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# Incorporation of Alginate/Chitosan Nano capsules Loaded with Sesame Oil or Omega-3 Oil in Cellulose Fabrics for Wound Healing Bandage

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#### Abstract

The present study focused on using the encapsulation technique to obtain functional fabric as a wound-healing bandage. First, the capsules are formed by using Emulsion solvent evaporation method using alginate and chitosan as shell materials and natural oils (Sesame oil and fish oil (OMEGA 3) as a core material. The effects of process parameters such as alginate: chitosan ratio, the effect of surfactant concentration, and the effect of oil concentration were investigated. The formed capsules have been characterized using a particle size analyzer, Transmission Electronic Microscopy (TEM), oil release study to determine their structure, and the optimal parameters were achieved by using alginate: chitosan (5:1), 2% surfactant concentration, and 10% oil concentration. The cellulosic fabric was then treated with capsules solution in the presence and absence of crosslinkers in three different ways. The treated cotton samples were characterized by scanning electron microscopy (SEM), oil release study, antimicrobial activity, and cytotoxicity assessment. It was found that the pad-dry-cure treatment gives the best homogeneous coated layer from capsules on the surface of the fabric and the presence of crosslinkers makes the oil release less than their absence.

Keywords: cellulose fabric, encapsulation, alginate, chitosan, sesame oil, fish oil, medical fabric.

# 1. Introduction

Nowadays, the importance of developing valueadded functional textile products increased in the textile market based on customer demand and due to increasing market possibilities and competition. Since the last decades, a lot of new finishing techniques have been developed to add functional properties to the fabric. In this context, micro-nanoencapsulation is one of these techniques which has been used to achieve the desired properties [1, 2]. Micro-nano encapsulation is an effective technique that enhances stability and controls the release properties of active ingredients (core material) by coating or surrounding them in polymeric material [3-5]. The polymeric shell materials could be synthetic or natural [6].

The rise in environmental concerns with the growing trend enhances the use of green chemistry and demands for environment-friendly processing of textiles [5]. So, in this paper the shell polymers used

were natural polymers consisting of alginate and chitosan and the core materials also are eco-friendly which are natural oils.

Alginate and chitosan are natural marine polymers extracted from brown seaweeds and crustacean shells respectively which have unique properties and structures [7]. The unique properties of alginate and chitosan polymers are attractive because of nontoxicity, biodegradability, and biocompatibility [8, 9]. Alginate is an anionic polymer naturally occurring, polysaccharides consist of linear chains of 1-4 linked alfa L-glycosidic acid and beta D-mannuronic acid arranged as blocks in the polymeric chain (see Figure 1) [8-10].

To enhance encapsulation efficiency and stability chitosan has been used with alginate due to electrostatic interaction between its amino groups and carboxyl groups present in alginate [8, 10]. Chitosan is a polysaccharide biopolymer that consists of

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copolymers linked by (1-4) D-glucosamine and Nacetyl-D-glucosamine units (see Figure 1) [8, 9, 11]. Chitosan is extracted from chitin by deacetylated [12]. The amount of acetylation on the amine group is a significant structural parameter for chitosan where chitin is a polymer with acetylation above 50 % while chitosan contains acetylation less than 50% [7]. The presence of amino groups makes it a cationic polymer, and this gives it exceptional biological and chemical characteristics like hydrophilicity, metal ion chelation ability, and gel-forming properties (see Figure 1) [12, 13]. Chitosan has antimicrobial activity versus a wide variety of organisms [14].

Essential oils have a particular interest because of numerous benefits like anti-inflammatory, antioxidant, insecticidal, emollient effect, healing, and anti-bacterial [15]. Essential oils consist of volatile and liquid aroma compounds extracted from often plant-sourced such as flowers and herbs [16, 17]. essential oils are eco-friendly and generally, so, they don't have any side effect arising out of using it [17].

In this study two essential oils (Sesame oil and OMEGA-3 oil) were used. Sesame oil is the oldest oil known and used by humans [18, 19]. Sesame oil has significant importance in the nutritional and medical fields [20]. It is better than other vegetable oils in resistance to oxidation [18]. It is used in many cosmetics, applications such as insecticides, pharmaceuticals, food industries, and fungicides [18, 19]. Sesame oil contains triacylglycerol, phospholipids, vitamin E, free fatty acids such as oleic acid, linoleic acid, palmitic acid. The presence of these components gives a unique health source to sesame oil [18-20]. OMEGA 3 oil's effect on disease management and prevention has been studied in the last few decades. OMEGA 3 oil showed proof of possibility in health benefits. The extraction from fish oil has large quantities of polyunsaturated fatty acids. Eicosapentaenoic acid and Docosahexaenoic acid are omega-3 fatty acid which is the dominant derived from fish oil [21].Omega-3 fatty acids are natural bioactive compounds with confirmed antiinflammatory effectiveness [22-25]. Omega-3 is a particular group of polyunsaturated fatty acids where the first twin covalent bond is between the third and fourth carbon atom counting from the methyl of the fatty acid [26].

