml of 10% arabic gum solution was prepared at 40-45°C and stirred for 1 hour at maximum speed with magnetic stirrer. 20 ml of *Aloe vera* gel:sweet almond oil at 1:1 ratio (with the addition of 0,1 ml SDS) was added and stirred for 30 min under the same conditions. 10 ml of 96% ethanol and 10 ml of 20% sodium sulfate solution were added drop by drop within 2 hours. The microcapsule solution was cooled to 10°C and before adding formaldehyde it was heated again to 25°C. 10 ml of 37% formaldehyde was added within 30 min. during temperature raising. When microcapsule solution was reached to beginning temperature, it was kept at that temperature and stirred for 2 hours with magnetic stirrer at maximum speed. Then, it was allowed for cooling in the refrigerator.

5.4.1.9.2. Recipe 11

100 ml of 10% arabic gum solution was prepared and stirred at maximum speed for 1 hour on magnetic stirrer at 40-45 °C. 20 ml of *Aloe vera* gel:sweet almond oil at 1:1 ratio (with the addition of 0,1 ml SDS) was added gradually. In order to increase pH

to about 9.3, NaOH solution was prepared at pH=13-14 and it was added drop by drop until the desired value is achieved. During pH increasing, the temperature of microcapsule solution was reduced to 10 ºC while stirring at 400 rpm. It was then heated again and when the temperature was reached to 25 ºC, 10 ml of 37% formaldehyde was added and the solution was continued to being heated to the beginning temperature. After 2 hours of stirring it was allowed for cooling.

5.4.1.9.3. Recipe 12

200 ml of 10% arabic gum solution was prepared at 55-60 ºC by stirring for 1 hour. 40 ml of *Aloe vera* gel:sweet almond oil at 1:1 ratio (with the addition of 0,3 ml SDS) was added gradually at 400 rpm. After each addition of core material 0,2 ml of SDS was added to reduce oil seen on the solution surface. Totally, 2 ml of SDS was used. The solution was stirred for 30 min at maximum speed. During addition of 20 ml of sodium sulfate (20%) solution within 5 min, stirring speed was reduced again to 400 rpm and then it was stirred at maximum speed for 2 hours. pH of the solution was increased to 9 at 400 rpm and then it was stirred for 30 min at maximum speed during temperature reduction to 5 ºC. At the same temperature 20 ml of 37% formaldehyde was added slowly and stirred for 15-20 min. Finally, the solution was heated to beginning temperature and stirred for 2 hours at that temperature. Then, it was cooled in the refrigerator.

5.4.1.9.4. Recipe 13

50 ml of 10% arabic gum solution was prepared at 50 ºC by stirring for 1 hour. 10 ml of *Aloe vera* gel:sweet almond oil at 1:1 ratio (with the addition of 2,5 ml of an emulsifier having TD value of 6) was added gradually and stirred at maximum speed for 30 min. 10 ml of sodium sulfate (20%) solution was added drop by drop within 1 hour under same conditions. pH of the solution was increased to 9,24 with NaOH solution at pH=13 while temperature was being reduced to room temperature. Before addition of 10 ml of 37% formaldehyde slowly, temperature was reduced to 5 ºC. the solution was stirred for 5 min at that temperature and then heated to 40-45 ºC. It was continued to being stirred for 5-6 hours at constant temperature and cooled in the refrigerator.

5.4.2. Microencapsulation with Microfludic Device

Microencapsulation process was also tried to perform by using microfluidic device purchased within the scope of the project supported by the Ministry of Industry. It works according to the microfluidic principle and so it performs exceptionally well with particle size reduction, cellular interruption, emulsions, etc. In the study, M-110L model of microfluidizer processor was used. Each pass through the machine only takes a few minutes for approximately 200 ml of solution. This sounds very advantageous when thinking stirrings with magnetic stirrers for hours. Another advantage is that smaller and more homogeneous microcapsules can be obtained. In the optimization of machine parameters like pressure, number of passes, temperature, ph etc. the microcapsule formation, wall thickness and uniformity in microcapsule sizes were taken into account. After optimization, microencapsulation processes achieved with microfludic device were explained below.

5.4.2.1. Recipe 14

The recipes belonging to the *AA, BB, CC,* and *DD* type microencapsule solutions were used for microencapsulation with microfludic device. The solution of wall material was allowed for stirring with magnetic stirrer for short time intervals after adding core material, sodium sulfate, and formaldehyde respectively. Then, the final mixture was passed through the machine 2 times under 30000 psi pressure. A cooling process was needed during the passage of the solution in order to prevent temperature raising due to high pressure. The microcapsule solutions obtained with this recipes were coded as AA_m, BB_m, CC_m, and DD_m. The same processes were also repeated without formaldehyde addition.

5.4.2.2. Recipe 15

10% arabic gum solution was prepared at 40-45 ºC and passed through the machine once under 30000 psi pressure. After addition of the core material (1:100 diluted *Aloe vera* with distilled water) at 1:5 core:wall ratio, the solution was passed through the machine once again under the same pressure. pH of the solution was increased with NaOH to about 9,08 and the temperature was reduced to 15 ºC. The solution

was passed through the machine 2 times under te same pressure. It was examined under optical microscope before and after freezing.

5.4.2.3. Recipe 16

The core and wall material, the ratio between them and the process pressure are the same with those of previous experiment. After preparation of the wall material at the same temperature, core material was added and pH of the solution was increased to 9,1. Then, the temperature of the solution was reduced to 10 ºC and formaldehyde (7 ml) addition took place. The solution was stirred for a few minutes with magnetic stirrer and passed through the machine once.

5.4.2.4. Recipe 17

100 ml of 10% arabic gum solution was prepared with magnetic stirrer at 40-45°C. 10 ml of 20% sodium sulfate and 10 ml of 96% ethanol was added drop by drop within 2 hours.20 ml of 100% *Aloe vera* gel was added slowly and the solution was stirred for 30 min. After temperature of the solution was reduced to 10°C, it was passed through the machine 2 times at 30000 psi. 5 ml of 37% formaldehyde solution was added drop by drop within 1 hour on the magnetic stirrer and heated again to the beginning temperature. Then, it was allowed for cooling.

5.4.2.5. Recipe 18

100 ml of 10% arabic gum solution was prepared at 40-45°C with magnetic stirrer at maximum speed. Mixture of 20 ml of *Aloe vera* gel:sweet almond oil at 1:1 ratio (with the addition of 0,1 ml SDS) was used as core material. After addition of core material to the wall material slowly, pH of the solution was increased to 9.3. The solution was passed through the machine 2 times at 30000 psi after 5 ml of 100% formaldehyde was added gradually. A cooling process was applied during the passage of the solution.

5.4.3. Microencapsulation with Complex Coacervation Technique

5.4.3.1. Recipe 19

Beside simple coacervation technique, microencapsulation of *Aloe vera* gel was also tried to perform by complex coacervation method. Gelatin and arabic gum were used as wall materials while *Aloe vera* gel diluted with distiled water to 1:10 ratio was core material. A 20 ml, 10% gelatin solution was prepared with distilled water and stirred at maximum speed with magnetic stirrer at 40-45 °C. 2 gr of arabic gum was added to the solution and stirred for 30 minutes. Stirring speed was reduced to 400 rpm after 30 minutes of addition of core material to the solution at 1:1 core: wall ratio. pH was first reduced to 4 with acetic acid while temperature was reduced to about 10-15 °C and pH was then increased to 9.3 with NaOH. After 30 minutes 10 ml of 37% formaldehyde addition took place at constant temperature and speed. The solution was stirred at maximum speed for 4-6 hours and allowed for cooling.

5.4.3.2. Recipe 20

In this experiment, 50 ml of 10% gelatin solution was prepared at 40-45 °C by magnetic stirrer at maximum speed. A 10 ml of SDS (%100): STS (%0,05) at 10:1 ratio was added drop by drop to gelatin solution. Then, 50 ml of %10 arabic gum solution was added to the same solution. pH of the solution was decreased to 4 at which the gelatin has the lowest solubility. After addition of 50 ml of core material which has been 1:20 diluted *Aloe vera* gel with distilled water, the mixture was stirred for 30 minutes. Stirrer speed was reduced to 600 rpm when 10 ml of %20 sodium sulfate was added drop by drop. The temperature of the solution was then decreased to about 10 °C and pH was increased to 9.2 with NaOH solution at pH=14. Finally, 5 ml of %37 formaldehyde was added to the solution and the solution was cooled in refrigerator after 30 minutes of stirring. 20 and more recipes were tried throughout the study. Some of them are listed in Table 5.2.

Table 5.2. The content of the recipes applied during study

Recipe	Core material	Wall material	Core:wall ratio	Desolvating agent
Recipe 1	Application method of AA, BB, CC, and DD using BTCA and SHP			
Recipe 2	1:100 AVG:water	AG/water/BTCA	1:5	Na_2SO_4
Recipe 3	Application method of a desired recipe using BTCA and SHP with condenzation			
Recipe 4	Application method of Recipes 9 and 19 using BTCA and SHP			
Recipe 5	1:100 AVG:water	AG/water	1:5	NaOH
Recipe 6	1:100 AVG:water	AG/water	1:2	NaOH, Na_2SO_4, ethanol
Recipe 7	1:100 AVG:water	AG/water	1:2	NaOH, Na_2SO_4, ethanol
Recipe 8	100% AVG	AG/water	1:5	NaOH, Na_2SO_4, ethanol
Recipe 9	1:10 AVG:water	AG/water	1:1	Na_2SO_4, ethanol
Recipe 10	1:1 AVG:SAO	AG/water	1:5	Na_2SO_4, ethanol
Recipe 11	1:1 AVG:SAO	AG/water	1:5	NaOH
Recipe 12	1:1 AVG:SAO	AG/water	1:5	Na_2SO_4, NaOH
Recipe 13	1:1 AVG:SAO	AG/water	1:5	Na_2SO_4, NaOH
Recipe 14	AA, BB, CC, and DD types microcapsule solutions from microfluidic device			
Recipe 15	1:100 AVG:water	AG/water	1:5	NaOH
Recipe 16	1:100 AVG:water	AG/water	1:5	NaOH
Recipe 17	100% AVG	AG/water	1:5	Na_2SO_4, ethanol
Recipe 18	1:1 AVG:SAO	AG/water	1:5	NaOH
Recipe 19	1:10 AVG:water	AG/gelatine/water	1:1	Acetic acid, NaOH
Recipe 20	1:20 AVG:water	AG/gelatine/water	1:2	Acetic acid, NaOH, Na_2SO_4

5.5. Textile Performance Tests

Abrasion resistance, breaking strength, spectrophotometric measurement, water vapour permeability test, dry/wet fastness, and UV resistance tests were applied at laboratory conditions to the samples in accordance with the standards of ASTM D 4966, ASTM D 5034, AATCC 173, BS 7209 and TS EN ISO 105-X12 respectively.

5.5.1. Abrasion Resistance

Martindale abrasion tester was used for determining abrasion resistance of the samples. Three circular specimens were cut for each fabric sample with 38 mm in

diameter. Beside mass measurement, fabric thickness was also measured after each 2500 abrasion cycle with Baker Fabric Thickness Gauge. The samples treated with microcapsule solutions containing 100% and 1:100 *Aloe vera* gel diluted with distilled water were also assessed in terms of their antibacterial activity after 500 rubbing cycles.

Figure 5.5. Preparation for abrasion test and Martindale abrasion tester

5.5.2. Breaking Strength

Breaking strength was measured by James H. Heal Titan Universal Strength Tester 2. 3 of warp and 3 of weft direction test specimens were prepared for each fabric sample with dimensions 50x300 mm.

Figure 5.6. Breaking strength tester and final situations of samples

T1 jaws and 3000 N load cell were preferred with a jaw speed of 100 mm/sec. Test results were recorded in terms of maximum force (N), extension (%), and CV %. Here, maximum force is the required force to break the test specimen while extension is the amount of increase in length of the test specimen.

5.5.3. Spectrophotometric Measurement

Test specimens were evaluated in terms of their L, a, b, DE, YIE (yellowness index), WIE (whiteness index) values using Datacolor 650 spectrofometre. Measurements were applied under D65 illuminant at 10^0. The results are average of four measurements and the samples were rotated 90º after each measurement.

5.5.4. Water Vapour Permeability

The water vapour permeability of the test samples was accomplished with SDL ATLAS water vapour permeability tester. Three test specimens and one specimen from reference fabric were cut with the same size of the test dish and sealed over the open mouth of the test dishes containing distilled water with a volume of 46 ml and temperature of 20 ºC ± 2. A supporting sample between the dish and test specimen should also be replaced. Then, each of corresponding cover rings of the dishes was sealed to the dishes with adhesive tape. After the dishes replaced on the turntable which has capability of carrying eight dishes simultaneously, it was started to rotate. Mass measurements were done after 1 and 5 hours from the start.

Water vapour permeability of test specimens were calculated by;

$$WVP = 24M/At \quad \text{(Eq. 1)}$$

where M: difference in mass after 1 and 5 hours
A: constant (0,0054113 m²)
t: 5 hours

Figure 5.7. Water vapor permeability tester

5.5.5. Dry and Wet Rubbing Fastness

Atlas CM-5 crockmeter was used in order to determine dry and wet rubbing fastness of test specimens. 5x14 cm of test specimens were cut and placed on to the corresponding section. A standart test fabric was rubbed ten times with test specimen. In wet rubbing fastness measurement the standart test fabric should be wet with distiled water. Assesment should be made under standard day light at 45º with gray scale.

Figure 5.8. Dry and wet rubbing tester and gray scale

5.5.6. UV Resistance

The samples treated with microcapsule solutions containing 100% and 1:100 *Aloe vera* gel diluted with distilled water were subjected to UV light with Electrotech Lightbox for 8 hours for their antibacterial assessment.

Figure 5.9. UV resistance tester with samples in it

5.5.7. Washing Durability

Denim fabric samples treated with desired microcapsule solutions were subjected to repeated washing cycles. The samples were cut in 5x10 cm dimensions for each tube

and replaced in the tube into which 10 small balls and 150 ml of a standard solution was added. This solution includes 4 gr/L ECE phosphate and 1 gr/L sodium perborate at 40 ^{0}C. Washing was accomplished with Termal washing machine according to A2S experiment conditions of TS EN ISO 105-C06 standard.

Figure 5.10. Washing machine and other apparatus

5.6. Antibacterial Activity Assessment

Bacterial cultures of *S.aureus* and *E. coli* were provided by Microbiology Laboratory of Uludağ University Medical Faculty. The antibacterial assessment of treated samples was evaluated by quantitative test method ASTM E2149-01. In this test, the cultures were incubated in a nutrient broth and diluted with buffer solution at pH 6,5 to a standardized concentration of 1.5-3.0 x 108 CFU/ml. For each sample to be tested, 50 mL of the buffer solution and 1 ml of standardized microbial solution were placed into 3 sterile flasks. One flask contained only bacterial suspension; another contained 1 gr of treated test sample and the last contained 1 gr of untreated test sample. All flasks were diluted with a series of dilutions using buffer solution and bacterial concentration was determined at time '0' by performing standard plate count techniques. The inoculated plates are incubated at 37°C for 24 hours and surviving cells were counted again. All these processes were repeated after all the flasks were shaken in a wrist-action shaker for 24 hours. The antimicrobial activity is expressed in % reduction of the organisms after contact with the test specimen at time '0'compared to the number of bacterial cells surviving after contact with the specimen after 24 hours. The percentage reduction (R) of bacteria was calculated using following formula:

R= 100 (B-A) / B (1) (Eq. 2)

where, A is the number of bacteria recovered from the inoculated treated test specimen swatches in the flask incubated for 24 hours; and B is the number of bacteria recovered from the inoculated treated test specimen swatches at '0' contact time.

5.7. Vacuum-drying of Microcapsule Solution

B type microcapsule solution was prepared and vacuum-dried for 5 hours at 70 °C, under 5x100 mbar vacuum degree at Food Engineering Department Laboratory in Gaziantep University. It was then examined under optical microscope.

5.8. FTIR Analysis

FTIR analysis of *Aloe vera* gel is made at 4 cm $^{-1}$ resolution with Perkin Elmer Frontier FT-IR device at Food Engineering Laboratory in University of Gaziantep.

5.9. Thermal Analysis and Particle Size Analysis

Thermogravimetric (TGA) and enthalpy (DSC) analysis of the microcapsules containing *Aloe vera* gel and the samples treated with these capsules were made at METU Central Laboratory, with Perkin Elmer STA 6000 TGA and Perkin Elmer Diamond Differential Scanning Calorimeter devices according to the TSE ISO EN 11358-1 and ISO EN 11357 - 3 standards. TGA analysis was performed from 30 to 650 ºC at a heating rate of 10 ºC/ min, while DSC analysis was applied in the N_2 gas environment, from 25 to 500 ºC with a heating rate of 10 ºC/min. The particle size analysis of the microcapsule solution was conducted in the wet state again in METU Central Laboratory with Mastersizer 2000 device.

5.10. SEM Images

SEM images of samples treated with microcapsule solution were taken using Zeis / Evo LS10 scanning electron microscope at USKIM in University of Kahramanmaraş Sütçü Imam. Samples were coated with gold before imaging.

5.11. HPLC Analysis

HPLC analysis of *Aloe vera* leaf gel was made in Pharmaceutical Botany Laboratories of Faculty of Pharmacy at Hacettepe University. The analysis was performed by HPLC Shimadzu LC-20AB device. Inertsil ODS-3V (C 18 column, 250 x 4.6 mm, 5 mm particle size) was used as column while Phenomenex (C18 column, 4 mm x 3.0 mm, 5 mm particle size) was used as front column. Aloin and Aloe emodin having analytical properties were used as standard substances. Stock solutions of aloin and aloe emodin were prepared with methanol at a concentration of 1000 µg/mL. All stock solutions were kept at +4 °C. The solutions used for calibration curve is diluted with methanol at 10:00, 1.00 and 0.10 mg / mL concentrations from the stock solution. Mobile phase A: B (distilled water / formic acid: methanol analytical HPLC) was performed as given in Table 5.1. The dedector has been studied for Aloin with 357 nm wavelength value and Aloe emodin with 432 nm wavelength having an injection volume of 5 mL and the flow rate 1.0 ml / min. Sample and detector temperatures were adjusted as 40 ° C, 4 ° C and 40 ° C respectively.

Table 5.3. Mobile phase change according to gradient method

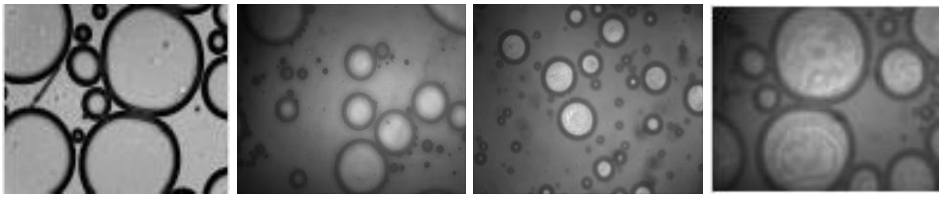
Time (min)	**Mobile phase A (distilled water and 1% formic acid)**	**Mobile phase B (Methanol)**
0.01	75	25
1.00	75	25
10.00	30	70
16.00	20	80
18.00	20	80
20.00	75	25

5.12. Paper Chromatography

In this process the descending-dripping paper chromatography was utilized. AG81 type paper was choosen. Solvent system includes *n*-BuOH-acetic acid-water (4:1:5) and β-naphthylamine solution was used as revelator. Drift time is 72 hours. Four of monosaccharides and/or disaccharides that are fructose, maltose, saccharose, dextrose were used as reference substance.

CHAPTER 6
RESULT AND DISCUSSION

6.1. Optical Microscope Images

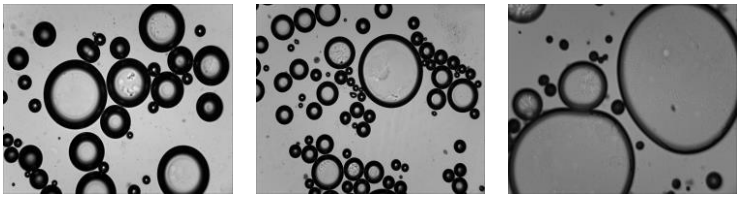


(a) (b) (c) (d)

Figure 6.1. Microcapsules prepared by using inner gel (a – b), intermediate gel (c) and the whole gel (d) as core material

Images showed that *Aloe vera* microcapsules prepared with simple coacervation technique have a wide range of size distribution and there is non-encapsulated fields in the solution. This case can be improved by changing the microencapsulation technique (based on the principle of microfluidic flow).