One of the most common textile raw materials in the world is cellulose. Cellulose fiber is an important natural fiber that provides an enormous zone of applications in the textile field because of its unique properties [27]. Some of these applications is to make functional textiles that add an additional functionality over its ordinary function such as antibacterial [28-30], self-cleaning [31], UV protection [32], heat-[33-36], Temperature storing pН and thermoresponsive[37, 38], flameproofing [39, 40], water and oil repellent [41, 42], electrical conductivity [43], insect repellent [44], and bandage wounds [23, 45-50]. At present time there is a desire for smart bandage wounds which must recover the healing by interacting with the wound out of release bioactive molecules besides its basic mechanical protection [22].

The purpose of this study is to produce encapsulated particles formed from alginate and chitosan as shell materials and sesame oil or omega-3 oil as core material (which are meant to be used in wound healing) and use these capsules to functionalize cellulose textile substrates by different techniques to obtain an effective wound bandage.



Figure 1: chemical structure of alginate and chitosan

#### 2. Experimental

# 2.1. Materials

Cellulose gauze fabric (95  $g/m^2$ ) was purchased fromGhazl El-Mahala textile industry Co. Egypt.

Alginate was purchased from FlukaBioChemica GmbH Co. Chitosan (high molecular weight > 600000 mole/gram) and tween 80 were purchased from Alfa Chemika Co. India. Sesame oil from Emtnan Co. Egypt, OMEGA 3 oil (omega 3) from south Egypt industries Co. Egypt. Ethanol, sodium hypophosphite (SHP), butane tetracarboxylic acid (BTCA), acetic acid, and calcium chloride were used in laboratory-grade chemicals.

Hostapal CV (an anionic textile auxiliary based on alkyl aryl polyglycol ether) has been used as a nonionic detergent.

## 2.2. Method

# 2.2.1. Preparation of alginate/chitosan/oil matrix (Alg/Chito/oil).

The Alginate/chitosan (Alg/Chito) were prepared by ionic gelation according to the following steps: First, Alginate (Alg; 6 mg/mL) was dissolved in distilled water until a solution become clear following by the addition of CaCl<sub>2</sub> (8 mL; 0.67 mg/ml) to increase the viscosity of the solution (solution I). In addition, Chitosan (Chito, (6 mg/mL) was dissolved in 1% (v/v) acetic acid solution (solution II), while oil drugs (Sesame or OMEGA 3) were dissolved in ethanol (solution III). Next, solution I was added dropwise to solution II and stirred in an ultrasonic bath to form a uniform solution. Thereafter, solution III was poured into the mixture of solution I and solution II and then ultrasonicated five times each for 5 mins. Finally, the mixed solution was centrifuged at 4°C before lyophilization.

## 2.2.2. Optimization of process parameters

Several parameters involved in the production of encapsulated oil in alginate/chitosan matrix were varied to conclude the best parameters between them. Optimization of alginate/chitosan matrix loaded with sesame oil or omega-3 oil was achieved by varying the ratio between alginate and chitosan combination (1:5, 2:4, 3:3, 4:2, and 5:1), varying the concentration of tween 80 (1, 2, 3, and 4 %) and oil (5, 10, 15, 20 %) and finally varying the fabric treatment method.

### 2.2.3. Functionalization of textile substrates

The gauze cellulose fabric was functionalized with alginate/chitosan matrix loaded with sesame oil or OMEGA 3 oil by impregnation technique as follow: butane tetracarboxylic acid (BTCA, 5%) and sodium hypophosphite (SHP, 3%) were added to the alginate/chitosan/oil matrix solution as crosslinkers. the fabrics were immersed in this formulation for 15 minutes. Then pressed with 100% wet pick up. Various patching methods were used to treat fabric, (i) first one the pad-wet patch, in this method each fabric was wrapped in a plastic bag after padding and left at room temperature to dry, (ii) second one is the pad-dry method, in this method each fabric was dried at 70°C after padding for 3 minutes, and (iii) third one is the pad-dry-cure method, in this method the padded fabrics was warped and dried at 100°C for 15 minutes, then cured at 145°C for 3 min. The treated fabrics were used for further examination.