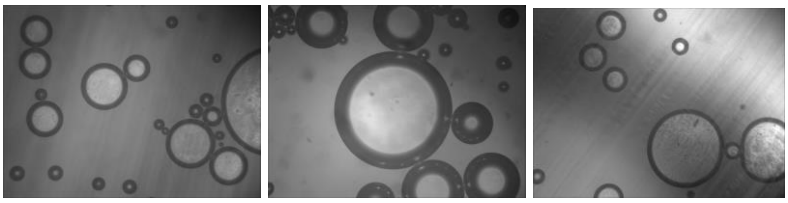
Within the scope of evaluating the antibacterial efficacy of *Aloe vera* gel, 1:1, 1:50 and 1:100 dilutions of the gel with distilled water were used as core material for microencapsulation. Optical microscopic images of the microcapsules prepared with these diluted gel solutions are shown in Figure 6.2.



(a) (b) (c)

Figure 6.2. Microcapsules prepared by using a) 1:1 *Aloe vera* gel / distilled water b)1:50 *Aloe vera* gel / distilled water c) 1:100 *Aloe vera* gel / distilled water

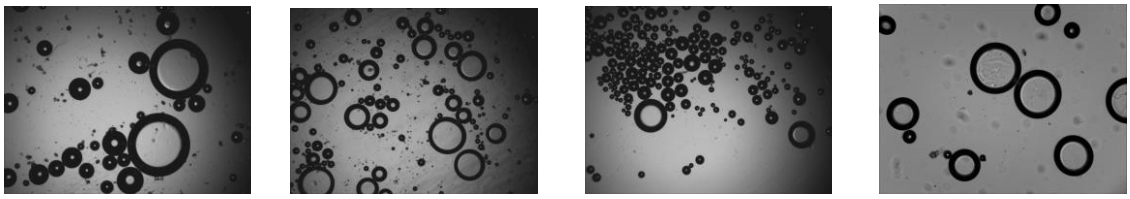
Images of microcapsules prepared by using 1:1, 1:50, and 1 :100 *Aloe vera* gel:distilled water as core material were given in Figure 6.3. According to the Figure 6.3, as a result of the gradual increasing of pH of the microcapsule solution, the microcapsules with similar sizes and thicker walls were produced.



(a) (b) (c)

Figure 6.3. Microcapsules prepared by using a) 1:1 b)1:50 and c) 1:100 diluted *Aloe vera* gel with distilled water

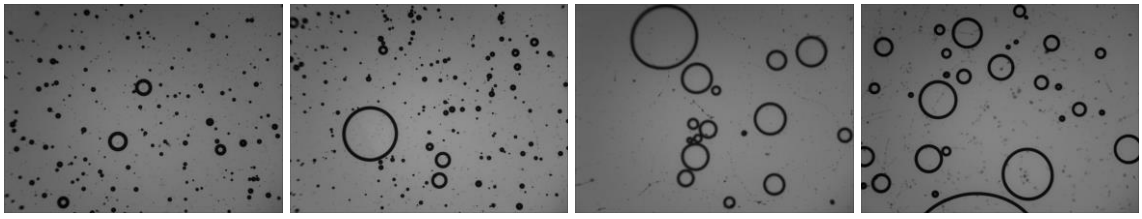
Optical microscope images of the microcapsules prepared by using lyophilized *Aloe vera* gel as internal phase were given in Figure 6.4. Images show that *Aloe vera* gel can be encapsulated within the different polar solvents.



(a) (b) (c) (d)

Figure 6.4. Microcapsules prepared by using lyophilized *Aloe vera* gel dissolved in acetone (a), ethanol (b), methanol (c), and water (d)

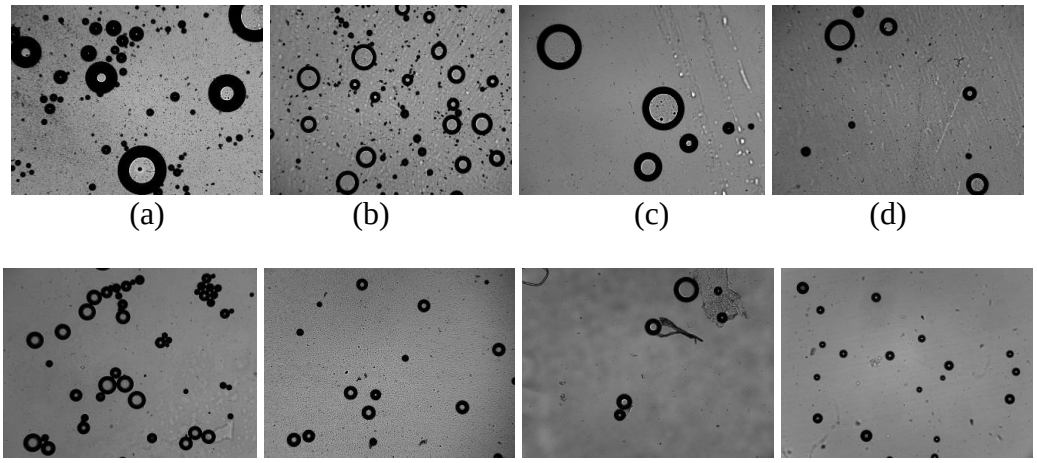
The microcapsule solutions prepared by dissolving arabic gum in glycerin and 1:1 glycerin:water mixture have microcapsules with different diameters and figures were given in Figure 6.5.



(a) (b) (c) (d)

Figure 6.5. Microcapsules prepared by using a) and b) %100 glycerin, c) and d) 1:1 glycerin:water mixture in the preparation of wall material.

The microcapsules produced by simple coacervation method according to the recipes of *AA*, *BB*, *CC*, and *DD* type (a, b, c, d) and with microfluidic device according to the recipes of AA_m, BB_m, CC_m, and DD_m (d, e, f, g) were examined under optical microscope. It can be observed that the microcapsules produced microfluidic device are smaller in sizes and more homogeneous.

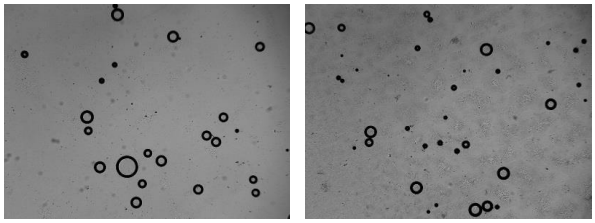


(a) (b) (c) (d)

(e) (f) (g) (h)

Figure 6.6. Microcapsules produced by simple coacervation and with microfluidic device.

Figure 6.7 shows the microcapsules produced according to the recipe 5 given in section 5.4.1.7.1 at pH= 9,29 and 9,85 by simple coacervation method in which the pH increasing process was achieved with NaOH solution about at pH=13. It can be observed that the microcapsules are smaller and more homogeneous in size.

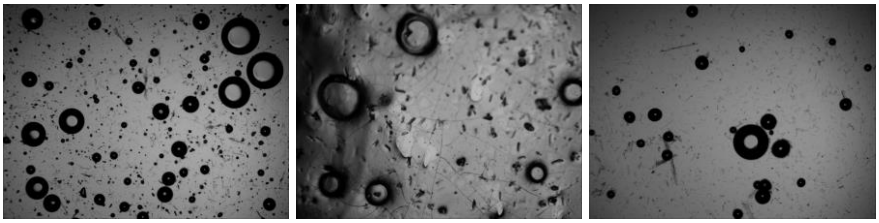


(a) (b)

Figure 6.7. Microcapsules produced at a) pH= 9,29 and b) 9,85

In order to evaluate microcapsule efficiency in the microcapsule solutions the solutions prepared according to the recipes of AA and CC types were simply dried in

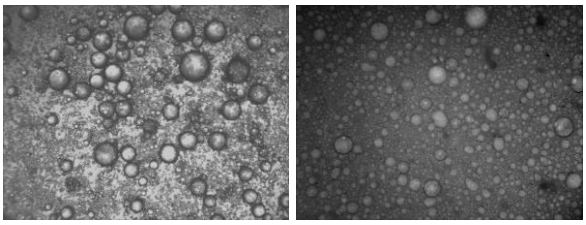
an oven at 80- 90ºC for 8 hours while BB type microcapsule solution was vacuum-dried at 70 ºC for 5 hours in the laboratory of Food Engineering Department.



(a) (b) (c)

Figure 6.8. a) AA, b) BB, and c) CC type microcapsule solutions after drying

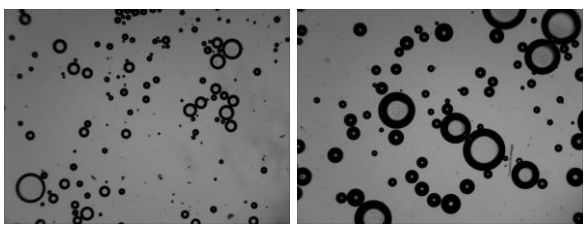
The figures from the microcapsule solutions in which the core material is a mixture of sweet almond oil and *Aloe vera* gel were given below. The solutions were prepared according to the recipes 10 and 11 given in section 5.4.1.9.1 and 5.4.1.9.2 respectively.



(a) (b)

Figure 6.9. Microcapsules prepared according to the a) recipe 10 and b) recipe 11

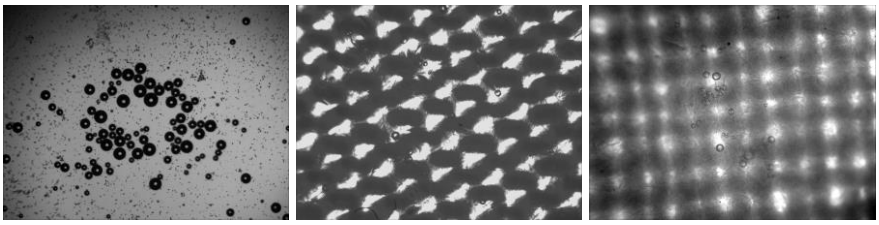
The microcapsules produced according to the recipes 15 and 16 given in sections 5.4.2.2, and 5.4.2.3 respectively, by using microfluidic device were given in Figure 6.10.



(a) (b)

Figure 6.10. Microcapsules prepared according to the a) recipe 15 and b) recipe 16

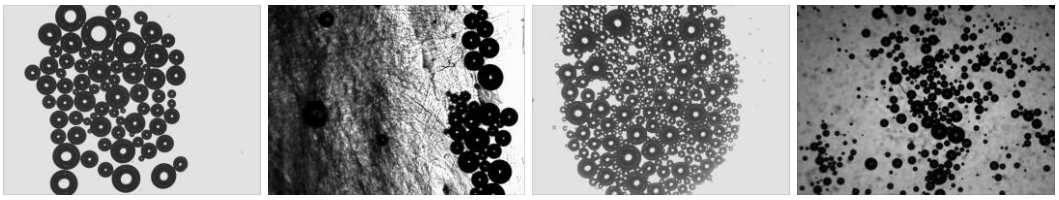
The figures given below is belong to the microcapsules prepared according to the recipe 17 given in section 5.4.2.4. The microcapsules were tried to be observed under optical microscope when a piece of fabric has been attached to lamella on which microcapsule solution was put. The aim is to filtrate microcapsules from non-encapsulated solution. After filtering, the residual microcapsules can be seen on the fabric surface.



(a) (b) (c)

Figure 6.11. Microcapsules when they are put on a) only lamella, b) fabric on lamella, and c) fabric and cotton on lamella.

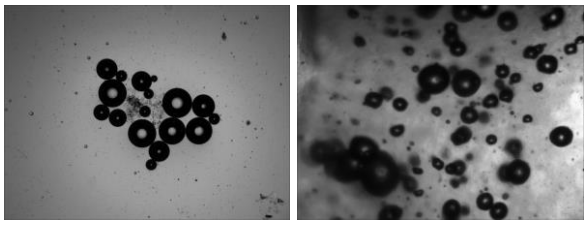
Figure 6.12 shows the microcapsules produced with simple coacervation method according to the recipes given in sections a) 5.4.1.7.2 (Recipe 6), b) 5.4.1.7.3 (Recipe 7), c) 5.4.1.7.4 (Recipe 8) and d) 5.4.1.8.



(a) (b) (c) (d)

Figure 6.12. Images of microcapsules prepared with different recipes

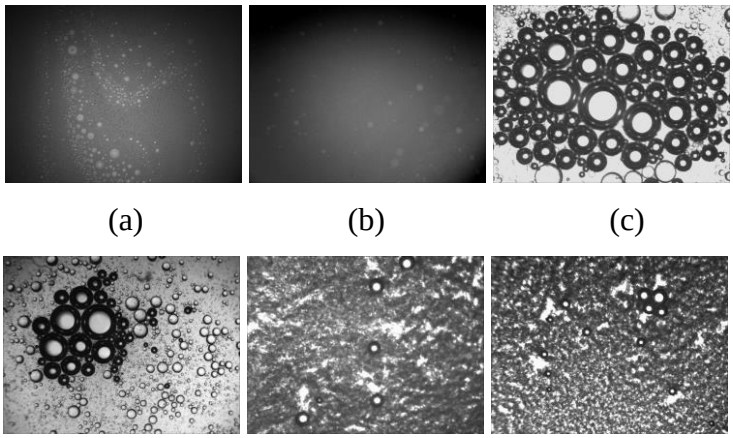
Beside simple coacervation, *Aloe vera* gel was also encapsulated with complex coacervation method by using gelatin in the wall material together with arabic gum. The figures of microcapsules produced with both methods according to the recipes 9 and 19 given in sections 5.4.1.7.5 and 5.4.3.1 respectively, were given below. In complex coacervation a 3-D effect was observed.



(a) (b)

Figure 6.13. Microcapsules produced with a) simple and b) complex coacervation techniques

Images of microcapsules produced with a, b) microfluidic device according to the recipe 18 given in section 5.4.2.5 and with simple coacervation technique according to the recipes given in section c, d) 5.4.1.9.3 (Recipe 12) and e, f) 5.4.1.9.4 (Recipe 13) were given below. The capsules contain sweet almond oil-*Aloe vera* gel mixture as core material.



(a) (b) (c)

(d) (e) (f)

Figure 6.14. Images of microcapsules with different recipes and techniques

6.2. Antibacterial Assessment

Antibacterial effectiveness of test samples are given in Table 6.1 when the samples are treated with 100 % and 40 gr/lt diluted microcapsule solution containing *Aloe vera* gel. In Table 6.2, the results are belong to the samples treated with 100 % microcapsule solution containing *Aloe vera* gel which was diluted with distilled water to 1:1, 1:50, and 1:100 weight ratios.

When we examine the Table 6.1, it can be said that *Aloe vera* has an excellent antibacterial property against both Gram negative and Gram positive bacteria. Table 6.2 shows that *Aloe vera* has a very high antibacterial efficiency even it was diluted. Additionally, *Aloe vera* has still antibacterial effect after 14 days of application.

Table 6.1. Antibacterial effectiveness of the samples treated with *Aloe vera* microcapsules containing A1) latex B1) inner gel C1) whole gel. A2, B2, and C2 represent the samples treated with 40 g/L diluted A1, B1, and C1. D is untreated sample.

Bacterial reduction (%)	Sample						
	A1	A2	B1	B2	C1	C2	D
S. aureus*	(-) 99,884	(-) 99,969	(-) 99,942	(-) 99,976	(-) 99,990	(-) 99,971	(-) 78,64
E. coli**	(-) 99,934	(-) 78,631	(-) 99,971	(-) 97,373	(-) 99,994	(-) 81,57	(-) 25,49

* Inoculum concentration was calculated for each sample as 8.76×10^7 (log 7.94) cfu.

** Inoculum concentration was calculated for each sample as 4.34×10^8 (log 8.64) cfu.

Table 6.2. Antibacterial effectiveness of the samples treated with microcapsule solution containing *Aloe vera* diluted with distilled water at A1) 1:1, A2) 1:50, A3) 1:100 weight ratios.

[a] represents antibacterial effectiveness results after 14 days of application.

Bacterial reduction (%)	Sample		
	A1	A2	A3
*S. aureus**	(-)99.63	(-)99.54	(-)96.50
*E. coli***	(-)100.00	(-)100.00	(-)99.65
Bacterial reduction (%)	A1[a]	A2[a]	A3[a]
*S. aureus**	(-)100.00	(-)100.00	(-)100.00
*E. coli***	(-)100.00	(-)99.87	(-)100.00

* Inoculum concentration was calculated for each sample as 1,02 x 108 (log 8,01) cfu.

** Inoculum concentration was calculated for each sample as 7,91 x 107 (log 7,90)cfu.

In Table 6.3, antibacterial activities of some microcapsules solutions in which the core materials were the solutions of lyophilized *Aloe vera* gel with acetone, ethanol, methanol and water, and the samples treated with these solutions were given. The results show that dissolving of *Aloe vera* gel in different polar solvents has no negative effect on the antibacterial activity. This result means that *Aloe vera* gel can be encapsulated as oil / water emulsion and applied to the fabrics instead of water / water emulsions which have been made so far.

Table 6.3. Antibacterial effectiveness of microcapsule solutions of lyophilized *Aloe vera* gel prepared with A) acetone, B) ethanol, C) methanol D) water where A1, B1, C1, and D1 are the samples treated with these solutions, respectively. E is untreated sample.

Bacterial reduction (%)	Samples								
	A	A1	B	B1	C	C1	D	D1	E
E. coli*	(-) 100,00	(-) 99,66	(-) 100,00	(-) 98,96	(-) 100,00	(-) 97,96	(-) 100,00	(-) 87,26	(+) 15,04

* Bacterial concentration was calculated for each sample as 1,44x 10^8 (log 8,16) cfu.

The resistance of antibacterial activity of denim fabric samples after treatment with 100% and 1:100 diluted *Aloe vera* gel microcapsules against abrasion was carried out through 500 cycles with Nu Martindale device under 12 kPa load. The resistance of antibacterial activity of denim and gabardine fabric samples after treatment with the same microcapsule solutions against UV light was performed by the subjecting of the samples under UV light for 8 hours in Electrotech Lightbox UV cabin. The results of these measurements are given in Table 6.4.

Table 6.4. Antibacterial effectiveness of A1, A2) gabardine fabric, B1, B2) denim fabrics treated with A1, B1) 100%, A2, B2) 1:100 diluted *Aloe vera* gel

microcapsules and subjected to UV light for 8 hours, denim fabrics treated with C1) 100% C2) 1:100 diluted *Aloe vera* gel microcapsules after 500 rubbing cycles, D is the blank sample.

Bacterial reduction (%)	Samples						
	A1	A2	B1	B2	C1	C2	D
*E. coli**	(-) 100,00	(-) 100,00	(-) 100,00	(-) 100,00	(-) 100,00	(-) 100,00	(+) 0,66

* Bacterial concentration was calculated for each sample as $1{,}72 \times 10^8$ (log 8,23) cfu.

The denim fabrics after being treated with microcapsules that contain 100% *Aloe vera* gel as core material and arabic gum as wall material dissolved in glycerin and 1:1 glycerin:water mixture, show high antibacterial activity as shown in Table 6.5 where C is blank sample.

Table 6.5. Antibacterial effectiveness of the fabrics after being treated with microcapsules prepared by dissolving Arabic gum in A) glycerin B) 1:1 glycerin:water mixture as the outer phase.

Bacterial reduction (%)	Sample		
	A	B	C
*E. coli**	(-)100,00	(-)100,00	(+)361,54

[a]Bacterial concentration transferred to each sample weighing 1 gram, is calculated as 1.33×10^5 (log 5.12) * cfu.

Antibacterial effectiveness values against *E. coli* after 10 and 15 washing cycles of denim fabric samples treated with microcapsule solutions prepared at pH= 9,29 and 9,85 according to recipe 5 given in section 5.4.1.7.1 were shown in Table 6.6. The samples were treated with a mixture of BTCA and SHP having 40 and 80 gr/L concentrations before microcapsule solution is applied.

Table 6.6. Antibacterial effectiveness values of denim fabric samples treated with recipe 5 after 10 and 15 washing cycles.

Bacterial reduction (%)	Samples			
	pH=9,29-40	pH=9,29-80	pH=9,85-40	pH=9,85-80
0 washing cycle	(-) 88,65	(-) 94,89	(-) 92,06	(-) 94,89
10 washing cycles	(-) 82,98	(-) 92,62	(-) 89,79	(-) 90,35
15 washing cycles	(-) 77,87	(-) 90,64	(-) 88,09	(-) 88,09

[a]Bacterial concentration transferred to each sample weighing 1 gram, is calculated as 1,50 x 10^5 (log 5,18)* cfu

Antibacterial effectiveness values of denim fabric samples treated with microcapsule solutions coded as AA, BB, CC, and DD against *E. coli* are given in Table 6.7. The results show that there is no change in antibacterial effectiveness of the samples when they are subjected to condensation (at 120 ºC for 8 min.) after drying at 85 ºC for 3 min.

Table 6.7. Antibacterial effectiveness of denim fabrics when they are subjected to condensation or not.

Bacterial reduction (%)	Samples			
	AA	BB	CC	DD
Condensation (+)	(-)100	(-)100	(-)100	(-)100
Condensation (-)	(-)100	(-)100	-	-

[a]Bacterial concentration transferred to each sample weighing 1 gram, is calculated as 1,83 x 10^5 (log 5,26)* cfu

Table 6.8 shows the antibacterial activity of denim fabrics treated with BB type microcapsule solution by using crosslinking agents according to the recipes 1 and 2 given in sections 5.4.1.6.1. and 5.4.1.6.2 respectively. I, II, III, and IV were prepared according to the recipe 1 while V, VI, VII, and VIII were prepared according to the recipe 2. In addition, I, III, V, and VII were subjected to wet condenzation while the others were subjected to dry condenzation.

Table 6.8. Antibacterial effectiveness values of denim fabrics treated with recipe 1 and 2.

Bacterial reduction (%)	Samples							
	I	II	III	IV	V	VI	VII	VIII
E. coli	(-) 86,54	(-) 89,74	(-) 92,95	(-) 97,44	(-) 87,18	(-) 78,85	(-) 90,38	(-) 81,41

[a]Bacterial concentration transferred to each sample weighing 1 gram, is calculated as 1,32 x 10^5 (log 5,12)* cfu

Antibacterial activities of denim fabrics treated with microcapsule solutions coded as AA_m, BB_m, and CC_m against *E. coli* are given below. Same solutions have been prepared without formaldehyde addition. E is untreated fabric.

Table 6.9. Antibacterial effectiveness values of denim fabrics treated with different recipes.

Bacterial reduction (%)	Samples			
	AA_m	BB_m	CC_m	E
Formaldehyde (+)	(-) 100,00	(-) 100,00	(-) 100,00	(+) 92,31
Formaldehyde (-)	(-) 94,87	(-) 90,38	(-) 95,51	

[a]Bacterial concentration transferred to each sample weighing 1 gram, is calculated as 2,04 x 10^3 (log 3,31)* cfu

Antibacterial effectiveness of denim samples was given below when sweet almond oil-*Aloe vera* gel mixture at 1:1 volume ratio has been used as core material. The process was applied according to the recipe 10 given in section 5.4.1.9.1. B is blank sample.

Table 6.10. Antibacterial activities of fabric samples treated with recipe 10.

Bacterial reduction (%)	Sample	
	A	B
*E. coli**	(-)100	(+)112,77

[a]Bacterial concentration transferred to each sample weighing 1 gram, is calculated as 2,40 x 10^5 (log 5,38)* cfu

Antibacterial activities of denim samples treated with microcapsule solutions prepared according to the recipes 12 and 18 given in sections A) 5.4.1.9.3 and B) 5.4.2.5 respectively, are given in Table 6.11 where C is blank sample.

Table 6.11. Antibacterial activities of denim fabric samples treated with recipe 12 and 18.

Bacterial reduction (%)	Sample		
	A	B	C
*E. coli**	(-)100	(-)100	(+)203,03

[a]Bacterial concentration transferred to each sample weighing 1 gram, is calculated as 1,68 x 10^5 (log 5,23)* cfu

6.3. Abrasion Resistance

Abrasion resistance of the samples treated with microcapsule solutions diluted 20gr/L, 50gr/L, and 100 gr/L were evaluated by calculating % change in mass after 2500, 5000, 7500, and 10000 abrasion cycles. Results on Table 6.11 and 6.12 are the average of three measurements in weight belonging to each type of fabric and application. According to these values % mass changes were calculated and given in Figure 6.14. It can be said that there is a reduction in abrasion resistance of denim fabric samples after application but it is not significant when compared to the weight loses above 5 % seen after finishing processes like water repellency and crease resistance.

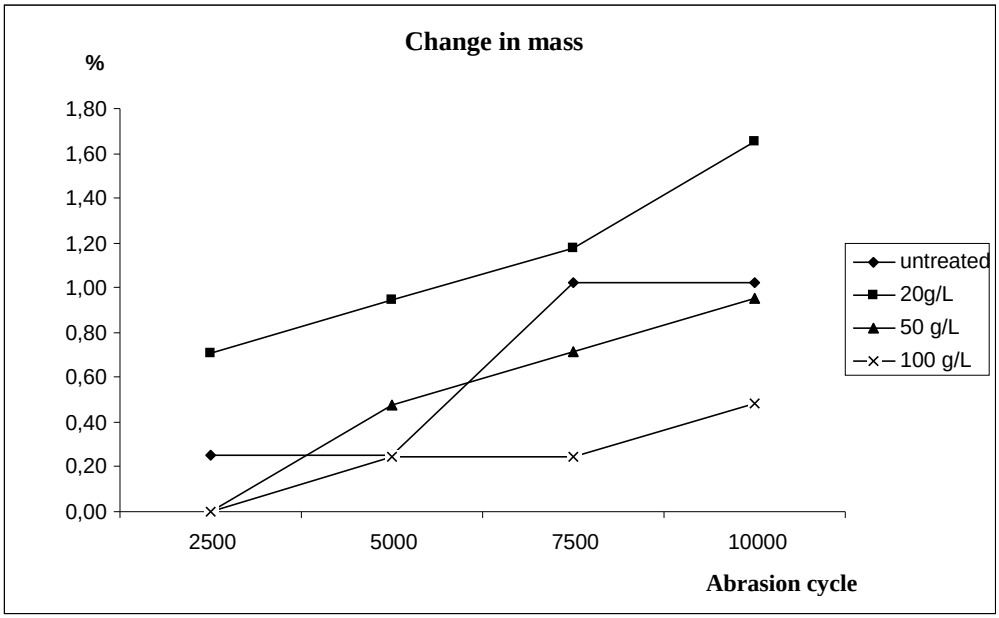
Table 6.12. Mass values of a) gabardine and b) denim fabric samples (gr) after abrasion cycles

Samples	0 cycle	2500 cycles	5000 cycles	7500 cycles	10000 cycles
Untreated	0,392	0,391	0,391	0,388	0,388
20g/L	0,424	0,421	0,42	0,419	0,417
50 g/L	0,42	0,42	0,418	0,417	0,416
100 g/L	0,411	0,411	0,41	0,41	0,409

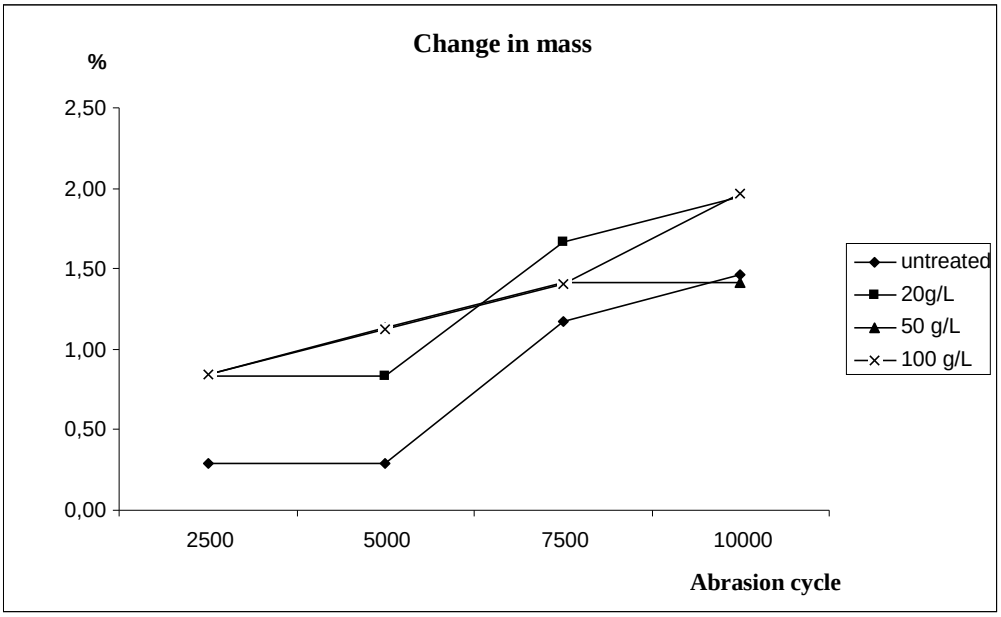
(a)

Samples	0 cycle	2500 cycles	5000 cycles	7500 cycles	10000 cycles
Untreated	0,341	0,34	0,34	0,337	0,336
20g/L	0,359	0,356	0,356	0,353	0,352
50 g/L	0,354	0,351	0,35	0,349	0,349
100 g/L	0,355	0,352	0,351	0,35	0,348

(b)



(a)



(b)

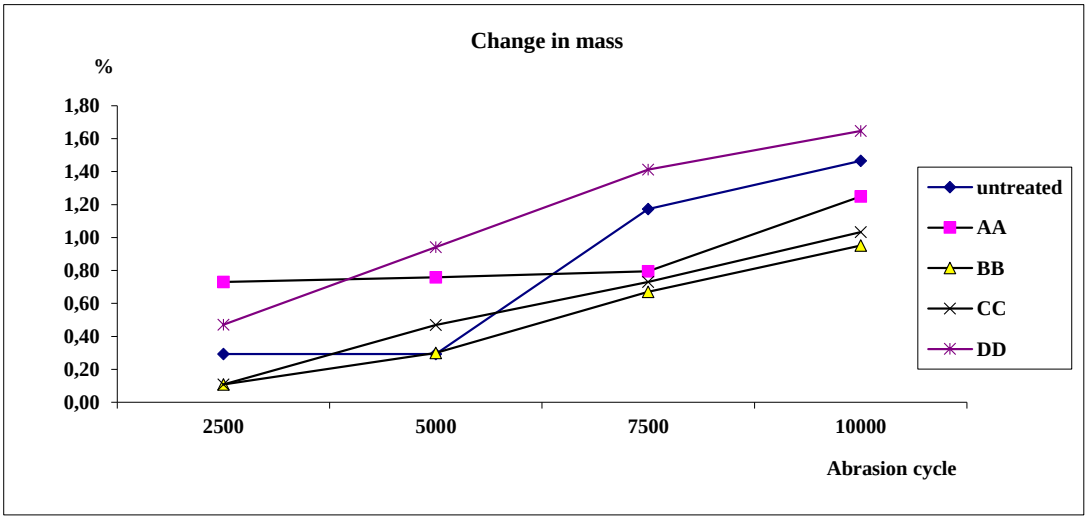
Figure 6.15. % mass change of a) gabardine and b) denim fabric samples after abrasion cycles

The same test procedure was applied for the denim fabric samples treated with microcapsule solutions prepared with simple coacervation method and microfluidic principle according to the recipes of *AA*, *BB*, *CC*, *DD*, and AA_m, BB_m, CC_m, DD_m type microcapsule solutions respectively. The results show that, there is a negligible (< 5%) mass loss after 10000 rubbing cycles for both techniques while there is much more mass loss in the samples treated with microcapsule solutions prepared according to microfluidic principle. The results were given below.

Table 6.13. Mass values of denim fabric samples (gr) after abrasion cycles

Samples	0 cycle	2500 cycles	5000 cycles	7500 cycles	10000 cycles
Untreated	0,341	0,340	0,340	0,337	0,336
AA	0,360	0,358	0,357	0,357	0,356
BB	0,368	0,367	0,366	0,365	0,364
CC	0,397	0,397	0,395	0,394	0,393
DD	0,425	0,423	0,421	0,419	0,418

Samples	0 cycle	2500 cycles	5000 cycles	7500 cycles	10000 cycles
Untreated	0,341	0,340	0,340	0,337	0,336
AA_m	0,360	0,358	0,357	0,357	0,356
BB_m	0,371	0,366	0,365	0,363	0,363
CC_m	0,421	0,407	0,405	0,404	0,403
DD_m	0,455	0,456	0,454	0,453	0,451



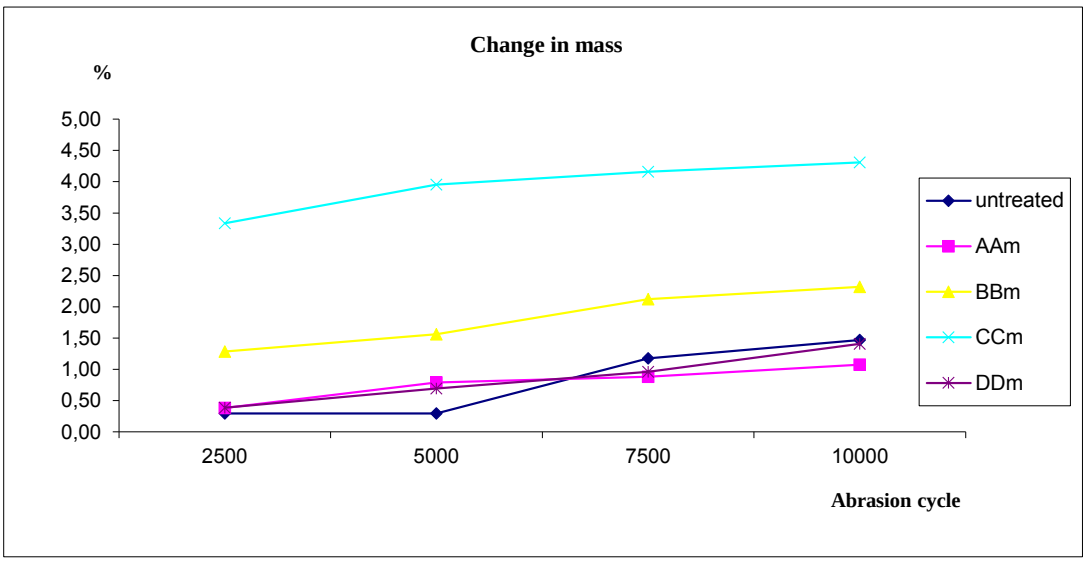
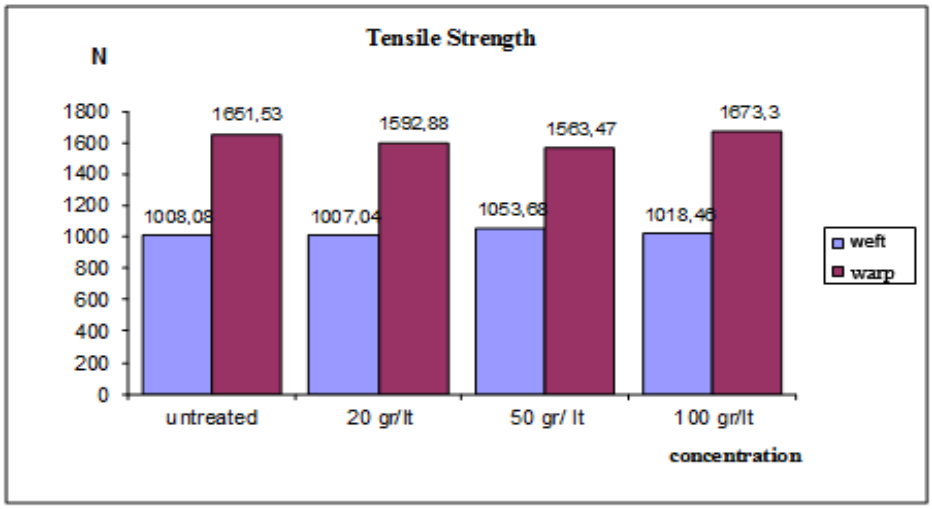


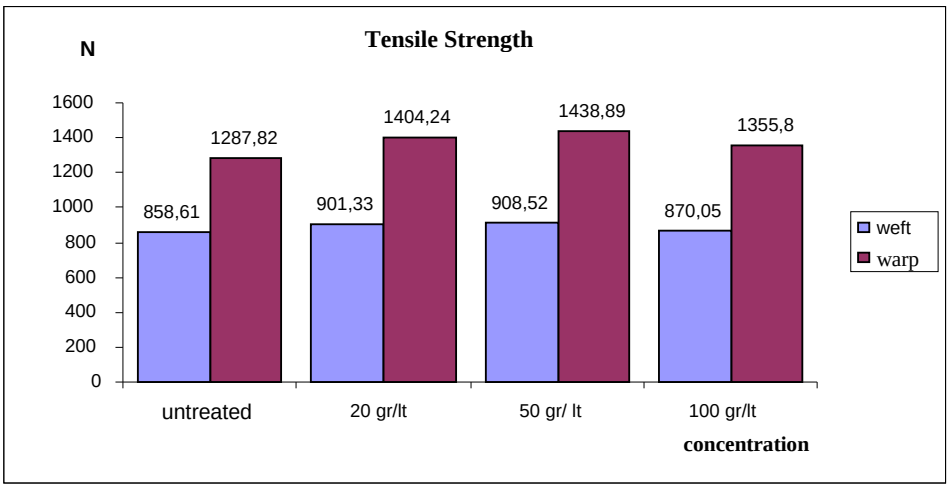
Figure 6.16. % mass change of denim fabric samples after abrasion cycles

6.4. Tensile Strength

Tensile strength of the samples was measured by Titan Tensile Strength tester. The average of three measurements in warp and weft direction is shown in Figure 6.16. CV values were found to be less than 10% for subsequent strength measurements of all types of fabric and application. Results show that fabric tensile strength after application of *Aloe vera* gel containing microcapsule solutions are not statistically significant but it results in an increase in the warp and weft direction generally.



(a)



(b)

Figure 6.17. Tensile strength values of a) gabardine and b) denim fabrics

The breaking strength test results belonging to denim fabric samples treated with microcapsule solutions coded as *AA*, *BB*, *CC*, *DD*, AA_m, BB_m, CC_m, and DD_m were

given in Figure 6.17. *Aloe vera* gel microcapsules do not have a negative impact on tensile strength of denim samples. An increase in tensile strength in both warp and weft directions was observed. However, the samples treated with glycerin containing microcapsule solutions have a higher values than the others due to its plasticizer property. CV values for all fabric and application types are found to be less than 10%.

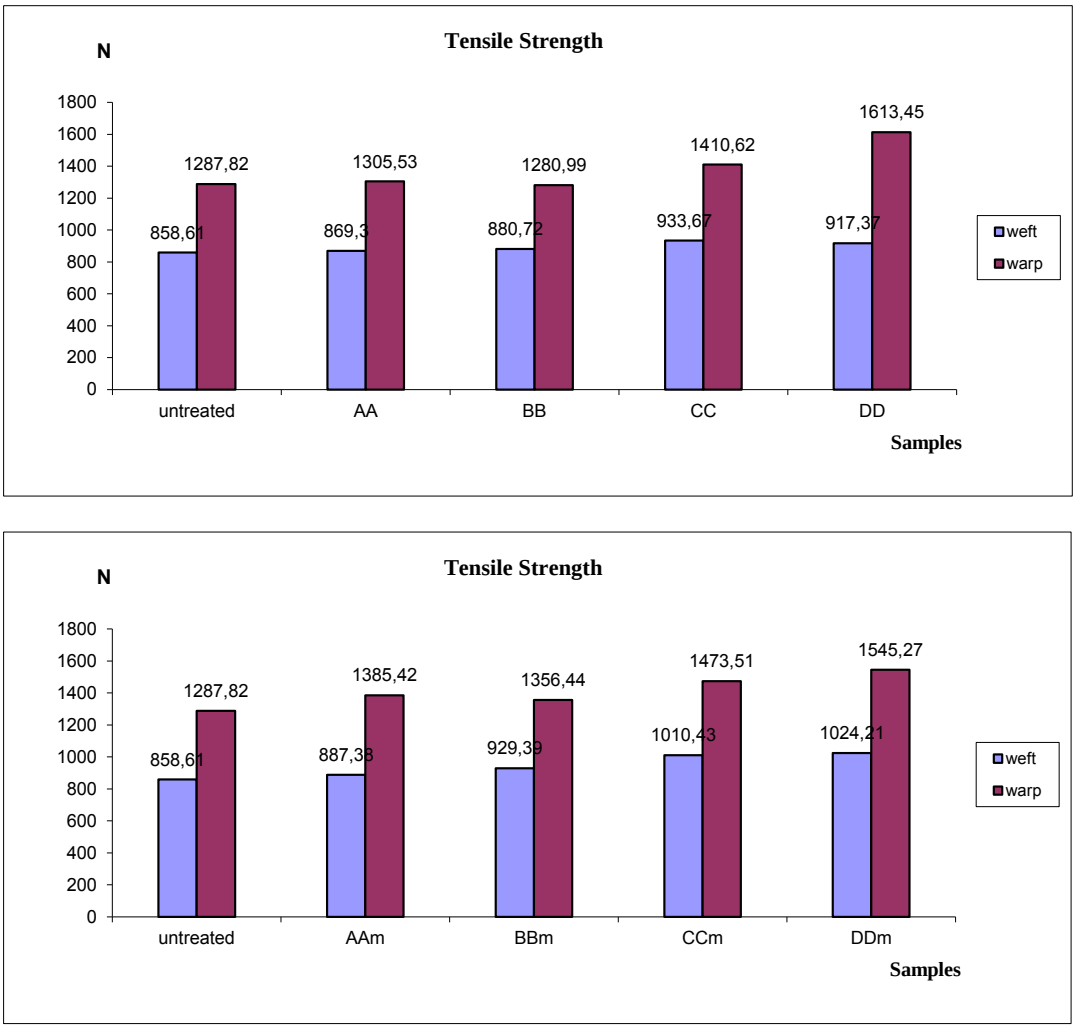


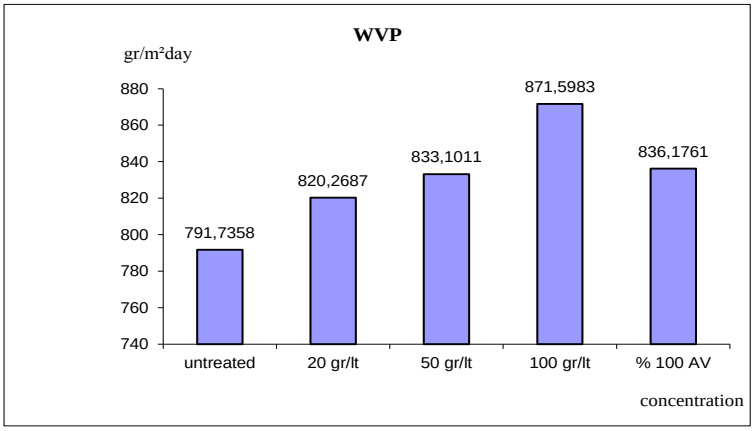
Figure 6.18. Tensile strength values of denim fabric samples

6.5. Water Vapor Permeability

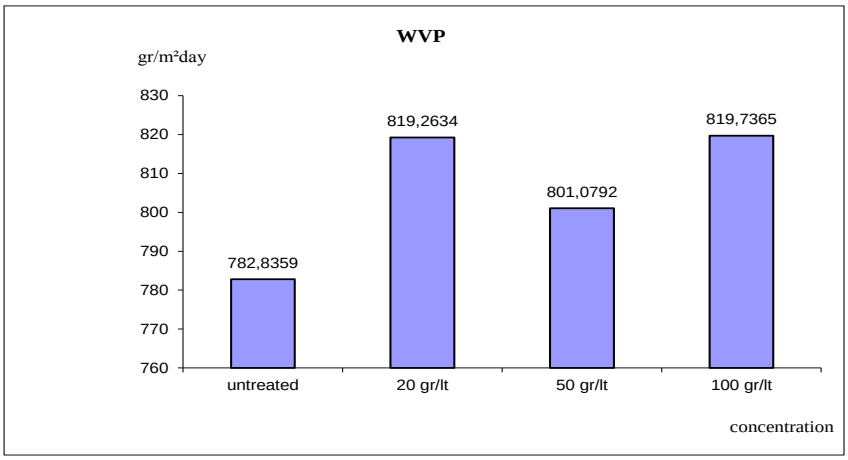
Water vapor permeability test is made to assess the breathability of the textile surfaces. Breathability is one of the determining factors of clothing comfort. The test is made with SDL Atlas Water Vapor Permeability Tester according to the BS 7209

standard. Water vapor permeability is expressed in terms of g/m²h which is the mass of water vapor passing thorough a unit area in a unit time.

Water vapor permeability values of gabardine (a) and denim (b) fabrics are shown in Figure 6.18. The results show that there is an increase in water vapor permeability after application of Aloe vera microcapsules when 20, 50 and 100 gr/L of microcapsule solutions were prepared.



(a)



(b)

Figure 6.19. Water vapor permeability values of a) gabardine, and b) denim fabric samples

Increase in tensile strength of denim fabrics treated with Aloe vera microcapsules can be explained by the installation of additional H bonds in the fabric structure, while the increase in water vapor permeability value is due to the increase in

humidity content of the fabric. These results show that microcapsules containing Aloe vera gel have moisturizing (wellness) effect on the denim fabric.

The water vapor permeability test results belonging to denim fabric samples treated with microcapsule solutions coded as *AA*, *BB*, *CC*, *DD*, AA_m, BB_m, CC_m, and DD_m were given below. The results show that microcapsule application slightly reduced water vapor permeability of the samples when they were not diluted with water but this reduction is not significant commercially. There is a higher reduction in vapor permeability of the samples when they are treated with glycerin containing microcapsule solutions.

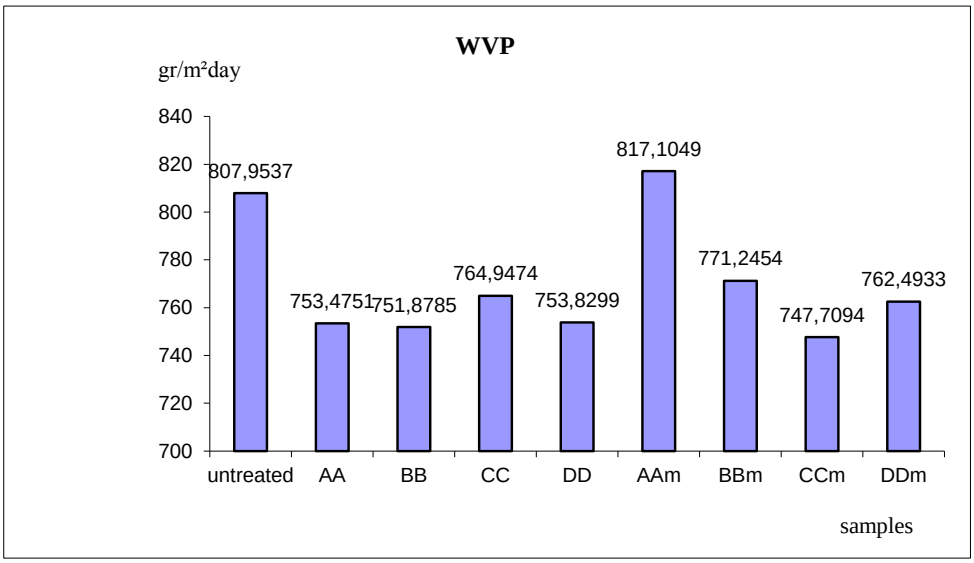


Figure 6.20. Water vapor permeability values of denim fabric samples

6.6. Dry and Wet Rubbing Fastness

Dry and wet rubbing fastness test was performed by Atlas rubbing fastness tester. Evaluation was made on 3 samples from each fabric. The average of the results are given in Table 6.14. *Aloe vera* gel microcapsules did not cause a negative impact on dry and wet rubbing fastness of denim fabric samples while a slight improvement was observed in the dry rubbing fastness.

Table 6.14. Dry and wet rubbing fastness of A) untreated denim fabric samples, and denim fabric samples treated with B) 1:100 diluted C) %100 *Aloe vera* gel microcapsules.

Degree of fastness (1-5)	Samples		
	A	B	C
Dry fastness	2/3	3	3
Wet fastness	1	1	1

The dry and wet fastness values of denim samples treated with microcapsule solutions coded as *AA, BB, CC, DD, AA_m, BB_m, CC_m,* and *DD_m* were given in Table 6.15. According to the test results, it can be said that *Aloe vera* gel microcapsules has no negative impact on dry and wet rubbing fastness of denim samples while there is a slight improvement in dry fastness. This improvement can be seen more clearly when the samples are treated with glycerin containing microcapsule solutions.

Table 6.15. Dry and wet rubbing fastness of denim fabric samples treated with different recipes; E is untreated sample.

Degree of fastness (1-5)	Samples								
	AA	AA_m	BB	BB_m	CC	CC_m	DD	DD_m	E
Dry fastness	2/3	3	3	3	3	3/4	3	3	2/3
Wet fastness	1	1	1	1	1	1	1	1	1

6.7. Color Difference

The color difference values of the fabrics treated with *Aloe vera* gel microcapsules are determined by Datacolor 650 spectrophotometer under D65/100 illuminator according to the CIELAB 1976 color space and given in Table 6.16. Results are the average of four measurements from each fabric which has been rotated 90° after each measurement. Color difference is found by taking untreated fabric as standard.

The results on Table 6.16 and 6.17 show that *Aloe vera* microcapsules do not lead yellowing on bleached gabardine fabrics (with a high L * luminance values) and do

not cause an increase in brightness on the fabric surface. It can be said that both denim and gabardine fabric have acceptable color difference. The results in Table 6.16 also shows that there is no a significant difference in color when 100% *Aloe vera* gel containing microcapsules solution is used as completely finishing bath or it is diluted with different concentrations.

Table 6.16. Color and color difference values of denim and gabardine fabric samples

Gabardine	L*	a*	b*	Δ	CIE Whiteness Index
Untreated	94,05	-0,32	3,21	-	70,80
20 gr\lt	93,30	-0,58	3,33	0,80	70,58
50 gr\lt	94,07	-0,44	3,28	0,14	69,55
100 gr\lt	93,99	-0,43	3,43	0,25	66,04
% 100 AV	93,46	-0,34	3,89	0,90	70,72

Denim	L*	a*	b*	Δ
Untreated	23,69	0,63	-3,22	-
20 gr\lt	22,04	0,89	-3,47	1,69
50 gr\lt	22,17	0,88	-3,40	1,56
100 gr\lt	22,26	0,83	-3,41	1,45
% 100 AV	22,14	0,55	-3,29	1,56

Table 6.17. Color and color difference values of denim samples treated with microcapsule solutions coded as *AA*, *BB*, *CC*, *DD*, AA_m, BB_m, CC_m, and DD_m ; E is untreated samples.

Samples	AA	BB	CC	DD	AA_m	BB_m	CC_m	DD_m	E
L*	23,04	23,58	23,24	23,59	22,06	22,37	21,93	22,37	24,11
a*	0,77	0,26	0,20	0,10	0,88	0,83	1,00	0,56	0,63
b*	-3,09	-2,72	-2,99	- 2,77	-3,21	-2,83	-3,07	-3,14	-2,95
Δ	1,49	1,14	1,42	1,06	1,72	1,38	1,86	1,36	-

6.8. FTIR Analysis

The IR spectrum of *Aloe vera* gel is observed in Figure 7.20. Spectrum shows a sharp and broad peak at 3200 cm^{-1} and a narrow moderate peak at 1630 cm^{-1}. These peaks

are evaluated as characteristic OH bending and C = O bending peaks. This results indicate that Aloe vera gel comprise water and -OH groups of phenolic compounds.

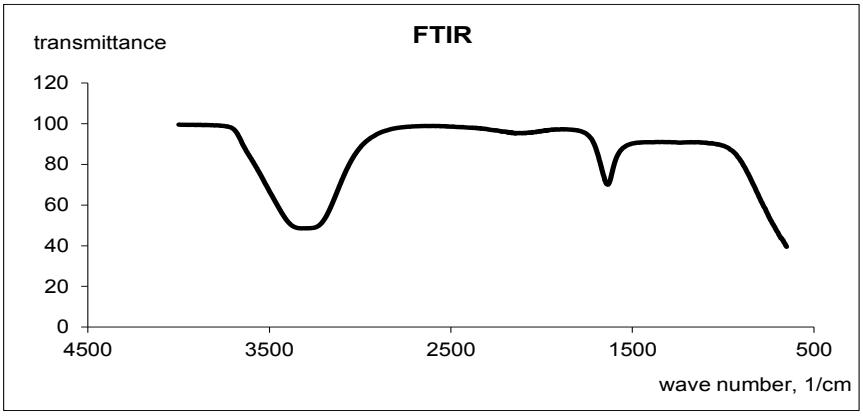
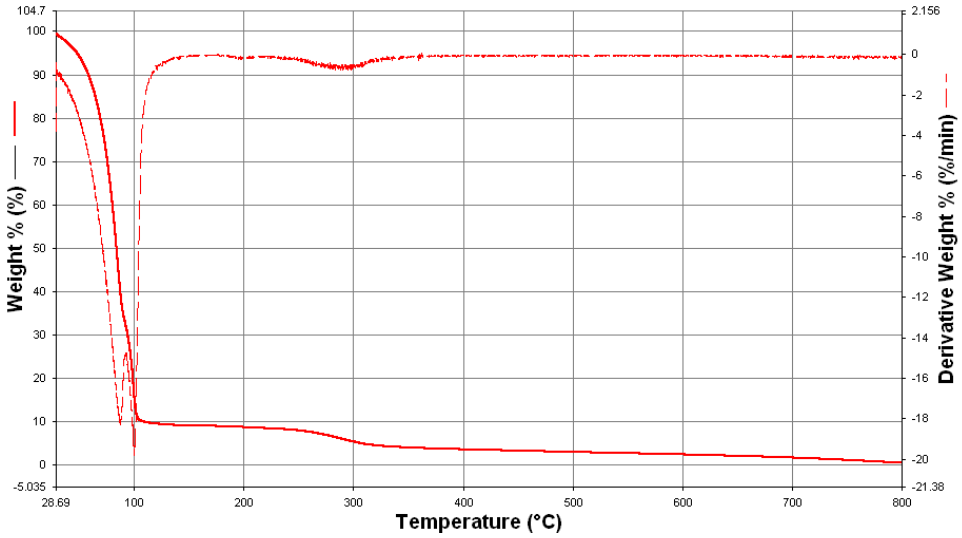


Figure 6.21. FTIR analysis of *Aloe vera* gel

6.9. Thermal Analysis

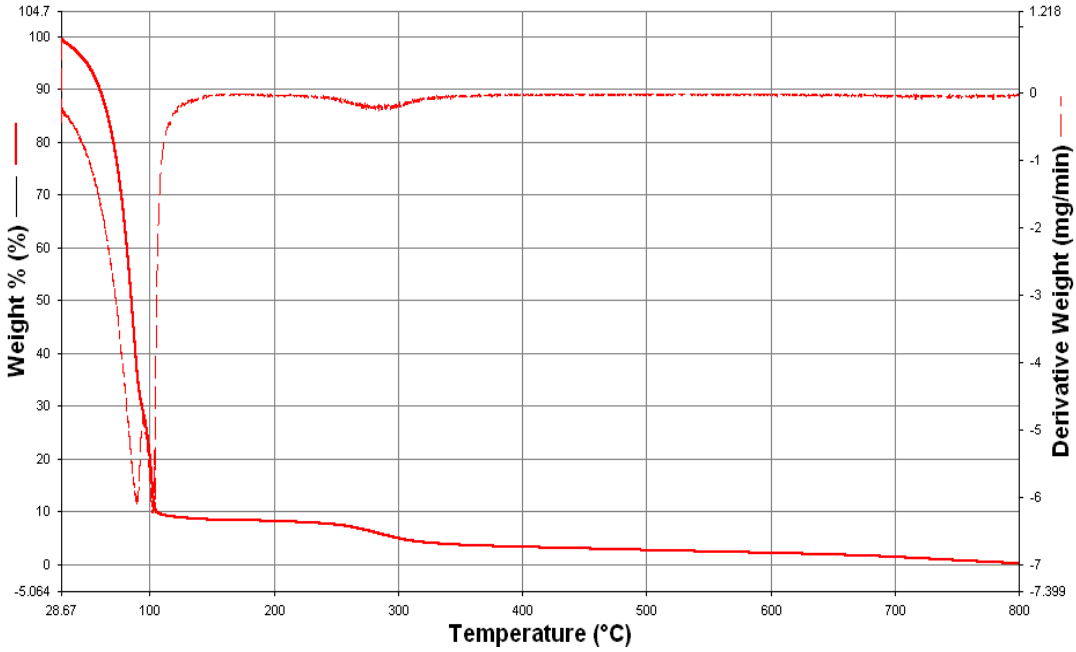
6.9.1. TGA Analysis

Thermogravimetric analysis (TGA) graphs belonging to the microcapsule solutions prepared with 100% and 1: 100 diluted *Aloe vera* gel and the denim fabrics untreated and treated with these solutions are shown in Figures 6.22-6.26.



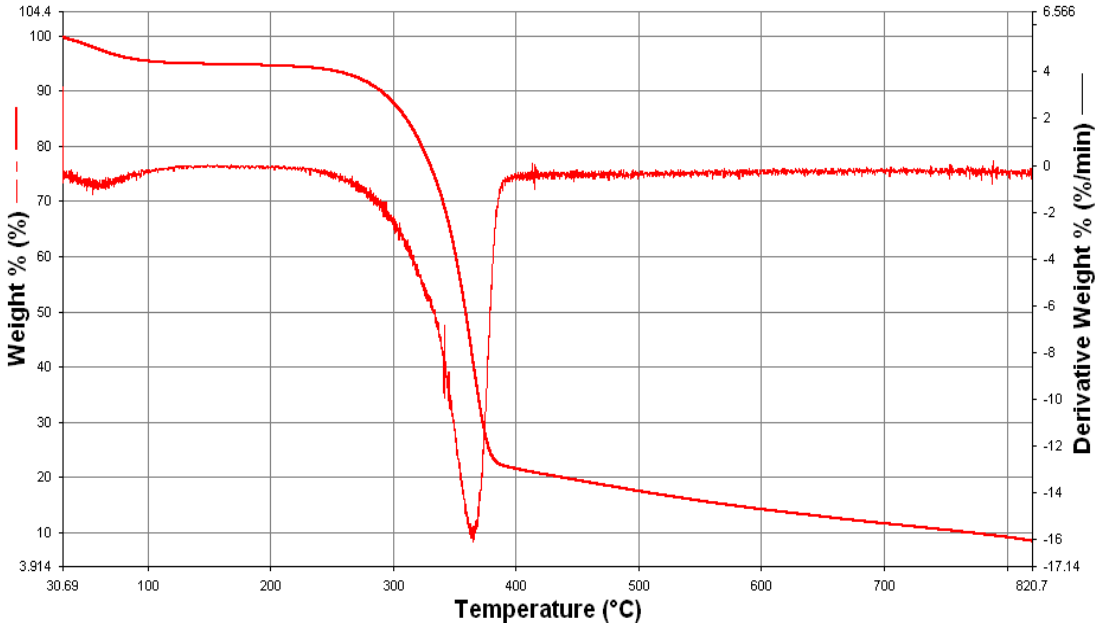
% Loss = 99.263%

Figure 6.22. TGA graph of 100 % *Aloe vera* gel microcapsule solution



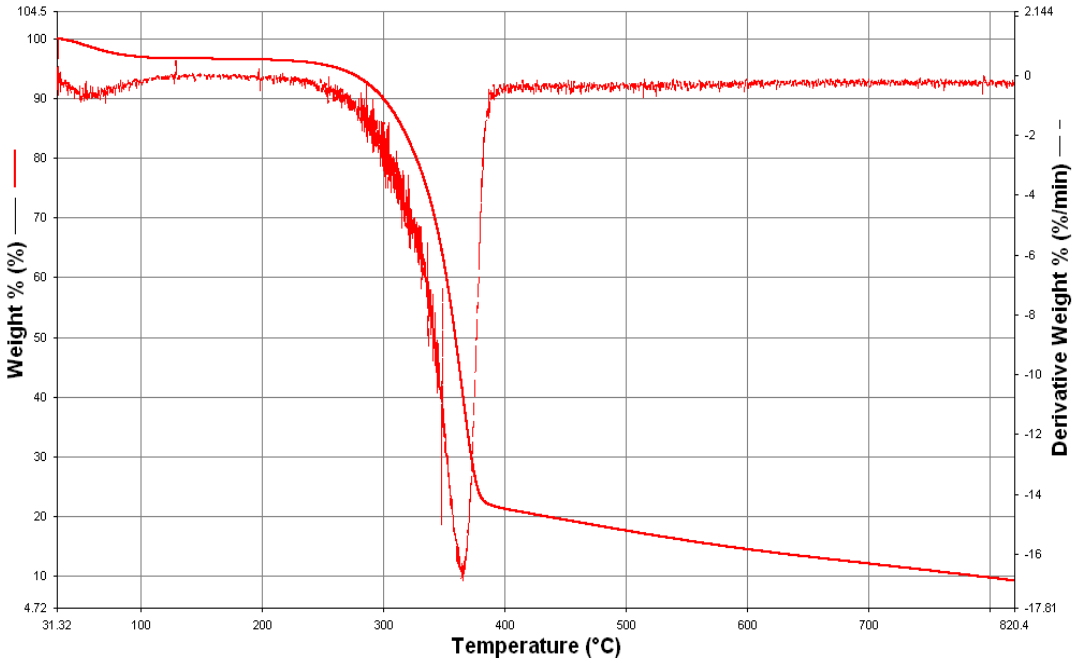
% Loss = 99.498%

Figure 6.23. TGA graph of microcapsule solution containing 1:100 diluted *Aloe vera* gel with distilled water



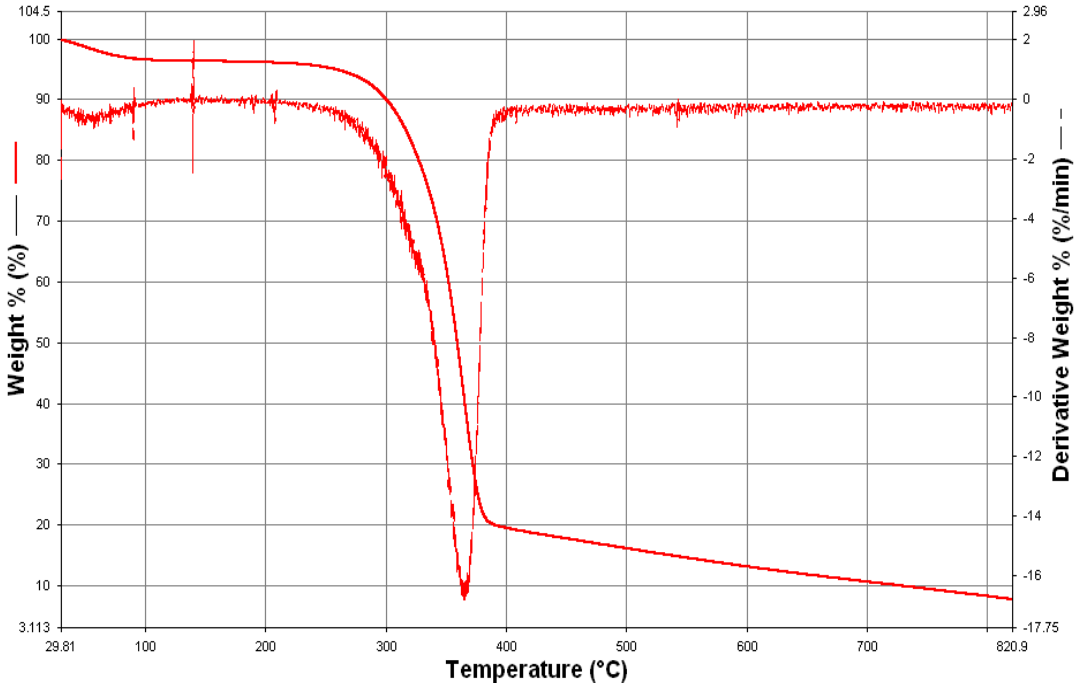
% Loss = 91.309%

Figure 6.24. TGA graph of denim fabric treated with microcapsule solution containing 1:100 diluted *Aloe vera* gel with distilled water



% Loss = 90.710%

Figure 6.25. TGA graph of denim fabric treated with microcapsule solution containing 100 % *Aloe vera* gel



% Loss = 92.169 %

Figure 6.26. TGA graph of untreated denim fabric

Figure 6.27 shows the TGA graph of all samples together where a) 100% b) 1:100 diluted AV gel containing microcapsule solutions and c, d) samples treated with these solutions respectively, e is untreated sample.

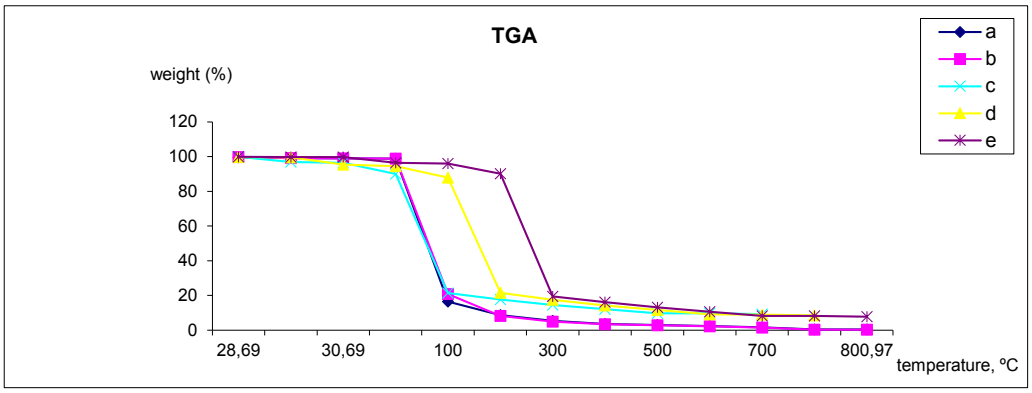
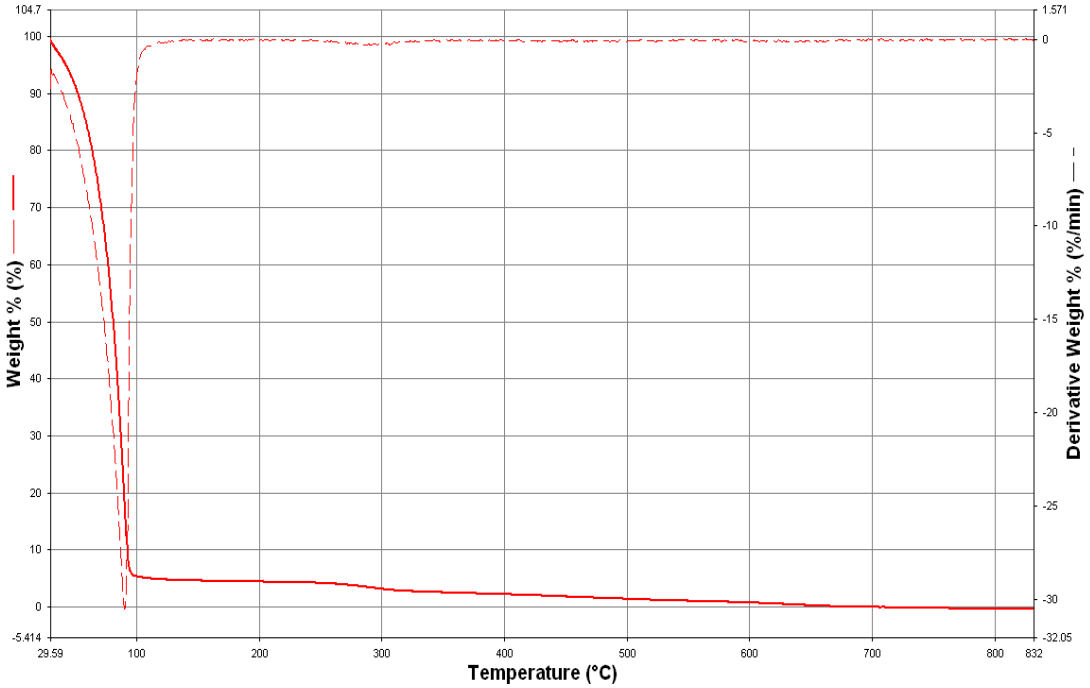


Figure 6.27. TGA graph of all the samples together

TGA analyzes of microcapsule solutions shows that a two-stage mass loss is seen for both solutions. In addition, % proportional weight change values for both solutions type has a shoulder peak in the range of 87,77 °C – 92,94 °C (for 100% *Aloe vera* gel) and 90,17 °C – 95,95 °C (for 1:100 diluted *Aloe vera* gel). These results suggest that microcapsule solutions containing *Aloe vera* gel lose weight quickly up to 87-90 °C (removal of water in solution), and a slowdown in mass loss is observed up to 92-95 °C (removal of the water within the gel microstructure). 99% of weight loss is observed for both solutions at the end of the test. TGA graphs being derived in a similar range shows that 100 % or 1:100 diluted *Aloe vera* gel used to obtain the microcapsules have no impact on the thermal stability of the solution.

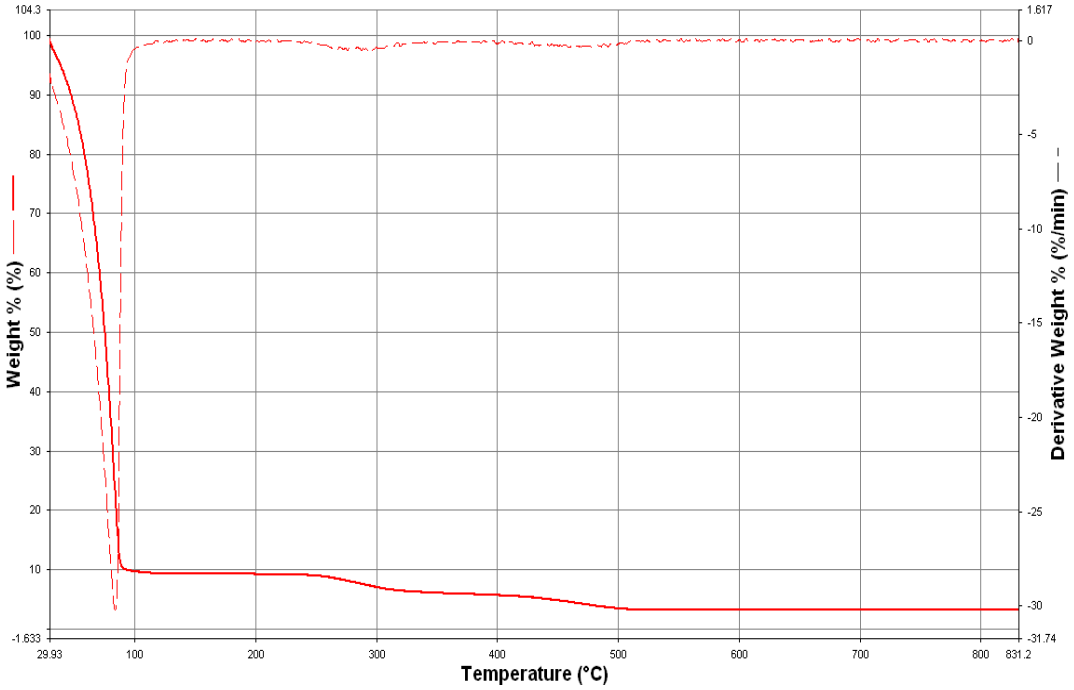
In TGA analyzes of denim fabrics, decay curves which have been observed in similar studies of cotton fabrics were seen (between 365-368 °C quick weight loss, around 10% residue), microcapsule application has not caused a negative impact on the thermal stability of the denim fabric.

Thermogravimetric analyses of microcapsule solutions coded as AA_m, BB_m and CC_m and the samples treated with these solutions were given in Figures 6.28-6.33.



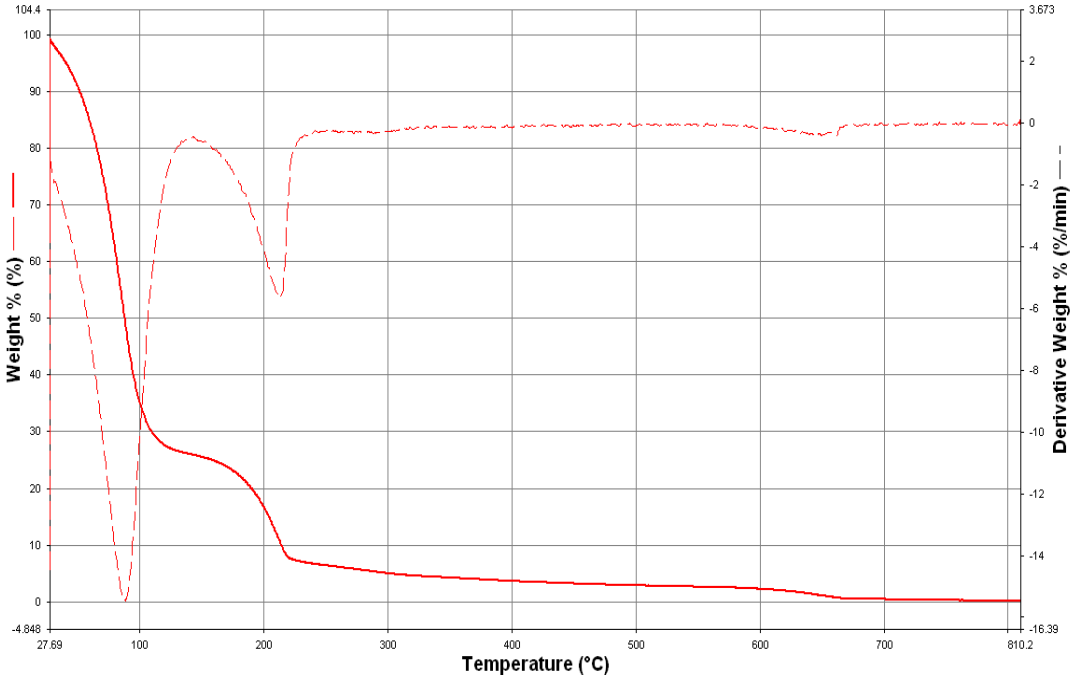
%Loss= 100%

Figure 6.28. TGA graph of AA_m coded microcapsule solution



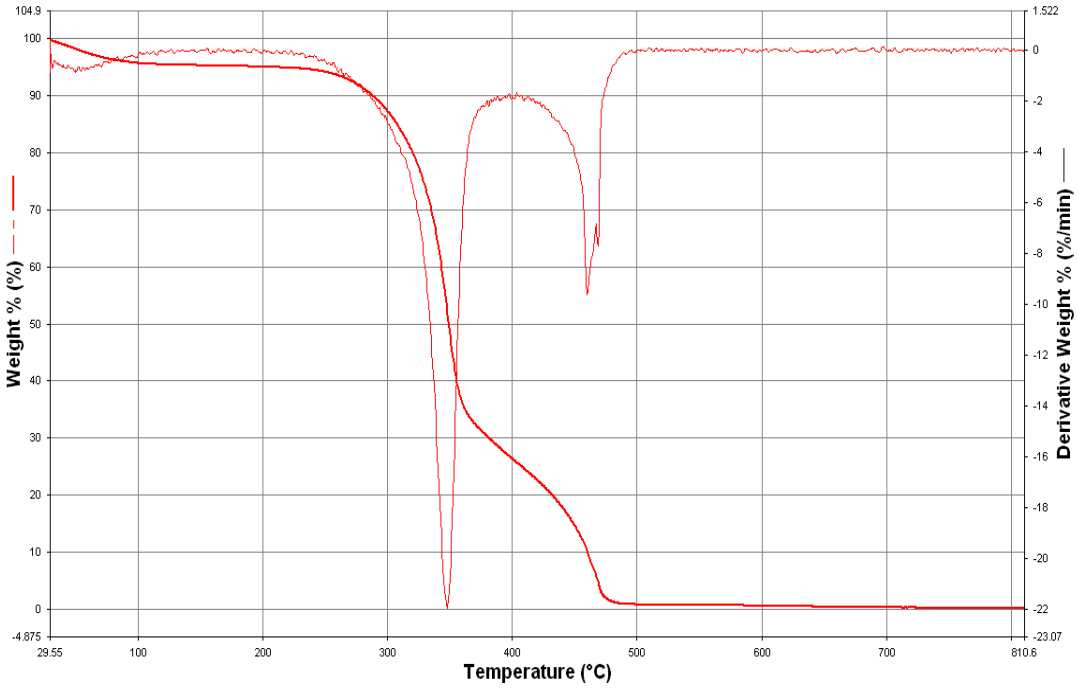
%Loss= 96.228%

Figure 6.29. TGA graph of BB_m coded microcapsule solution



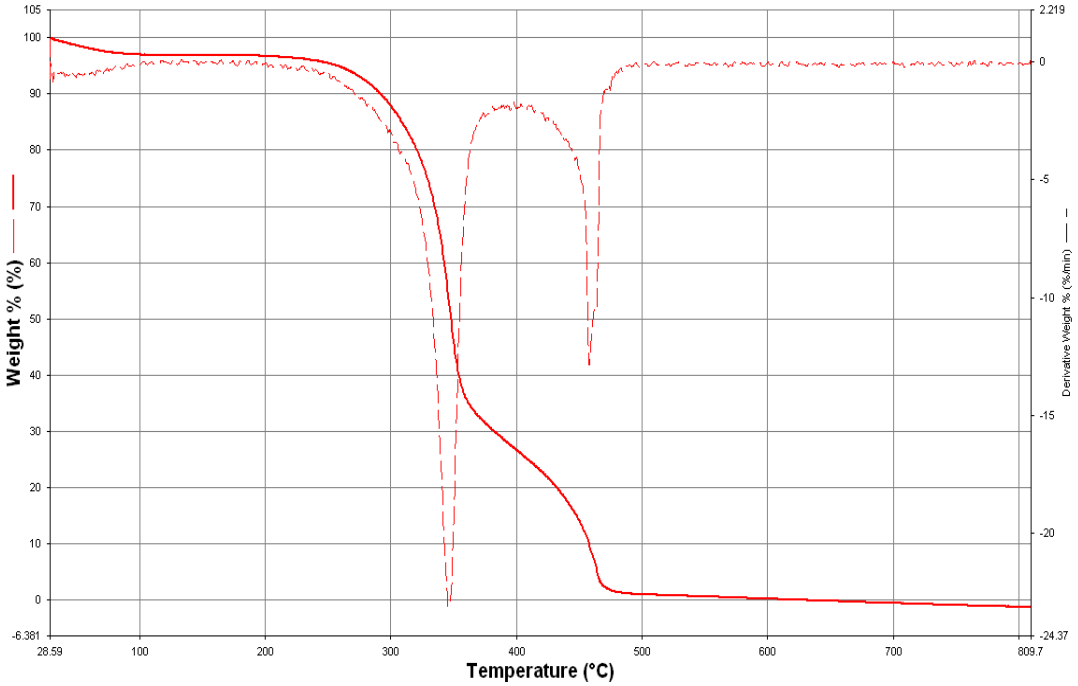
%Loss= 99.276%

Figure 6.30. TGA graph of CC_m coded microcapsule solution



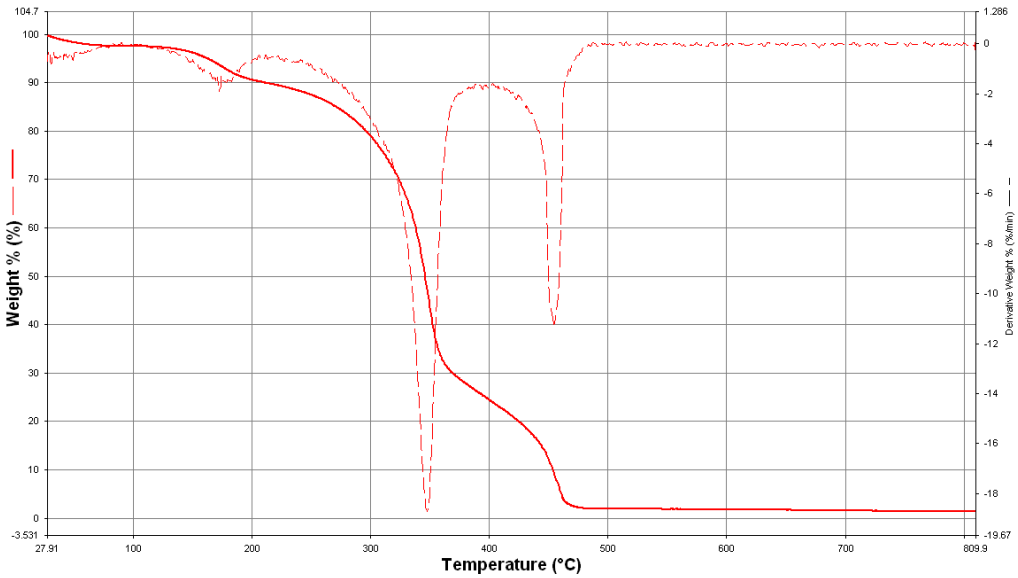
%Loss= 99.779%

Figure 6.31. TGA graph of denim sample treated with AA_m coded microcapsule solution



%Loss= 100%

Figure 6.32. TGA graph of denim sample treated with BB_m coded microcapsule solution



%Loss= 98.386%

Figure 6.33. TGA graph of denim sample treated with CC_m coded microcapsule solution

Figure 6.34 shows the TGA graph of all samples together where D, E, F are denim fabrics treated with microcapsule solutions coded as AA_m, BB_m, CC_m, respectively.

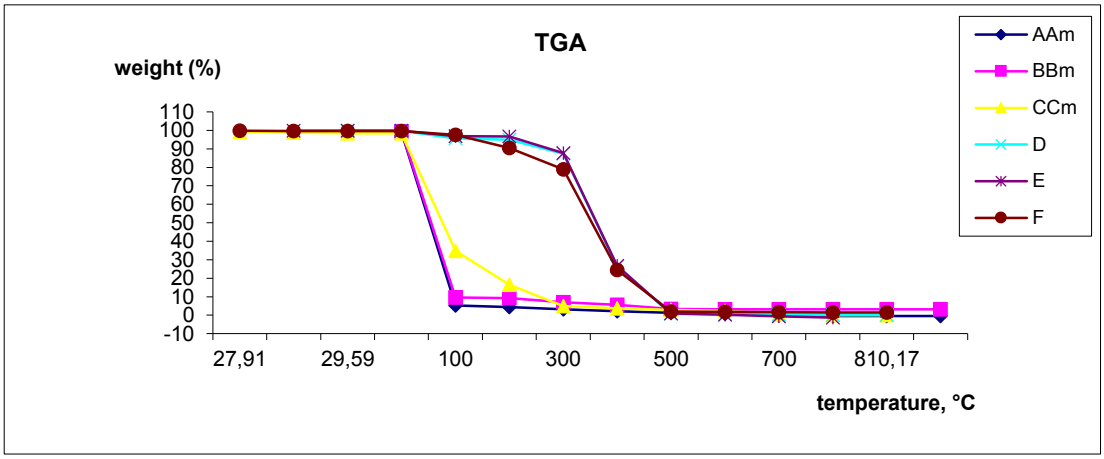


Figure 6.34. TGA graph of all samples together

TGA analysis of AA_m, BB_m, and CC_m coded microcapsule solutions show that there is a two-stage mass loss is observed for AA_m and BB_m while it results in 3 stages for CC_m. This is due to the glycerin content in the wall material of the microcapsule solution coded as CC_m. It can be seen that, for first two microcapsule solutions (AA_m and BB_m) there is a slow reduction in mass after about 93-94,5 ºC. However, the mass loss of AA_m (94%) is much more than that of BB_m (90%) at the same temperature. 100% loss for AA_m and 96% loss for BB_m is observed at the end of the test. The results are different for CC_m. There is a quick reduction in mass up to about 107 ºC due to the water content. Mass loss continues a bit slowly up to about 219 ºC due to the glycerin content and it continues much more slowly after that point. Mass loss for CC_m is 99% when the test is completed.

Denim samples treated with AA_m and BB_m microcapsule solutions exhibit similar characteristics with each other. It is a bit different for the sample treated with CC_m type microcapsule solution. At the same temperature, mass loss is lesser for CC_m than the other two samples. However, all these three samples have different thermal properties than untreated one. Because a quick weight loss has been observed for untreated denim sample between 365-368 °C with around 10% residue while the same amount of residue is observed at around 459, 457, and 453°C for denim samples treated with AA_m, BB_m, and CC_m, respectively. It can be said that, microcapsule application has a good impact on denim samples.

6.9.2. DSC Analysis

Enthalpy (DSC) analysis of denim samples are shown in Figures 6.35-6.37.

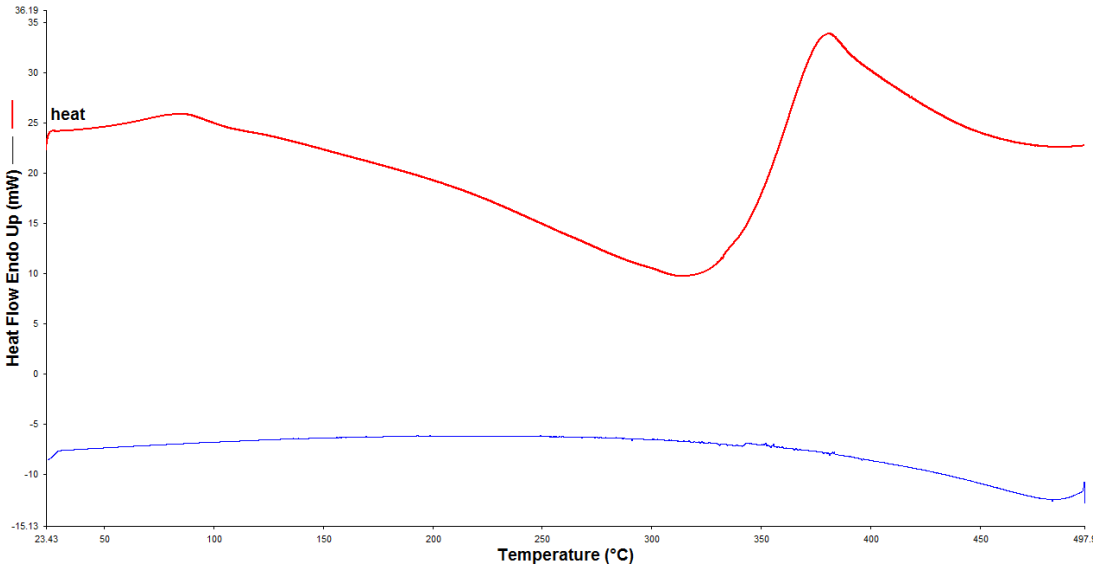


Figure 6.35. DSC graph of denim fabric treated with microcapsule solution containing 1:100 diluted *Aloe vera* gel with distilled water

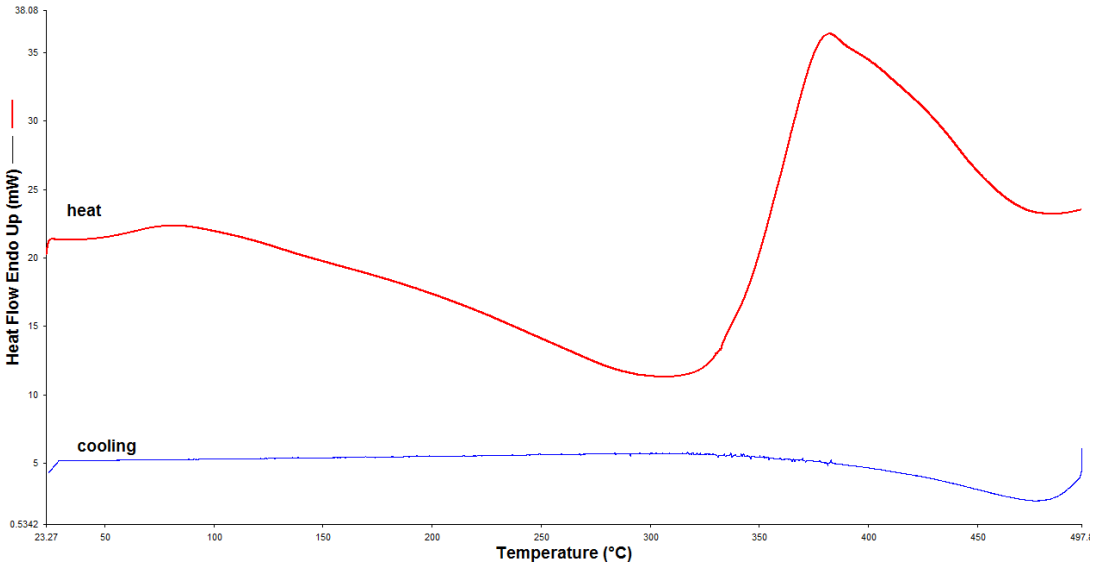


Figure 6.36. DSC graph of denim fabric treated with microcapsule solution containing 100% diluted *Aloe vera* gel with distilled water

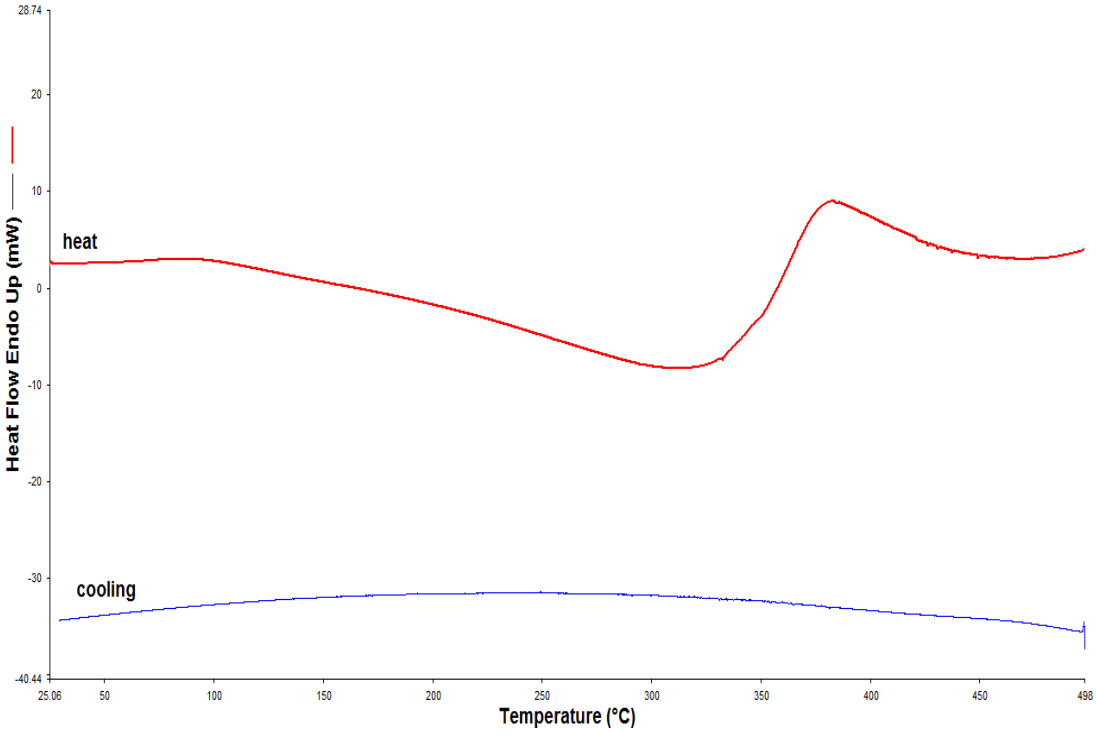


Figure 6.37. DSC graph of untreated denim fabric

Figure 6.38 shows the DSC graph of all samples together where c and d the samples treated with 100% and 1:100 diluted AV gel containing microcapsule solutions (BB type) respectively. e is untreated sample.

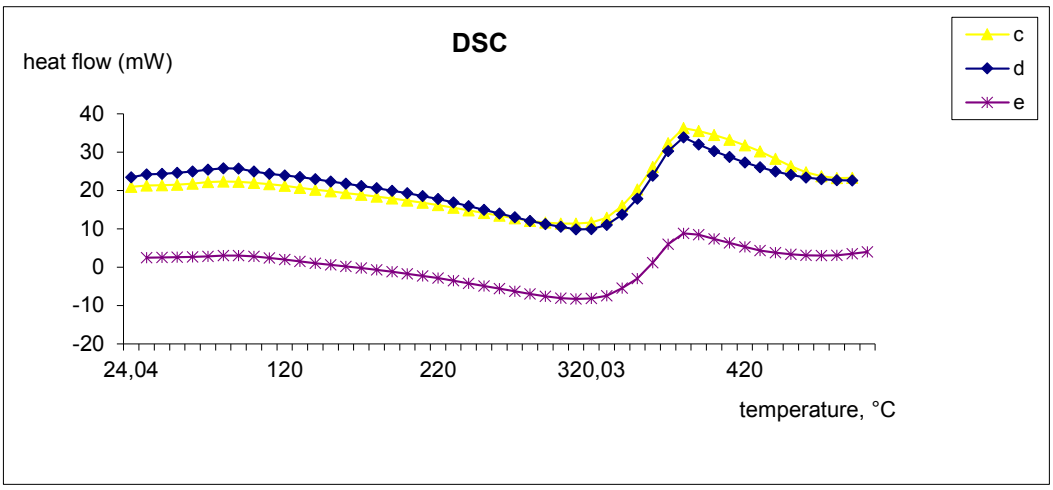


Figure 6.38. DSC analysis of all samples together

DSC analyzes demonstrate that a higher specific heat capacity (higher heat flux values) is obtained for the fabric samples treated with microcapsule solution containing *Aloe vera* gel. This means that treated denim fabric has a higher thermal

absorbency and thermal conductivity values. Because thermal conductivity and thermal absorbance values of a fabric is associated with the moisture content in evaluating of the fabric comfort in many studies. DSC analysis confirmed the dampening effect (wellness) of microcapsules containing *Aloe vera* gel on denim fabric. In addition, when DSC analyzes are examined, it can be seen that the endothermic water rupture which has been observed around 385^0C in denim fabrics occurred with more thermal energy (greater shoulder peak) after treatment with microcapsules. This case is also explained by the increase in amount of humidity within the fabric.

DSC analysis results of the samples treated with microcapsule solutions coded as AA_m, BB_m, and CC_m were shown in Figures 6.39-6.41.

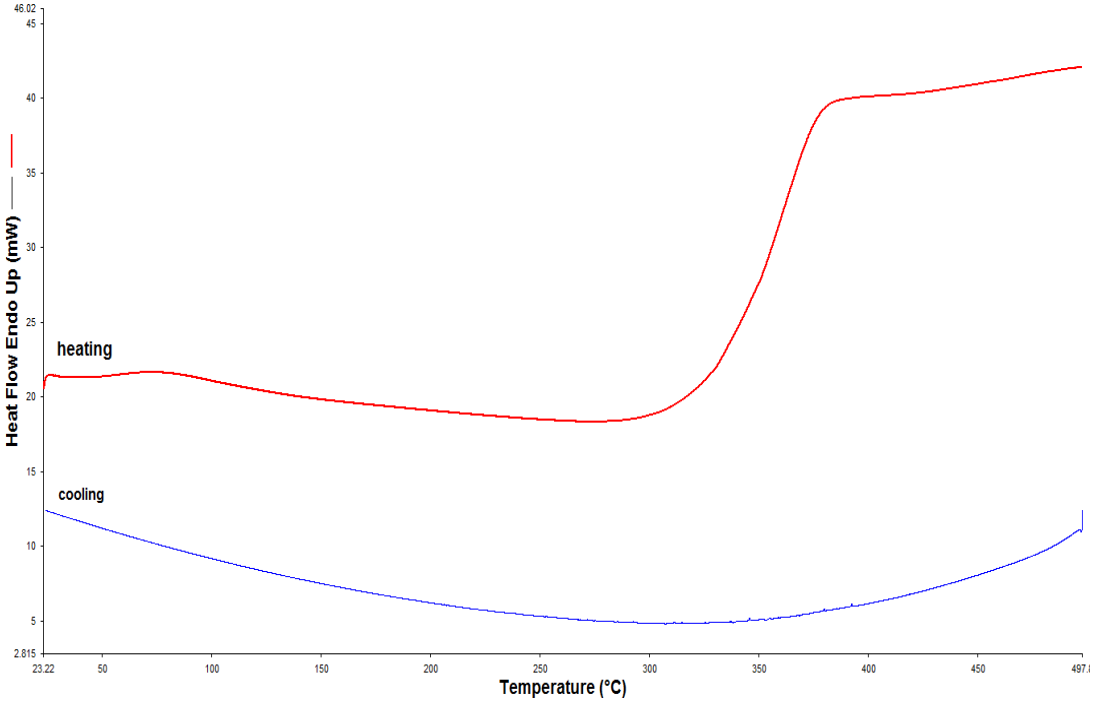


Figure 6.39. DSC analysis of denim fabric treated with AA_m coded microcapsule solution.

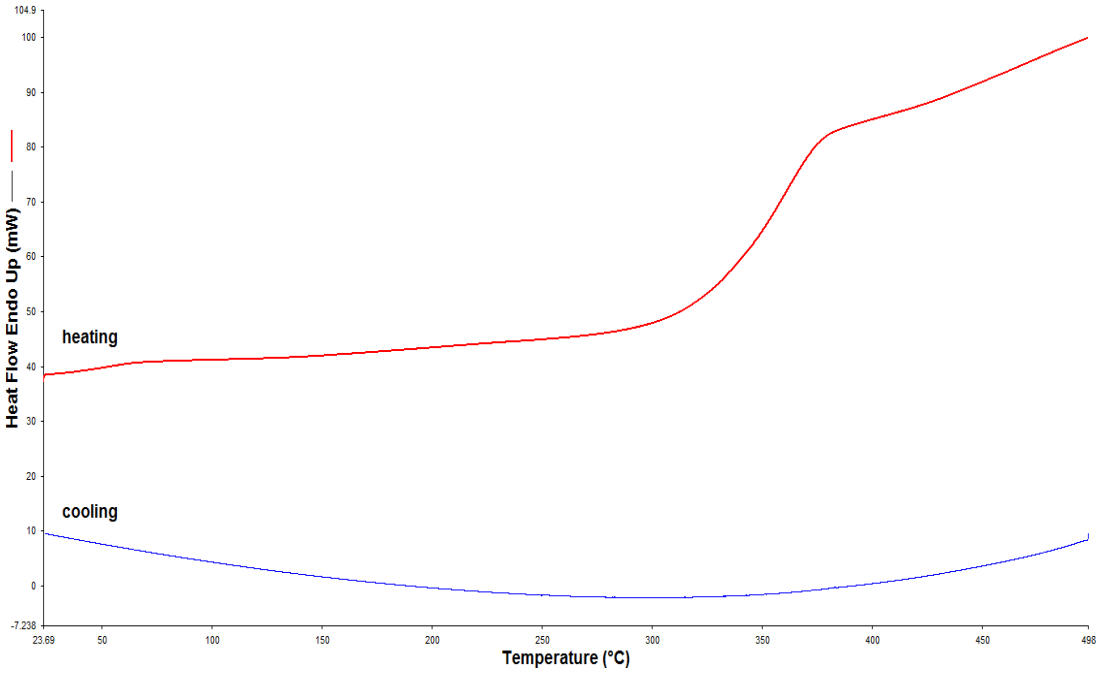


Figure 6.40. DSC analysis of denim fabric treated with BB_m coded microcapsule solution.

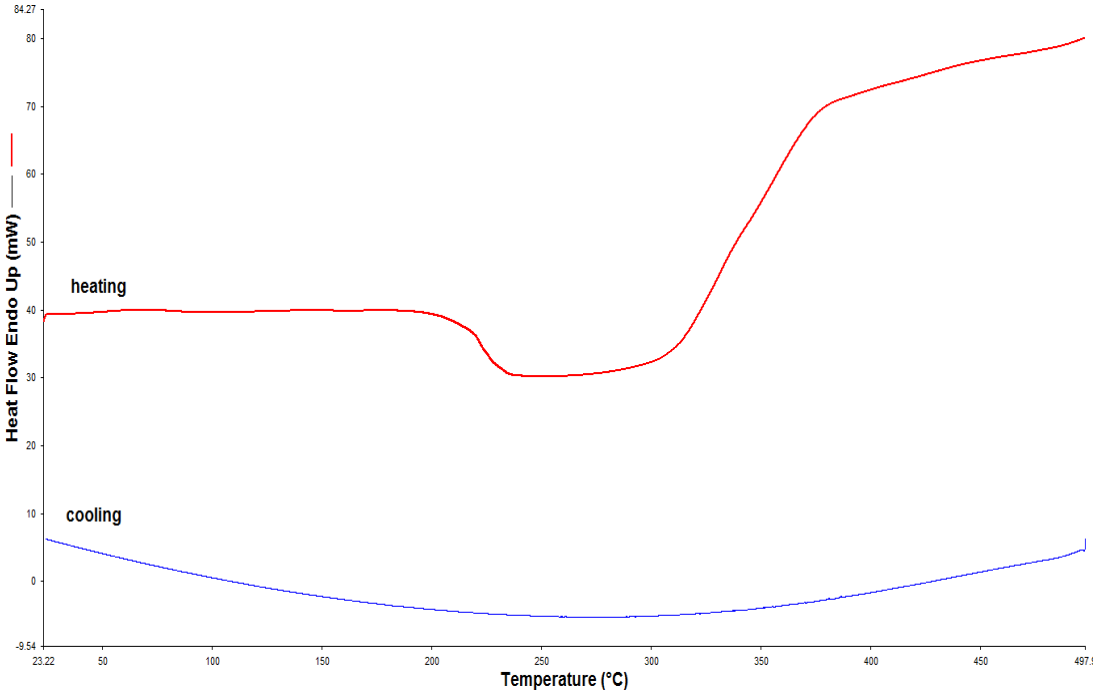


Figure 6.41. DSC analysis of denim fabric treated with CC_m coded microcapsule solution.

Figure 6.42 shows the DSC graph of all samples together where D, E, and F are denim samples treated with microcapsule solutions coded as AAm, BBm, CCm respectively.

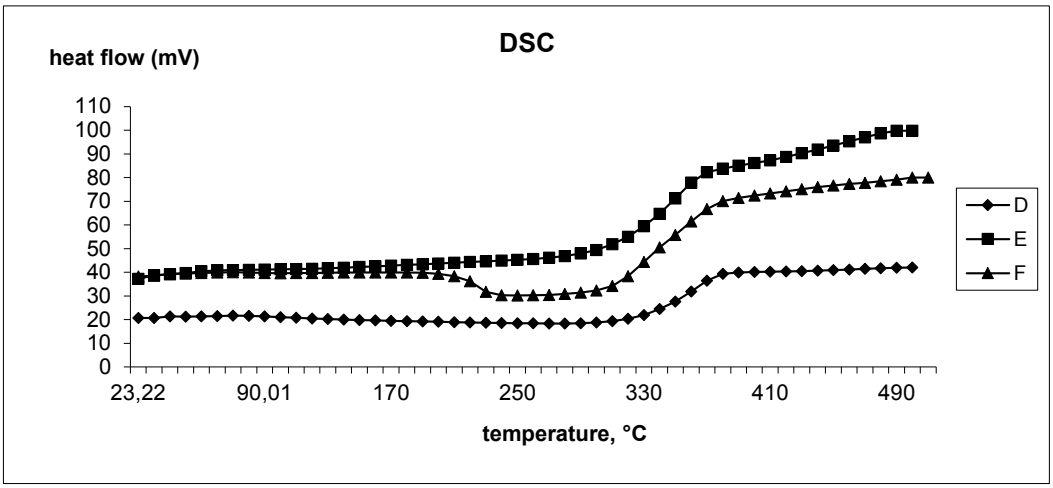
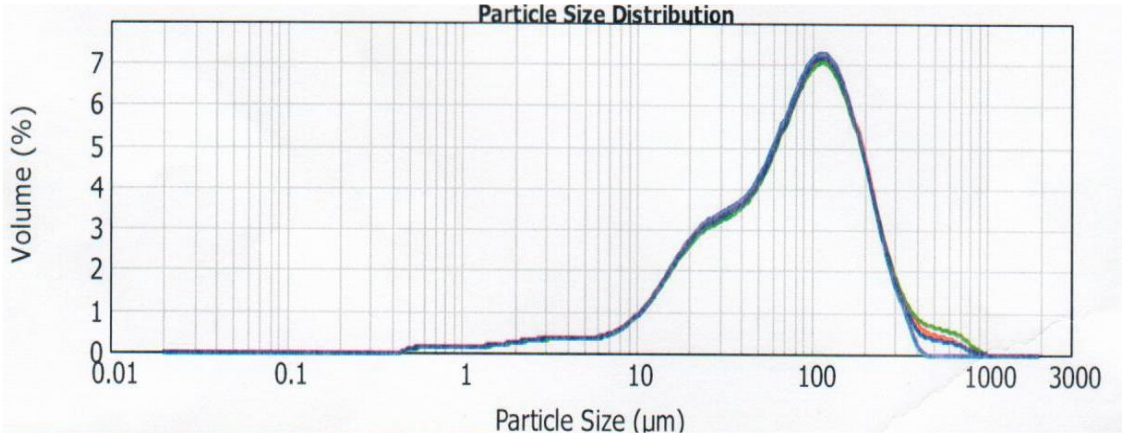


Figure 6.42. DSC graph of all samples together

The results show that glycerin content in microencapsulation provide a limited heat absorption poperty to the samples. It can be clearly seen in Figure 6.42 where a tub occurs on heating line. In addition, thermal conductivity values are higher for the samples treated with microcapsule solution having 1:5 core:wall ratio.

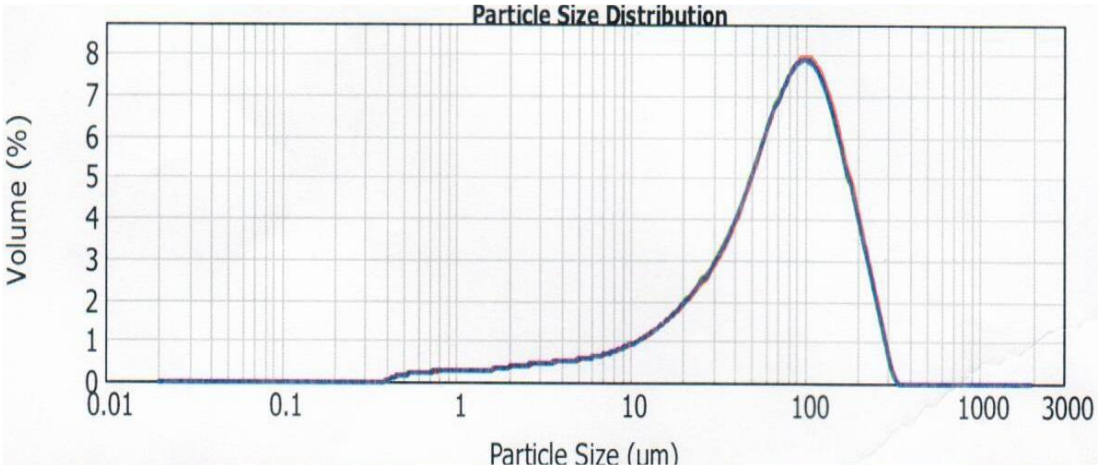
6.10. Particle Size Analysis

Particle size distribution analysis of the solutions containing microcapsules prepared according to the simple coacervation method of is given in Figures 6.43-6.44. The values found when the mean particle size of particles in solution (microcapsules) are calculated as a function of the surface area and volume are given in Table 6.18. According to these results it is possible to produce smaller capsules (11 to 310 μm) by the use of 1:100 diluted *Aloe vera* gel, but when 100% *Aloe vera* gel is used the resulting microcapsules have a smaller particle size variation (larger polydispersity value).



Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %
0.010	0.00	0.105	0.00	1.096	0.13	11.482	1.21	120.226	6.35	1258.925	0.00
0.011	0.00	0.120	0.00	1.259	0.14	13.183	1.55	138.038	5.95	1445.440	0.00
0.013	0.00	0.138	0.00	1.445	0.16	15.136	1.90	158.489	5.28	1659.587	0.00
0.015	0.00	0.158	0.00	1.660	0.19	17.378	2.24	181.970	4.40	1905.461	0.00
0.017	0.00	0.182	0.00	1.905	0.22	19.953	2.53	208.930	3.44	2187.762	0.00
0.020	0.00	0.209	0.00	2.188	0.26	22.909	2.76	239.883	2.50	2511.886	0.00
0.023	0.00	0.240	0.00	2.512	0.28	26.303	2.91	275.423	1.69	2884.032	0.00
0.026	0.00	0.275	0.00	2.884	0.30	30.200	3.03	316.228	1.10	3311.311	0.00
0.030	0.00	0.316	0.00	3.311	0.31	34.674	3.16	363.078	0.64	3801.894	0.00
0.035	0.00	0.363	0.00	3.802	0.31	39.811	3.35	416.869	0.39	4365.158	0.00
0.040	0.00	0.417	0.00	4.365	0.31	45.709	3.66	478.630	0.32	5011.872	0.00
0.046	0.00	0.479	0.00	5.012	0.32	52.481	4.09	549.541	0.28	5754.399	0.00
0.052	0.00	0.550	0.08	5.754	0.34	60.256	4.64	630.957	0.23	6606.934	0.00
0.060	0.00	0.631	0.11	6.607	0.40	69.183	5.24	724.436	0.13	7585.776	0.00
0.069	0.00	0.724	0.12	7.586	0.51	79.433	5.81	831.764	0.04	8709.636	0.00
0.079	0.00	0.832	0.13	8.710	0.68	91.201	6.24	954.993	0.00	10000.000	
0.091	0.00	0.955	0.13	10.000	0.92	104.713	6.44	1096.478	0.00		
0.105		1.096	0.13	11.482		120.226		1258.925			

Figure 6.43. Particle size analysis of microcapsule solution containing 100% *Aloe vera* gel



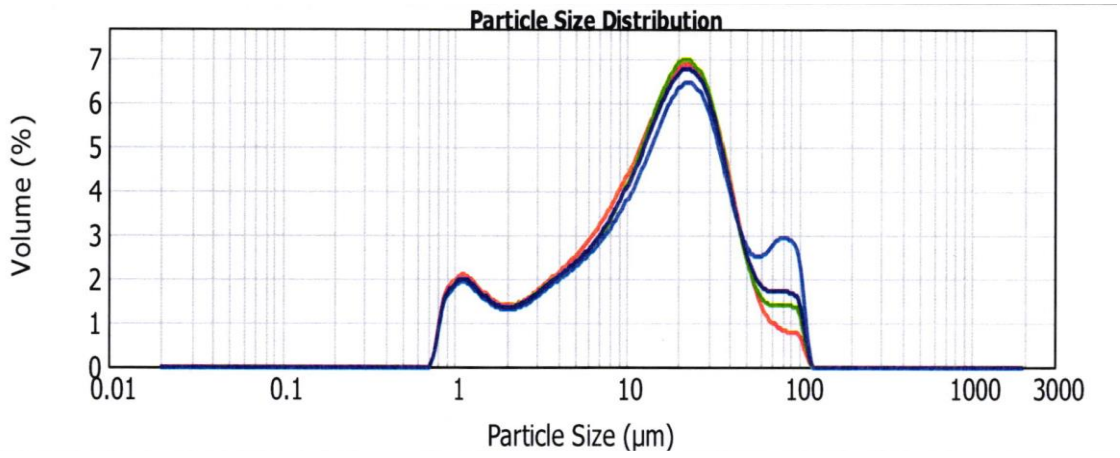
Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %
0.010	0.00	0.105	0.00	1.096	0.23	11.482	1.01	120.226	6.57	1258.925	0.00
0.011	0.00	0.120	0.00	1.259	0.24	13.183	1.17	138.038	5.85	1445.440	0.00
0.013	0.00	0.138	0.00	1.445	0.26	15.136	1.35	158.489	4.92	1659.587	0.00
0.015	0.00	0.158	0.00	1.660	0.29	17.378	1.57	181.970	3.87	1905.461	0.00
0.017	0.00	0.182	0.00	1.905	0.32	19.953	1.82	208.930	2.85	2187.762	0.00
0.020	0.00	0.209	0.00	2.188	0.35	22.909	2.10	239.883	1.81	2511.886	0.00
0.023	0.00	0.240	0.00	2.512	0.38	26.303	2.43	275.423	0.79	2884.032	0.00
0.026	0.00	0.275	0.00	2.884	0.40	30.200	2.81	316.228	0.03	3311.311	0.00
0.030	0.00	0.316	0.00	3.311	0.43	34.674	3.27	363.078	0.00	3801.894	0.00
0.035	0.00	0.363	0.00	3.802	0.45	39.811	3.82	416.869	0.00	4365.158	0.00
0.040	0.00	0.417	0.10	4.365	0.47	45.709	4.45	478.630	0.00	5011.872	0.00
0.046	0.00	0.479	0.14	5.012	0.50	52.481	5.14	549.541	0.00	5754.399	0.00
0.052	0.00	0.550	0.18	5.754	0.55	60.256	5.83	630.957	0.00	6606.934	0.00
0.060	0.00	0.631	0.21	6.607	0.60	69.183	6.45	724.436	0.00	7585.776	0.00
0.069	0.00	0.724	0.22	7.586	0.67	79.433	6.90	831.764	0.00	8709.636	0.00
0.079	0.00	0.832	0.22	8.710	0.76	91.201	7.11	954.993	0.00	10000.000	
0.091	0.00	0.955	0.22	10.000	0.87	104.713	7.00	1096.478	0.00		
0.105		1.096		11.482		120.226		1258.925			

Figure 6.44. Particle size analysis of microcapsule solution containing 1:100 diluted *Aloe vera* gel

Table 6.18. Surface and volume weighted mean particle size and homogeneity values of A) %100 B) 1:100 diluted aloe vera containing microcapsule solutions

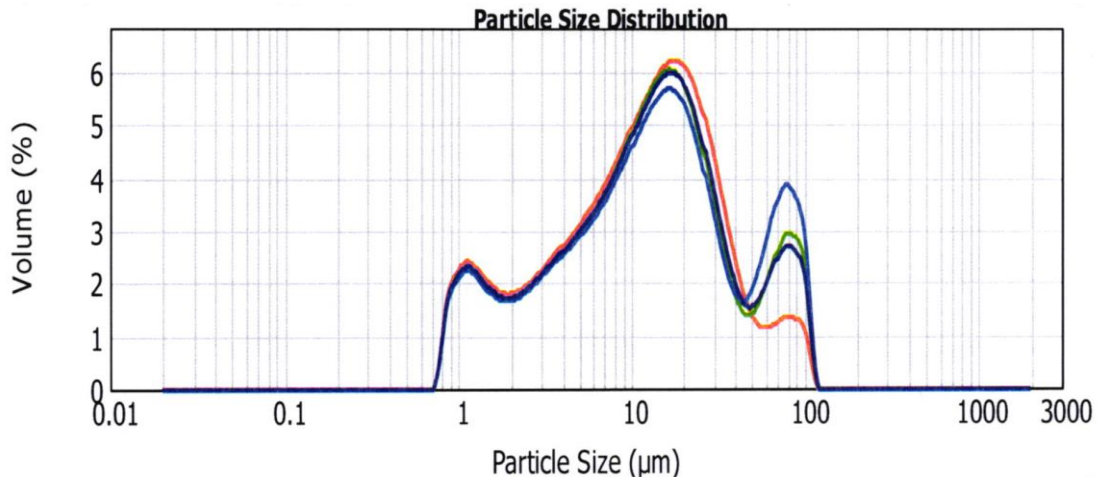
Samples	Surface weighted mean particle size (µm)	Volume weighted mean particle size (µm)	Polydispersity
A	24,274	104,096	0,807
B	18,889	87,373	0,666

Particle size distribution analysis of the solutions containing microcapsules coded as AA_m, BB_m, and CC_m is given in Figures 6.45-6.47. The values found when the mean particle size of particles in solution (microcapsules) are calculated as a function of the surface area and volume are given in Table 6.19.



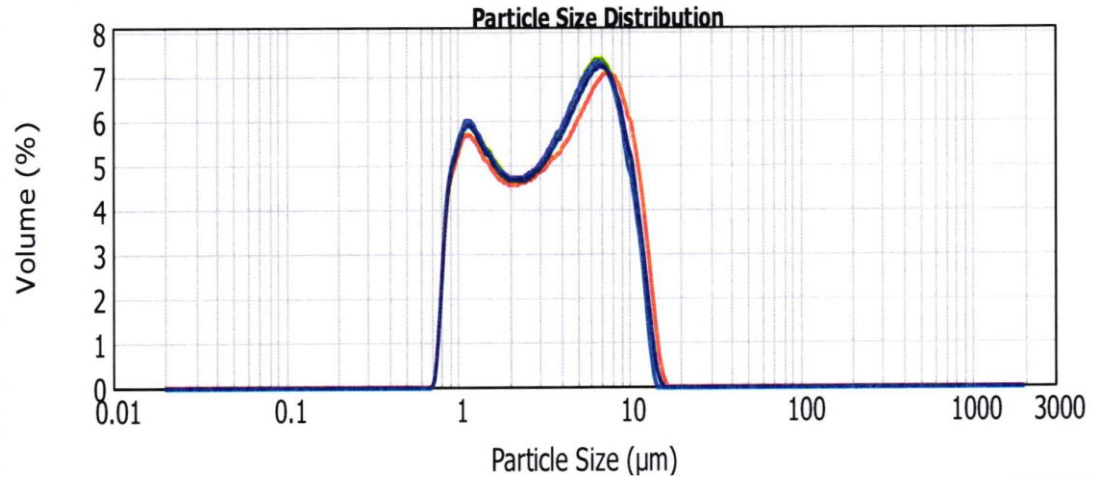
Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %
0.010	0.00	0.105	0.00	1.096	1.79	11.482	4.39	120.226	0.00	1258.925	0.00
0.011	0.00	0.120	0.00	1.259	1.60	13.183	4.93	138.038	0.00	1445.440	0.00
0.013	0.00	0.138	0.00	1.445	1.39	15.136	5.44	158.489	0.00	1659.587	0.00
0.015	0.00	0.158	0.00	1.660	1.24	17.378	5.86	181.970	0.00	1905.461	0.00
0.017	0.00	0.182	0.00	1.905	1.20	19.953	6.10	208.930	0.00	2187.762	0.00
0.020	0.00	0.209	0.00	2.188	1.25	22.909	6.07	239.883	0.00	2511.886	0.00
0.023	0.00	0.240	0.00	2.512	1.36	26.303	5.73	275.423	0.00	2884.032	0.00
0.026	0.00	0.275	0.00	2.884	1.52	30.200	5.07	316.228	0.00	3311.311	0.00
0.030	0.00	0.316	0.00	3.311	1.69	34.674	4.18	363.078	0.00	3801.894	0.00
0.035	0.00	0.363	0.00	3.802	1.87	39.811	3.23	416.869	0.00	4365.158	0.00
0.040	0.00	0.417	0.00	4.365	2.05	45.709	2.40	478.630	0.00	5011.872	0.00
0.046	0.00	0.479	0.00	5.012	2.24	52.481	1.83	549.541	0.00	5754.399	0.00
0.052	0.00	0.550	0.00	5.754	2.46	60.256	1.57	630.957	0.00	6606.934	0.00
0.060	0.00	0.631	0.00	6.607	2.72	69.183	1.56	724.436	0.00	7585.776	0.00
0.069	0.00	0.724	0.44	7.586	3.04	79.433	1.53	831.764	0.00	8709.636	0.00
0.079	0.00	0.832	1.46	8.710	3.43	91.201	1.42	954.993	0.00	10000.000	
0.091	0.00	0.955	1.72	10.000	3.89	104.713	0.35	1096.478	0.00		
0.105		1.096		11.482		120.226		1258.925			

Figure 6.45. Particle size analysis of AA_m coded microcapsule solution



Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %
0.010	0.00	0.105	0.00	1.096	2.08	11.482	4.90	120.226	0.00	1258.925	0.00
0.011	0.00	0.120	0.00	1.259	1.89	13.183	5.21	138.038	0.00	1445.440	0.00
0.013	0.00	0.138	0.00	1.445	1.68	15.136	5.38	158.489	0.00	1659.587	0.00
0.015	0.00	0.158	0.00	1.660	1.56	17.378	5.35	181.970	0.00	1905.461	0.00
0.017	0.00	0.182	0.00	1.905	1.56	19.953	5.05	208.930	0.00	2187.762	0.00
0.020	0.00	0.209	0.00	2.188	1.65	22.909	4.49	239.883	0.00	2511.886	0.00
0.023	0.00	0.240	0.00	2.512	1.80	26.303	3.73	275.423	0.00	2884.032	0.00
0.026	0.00	0.275	0.00	2.884	1.98	30.200	2.87	316.228	0.00	3311.311	0.00
0.030	0.00	0.316	0.00	3.311	2.18	34.674	2.09	363.078	0.00	3801.894	0.00
0.035	0.00	0.363	0.00	3.802	2.38	39.811	1.56	416.869	0.00	4365.158	0.00
0.040	0.00	0.417	0.00	4.365	2.59	45.709	1.39	478.630	0.00	5011.872	0.00
0.046	0.00	0.479	0.00	5.012	2.81	52.481	1.58	549.541	0.00	5754.399	0.00
0.052	0.00	0.550	0.00	5.754	3.07	60.256	1.99	630.957	0.00	6606.934	0.00
0.060	0.00	0.631	0.00	6.607	3.36	69.183	2.39	724.436	0.00	7585.776	0.00
0.069	0.00	0.724	0.51	7.586	3.71	79.433	2.39	831.764	0.00	8709.636	0.00
0.079	0.00	0.832	1.69	8.710	4.09	91.201	2.08	954.993	0.00	10000.000	
0.091	0.00	0.955	1.99	10.000	4.51	104.713	0.49	1096.478	0.00		
0.105		1.096		11.482		120.226		1258.925			

Figure 6.46. Particle size analysis of BB_m coded microcapsule solution



Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %
0.010	0.00	0.105	0.00	1.096	5.30	11.482	2.46	120.226	0.00	1258.925	0.00
0.011	0.00	0.120	0.00	1.259	5.01	13.183	0.59	138.038	0.00	1445.440	0.00
0.013	0.00	0.138	0.00	1.445	4.61	15.136	0.00	158.489	0.00	1659.587	0.00
0.015	0.00	0.158	0.00	1.660	4.32	17.378	0.00	181.970	0.00	1905.461	0.00
0.017	0.00	0.182	0.00	1.905	4.19	19.953	0.00	208.930	0.00	2187.762	0.00
0.020	0.00	0.209	0.00	2.188	4.18	22.909	0.00	239.883	0.00	2511.886	0.00
0.023	0.00	0.240	0.00	2.512	4.27	26.303	0.00	275.423	0.00	2884.032	0.00
0.026	0.00	0.275	0.00	2.884	4.47	30.200	0.00	316.228	0.00	3311.311	0.00
0.030	0.00	0.316	0.00	3.311	4.77	34.674	0.00	363.078	0.00	3801.894	0.00
0.035	0.00	0.363	0.00	3.802	5.15	39.811	0.00	416.869	0.00	4365.158	0.00
0.040	0.00	0.417	0.00	4.365	5.59	45.709	0.00	478.630	0.00	5011.872	0.00
0.046	0.00	0.479	0.00	5.012	6.03	52.481	0.00	549.541	0.00	5754.399	0.00
0.052	0.00	0.550	0.00	5.754	6.38	60.256	0.00	630.957	0.00	6606.934	0.00
0.060	0.00	0.631	0.00	6.607	6.48	69.183	0.00	724.436	0.00	7585.776	0.00
0.069	0.00	0.724	1.25	7.586	6.21	79.433	0.00	831.764	0.00	8709.636	0.00
0.079	0.00	0.832	4.14	8.710	5.46	91.201	0.00	954.993	0.00	10000.000	
0.091	0.00	0.955	4.95	10.000	4.16	104.713	0.00	1096.478	0.00		
0.105		1.096		11.482		120.226		1258.925			

Figure 6.47. Particle size analysis of CC_m coded microcapsule solution

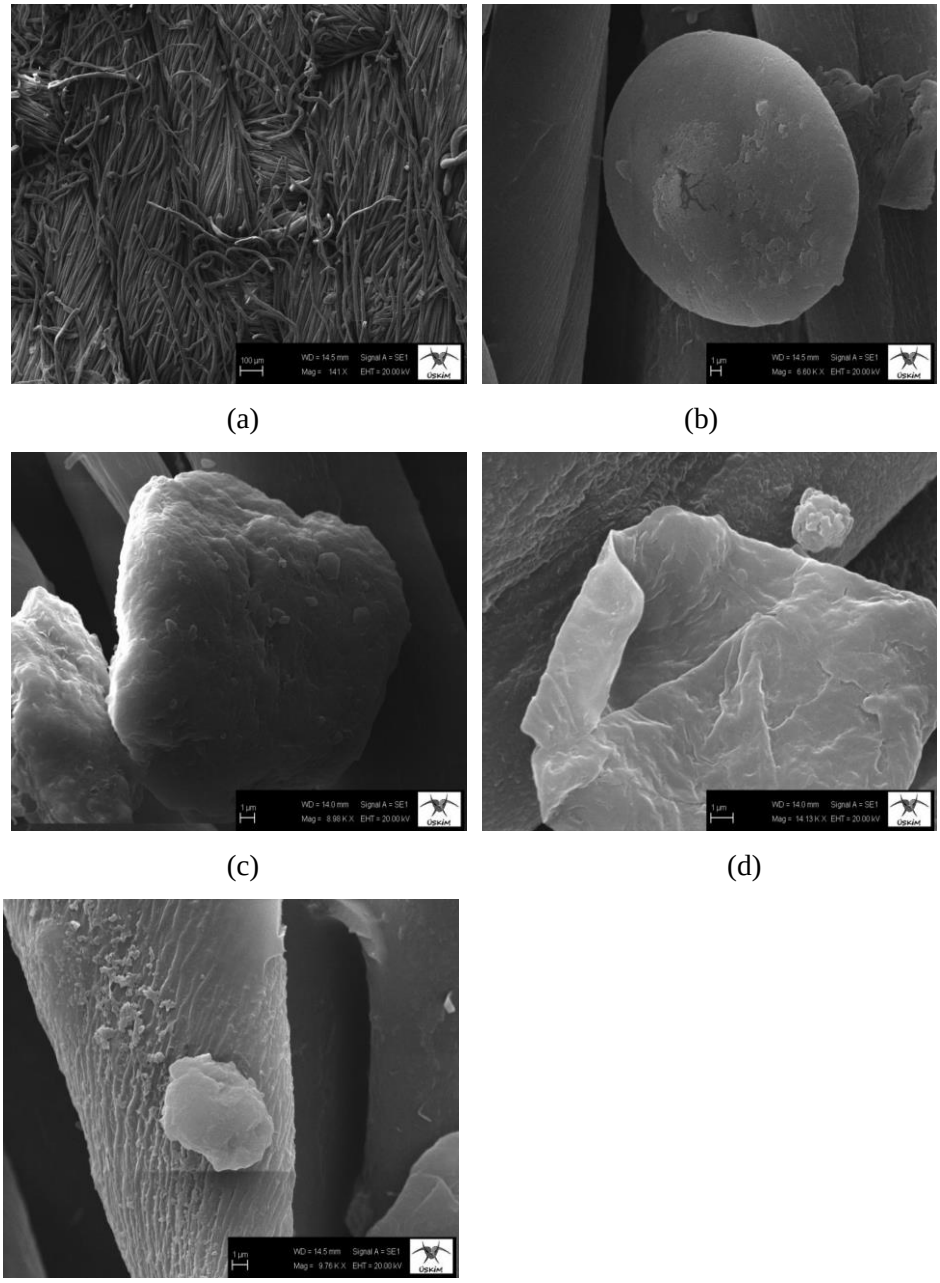
Table 6.19. Surface and volume weighted mean particle size and homogeneity values of AA_m, BB_m, and CC_m coded microcapsule solutions.

Samples	Surface weighted mean particle size (µm)	Volume weighted mean particle size (µm)	Polydispersity
AA_m	6,028	21,528	0,902
BB_m	5,194	20,405	1,2
CC_m	2,472	4,477	0,719

According to the particle size analyzes it can be said that, average particle size of microcapsule solutions produced with simple coacervation method is higher than those of produced according the microfluidic principle. Also, glycerin content in microencapsulation makes it clear that microfluidic shear forces have an effect in reducing particle size.

6.11. SEM Images

SEM images taken from denim fabric samples treated with microcapsule solutions containing *Aloe vera* gel are given in Figure 6.48. In Figure 6.48 a, b, and c microcapsules in different cross-sections are observed on the cotton fibers, while degradations on the outer phase (shell) and embedment of internal phase on the fiber surfaces can be seen in d and e.



(a) (b)

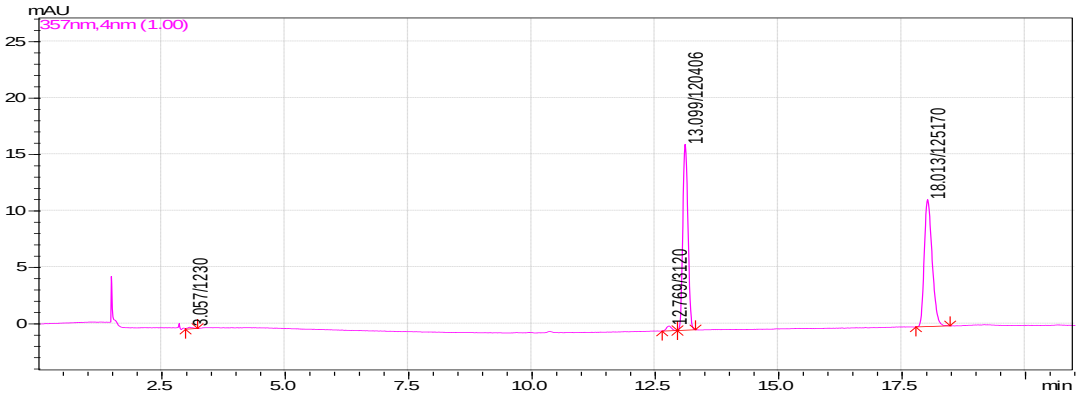
(c) (d)

(e)

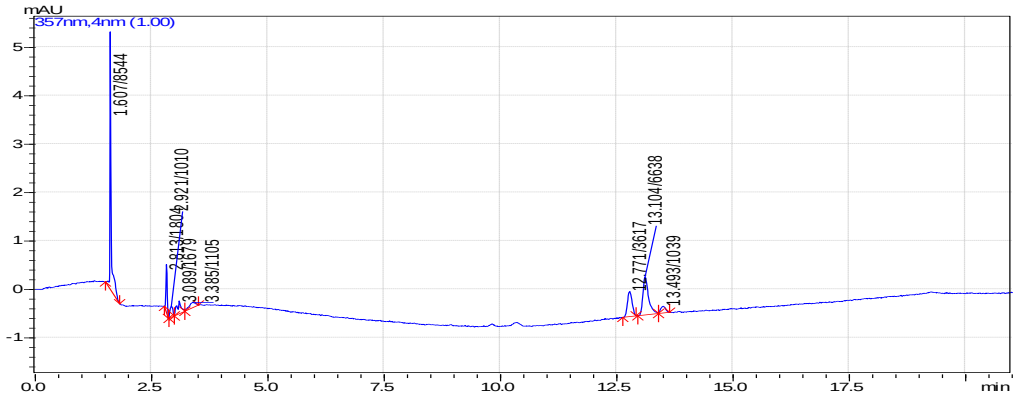
Figure 6.48. Sem images of denim fabric samples with *A. vera* microcapsules

6.12. HPLC Analysis

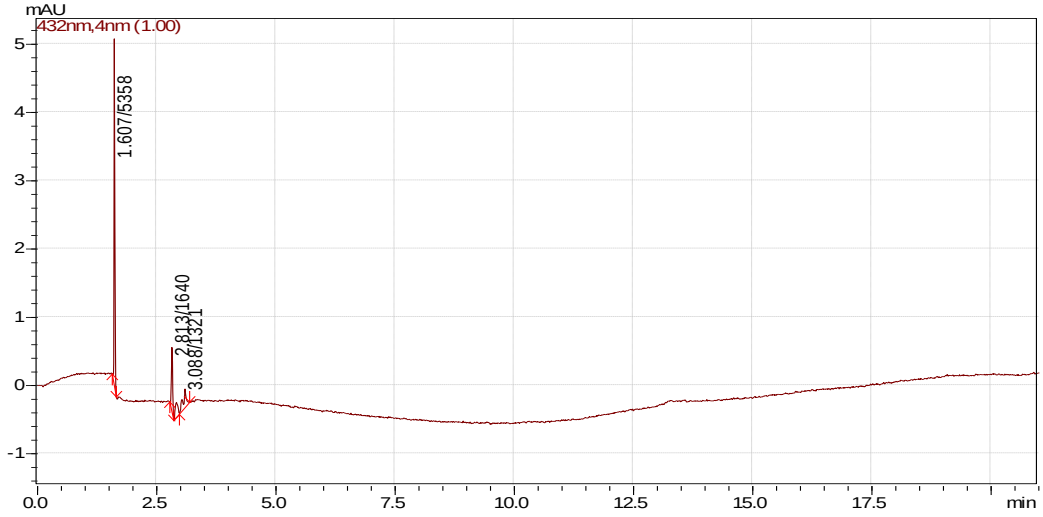
HPLC Analysis of Aloe vera leaf gel was made under the conditions given in section 5.11. The results were given below.



(a)



(b)



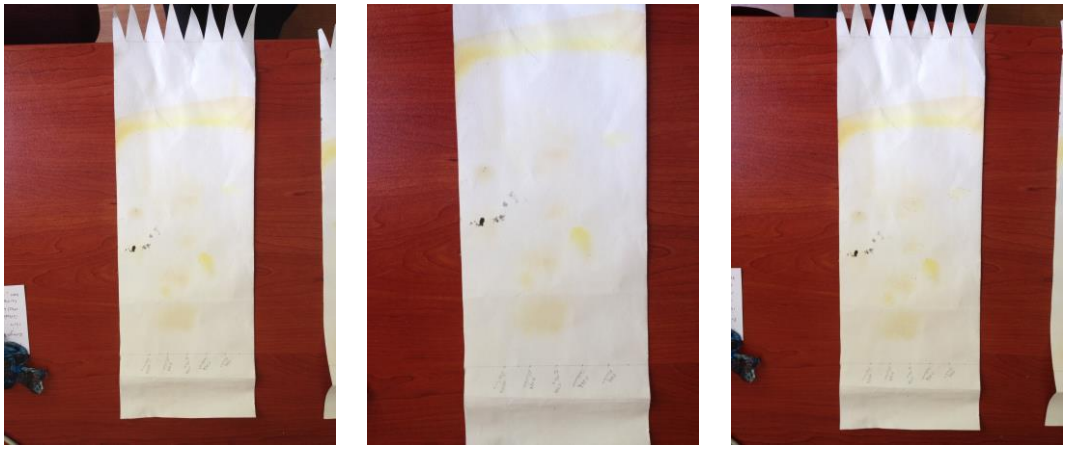
(c)

Figure 6.49. a) HPLC chromatography of aloin and aloe emodin standards injected at 10 μg/ml at optimum conditions, b) HPLC chromatography of aloin (357 nm) present in the *Aloe vera* gel at optimum conditions (qualitative method), c) HPLC chromatography of aloe emodin (432 nm) present in the *Aloe vera* gel at optimum conditions (qualitative method).

HPLC chromatograms indicate that the gel obtained from the *Aloe vera* plant leaves does not contain aloe emodin as shown in the chromatogram of (c) and contain low amount of aloin as shown in the chromatogram of (b). TLC (Thin Layer Chromatography) analysis show that, high antibacterial effectiveness of the gel is associated with the high content of polysaccharides and phenolic compounds when this value is much lower for anthraquinone compounds derived from aloin and aloe emodin. Because a high amount of transfer of anthraquinone compounds having laxative and purgative properties, from the leaf to the gel may cause a reduction in antibacterial/ antimicrobial effectiveness. Anthraquinone compounds may contribute antimicrobial effect only when they are present in the gel with a low degree.

6.13. Paper Chromatography

When the Rf values (drift distances) were marked on the chromatogram for sugars, it can be seen that the stains are observed at almost the same Rf values with those of saccharides / polysaccharides present in our sample. The important thing in this chromatogram is formation of a yellow-orange color from saccharides after spraying revelator and caramelization of them with heat. This Rf values and colors indicate the presence of saccharides / polysaccharides in our sample. The papers were shown in Figure 6.50.



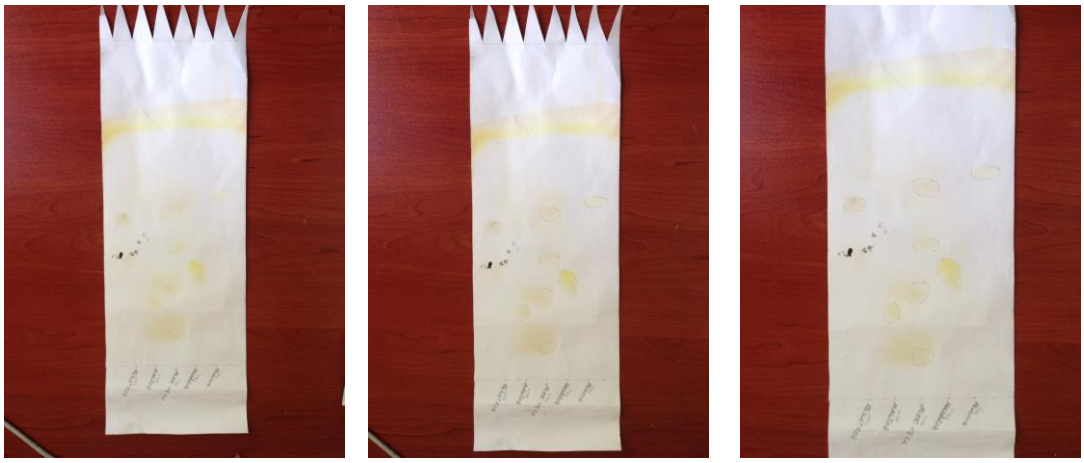


Figure 6.50. Paper chromatography of *Aloe vera* gel

6.14. Conclusion

Throughout this study *Aloe vera* leaf gel was examined for its antibacterial property. In order to utilize its antibacterial property, the gel was tried to encapsulate with simple coacervation, complex coacervation and microfluidic device. During microencapsulation *Aloe vera* gel (100% and diluted with distilled water at different ratios) was used as core material while Arabic gum and gelatin (only for complex coacervation) were used as wall materials. Although gabardine and denim fabrics were used at the beginning of the study, denim fabrics were preferred to be used for other stages. Fabric samples treated with microcapsule solutions were tested for their antibacterial effectiveness (before and after repeated washing cycles) and some physical properties such as abrasion resistance, tensile strength, water vapor permeability, dry and wet fastness (only for denim fabrics), and color differences properties.

- *Optical Microscope Images*

All microcapsule solutions were examined under optical microscope before application to the samples. The effects of changing some process parameters were observed on microcapsule dimensions, homogeneity, distribution, and efficiency etc. Increasing pH gradually results in smaller and more homogeneous capsules in size. Microcapsule solutions produced with microfluidic device contain smaller capsules when compared to both coacervation methods.

- *Antibacterial Effectiveness*

Antibacterial activity of treated samples were evaluated by quantitative test method ASTM E2149-01 for both *S.aureus* and *E. coli* bacteria. The samples showed very high (100%) antibacterial activity without washing and the maximum antibacterial activity against *E.coli* after 15 washing cycles was found to be about 90% which is a very pleasant value.

- *Abrasion Resistance*

Abrasion resistance test results show that, there is a negligible (< 5%) mass loss after 10000 rubbing cycles for all microencapsulation techniques used in this study. However, there is much more mass loss in the samples treated with microcapsule solutions prepared with microfluidic device.

- *Tensile Strength*

Tensile strength test results show that there is an increase in tensile strength of the samples in warp and weft direction after application of *Aloe vera* gel containing microcapsule solutions although it is not statistically significant. However, the samples treated with glycerin containing microcapsule solutions have a higher tensile strength values due to its plasticizer property. This can be explained by the installation of additional H bonds in the fabric structure.

- *Water Vapor Permeability*

Water vapor permeability test results show that there is an increase in water vapor permeability after application of *Aloe vera* gel microcapsules when 20, 50 and 100 gr/L of microcapsule solutions were prepared due to the increase in humidity content of the fabric. Microcapsule application slightly reduced water vapor permeability of the samples when they are completely used as finishing bath but this reduction is not significant commercially. In addition, there is a higher reduction in vapor permeability of the samples when they are treated with glycerin containing microcapsule solutions due to the film-forming property of glycerin on the fabric surface. The increase in both tensile strength and water vapor permeability show that microcapsules containing *Aloe vera* gel have moisturizing (wellness) effect on the denim fabric.

- *Dry and Wet Rubbing Fastness*

According to dry and wet rubbing fastness test results, it can be said that *Aloe vera* gel microcapsules have no negative impact on dry and wet rubbing fastness of denim

samples while there is a slight improvement in dry fastness. This improvement can be seen more clearly when the samples are treated with glycerin containing microcapsule solutions.

- *Color Difference*

When we look at spectrophotometric test results, it is clear that *Aloe vera* gel microcapsules do not lead yellowing for gabardine fabrics and do not cause an increase in brightness on the fabric surface. Namely, both denim and gabardine fabrics have acceptable color difference for all methods.

- *FTIR Analysis*

FTIR analysis of *Aloe vera* gel confirms that the gel comprise water and -OH groups of phenolic compounds.

- *TGA Analysis*

When TGA graphs are examined, it is possible to say that application of microcapsule solutions obtained with simple coacervation technique has not caused a negative impact on the thermal stability of the denim fabric. Moreover, there is an increase in thermal stability of denim fabrics when they are treated with microcapsule solutions produced with microfluidic device. This improvement is much more distinctive for glycerin containing samples.

- *DSC Analysis*

DSC analysis demonstrate that denim fabric treated with *Aloe vera* gel containing microcapsule solution has a higher thermal absorbency and thermal conductivity values. This proves that *Aloe vera* gel has a dampening effect (wellness) on the samples and it increases amount of humidity within the fabric. Because endothermic water rupture which has been observed around 385^0C in denim fabrics occurred with more thermal energy (greater shoulder peak) after treatment with microcapsules. The results also show that glycerin content in microencapsulation provide a limited heat absorption poperty to the samples. In addition, thermal conductivity values are higher for the samples treated with microcapsule solution having 1:5 core:wall ratio.

- *Particle Size Analysis*

According to the particle size analyzes it can be said that, average particle size of microcapsule solutions produced with simple coacervation method is higher than those of produced with microfluidic device. Also, glycerin content in

microencapsulation makes it clear that microfluidic shear forces have an impact in reducing particle size.

- *SEM Images*

SEM images confirm the microcapsule existence within the fabric structure although some capsules have degradations on wall surface.

- *HPLC- TLC Analysis and Paper Chromatography*

According to the chromatograms obtained after HPLC analysis, it can be said that *Aloe vera* leaf gel does not contain aloe emodin and contain low amount of aloin. Moreover, TLC analysis show that, high antibacterial effectiveness of the gel is due to the high content of polysaccharides and phenolic compounds when this value is much lower for anthraquinone compounds derived from aloin and aloe emodin. Because, anthraquinone compounds may contribute antimicrobial effect only when they are present in the gel with a low degree. The presence of saccharides / polysaccharides has been proven with paper chromatography.

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