#### 2.3. Analysis and Measurements

#### 2.3.1. Particle size investigation

A particle size analyzer was used to determine the nanoparticles' mean diameter and size dispersion (Malvern Instruments Ltd.). The morphological structure of the prepared alginate/chitosan matrix was observed through transmission electron microscopy (TEM) on a JEOL (JEM-1230) Japan, an instrument with an acceleration voltage of 120 kV.

#### 2.3.2. Fabric morphology characterization

Scanning electronic microscopy was used to examine the treated textiles' microscopic properties (SEM). With a scanning electron microscope, the fabrics were coated with a 20 nm thick gold coating and examined and photographed (Quanta 250 FEG)

#### 2.3.3. Release study

In the beginning, the standard calibration curves for both oils was measured at different oil concentrations (0.5, 1, 5, 10, 20, 30, and 40 mg/mL) and subjected to UV spectrophotometer for measuring the absorbance at the highest peak. After that, the calibration curve was obtained as illustrated in **Figure 2**.

Release study of the encapsulated oils from alginate/chitosan matrix or treated gauze treated fabric were investigated as follows: 1 g of encapsulated oils in alginate/chitosan matrix was immersed in 100 ml of phosphate buffer saline (PBS), pH 7.5 at 37°C, and placed and kept on a simple shaker (multi Mix MTR-22) with a specific program for shaking and rotation. Over 6 hr at predetermined time intervals (15, 30, 60, 120, 180, 240, 300, and 360 min), 5 ml aliquots were withdrawn, and the concentration of oil released was monitored by a UV-vis spectrophotometer (carry 100). After measurement mina was replaced with a fresh solution to keep the total volume constant. The oil release percent can be determined by the following equation: **[51]** 

Oil release [%] =  $\frac{C_t}{C_o} \times 100$ 

where  $C_o$  and  $C_t$  represent the initially loaded oil and the amount of oil released at time t, respectively. All studies were performed in triplicate.



Figure 2: Standard calibration curve for sesame oil and OMEGA 3 oil at a different known concentration

### 2.3.4. Antimicrobial assessment

Antimicrobial activity was assessed qualitatively and quantitatively against Gram-positive bacteria, Gram-negative bacteria, and fungi (S. aureus (ATCC 29213), E. coli (ATCC 25922), and Candida albicans (ATCC 10231).

The method of disc diffusion to analyze the antimicrobial activity of treated fabrics has been used through the AATCC Test Method (147-2016). [38, 39, 52, 53] Squares of 1 cm<sup>2</sup> of each fabric are prepared in an aseptic manner to test the antibacterial properties of the treated fabrics as Bershteein et al. [54] by testing the diameter of the inhibition zone (r) [55]. The 0.5 ml from suspension agar (45 ml/L Nutrient agar) was put in a petri-dish with 100 µL of each examined bacterium and the petri-dish was seeded and solidified and then pieces of treated fabrics squares were put in the plate. The test plates incubate at 37°C for 24 h. the diameter of the inhibition zone (r) was measured in millimeters, then the area of inhibition zones was calculated according to the following equation

area of the zone inhibition  $(mm^2) =$ 

total area - fabric area

Antimicrobial activity was assessed quantitatively using the AATCC textile standards method (AATCC 100-2004; bacterial reduction method) [56]. Each cloth was produced aseptically in a square  $(1 \times 1 \text{ cm})$ . Each square was put in a sterile vial, and the textiles were pre-treated for 10 minutes in 800 mL distilled water. Then, to make up a total amount of 3 ml, tryptone soy broth (2.2 ml) was added to each vial. Each vial holding the textiles received an aliquot (10 ml,  $1.6 \times 10^3$  ml) of microbe solution. There were also control broths with and without microbial injection. The vials were then incubated at 35°C, 220 rpm, with agitation. At 24 hours, aliquots of 10 mL broth were sampled, and serial dilution in broth was made for the aliquots. On the plates were distributed duplicate aliquots (50 ml) of the serially diluted samples. Bacterial counts were obtained after the plates were incubated at 35°C. After 24 hours, the bacteriostatic activity was evaluated, and the percent decrease of tiny organisms was determined using the following formula:

$$R\% = \frac{A-B}{A} \times 100$$

where R = the reduction rate, A and B are the number of bacterial colonies from untreated and treated fabrics, respectively.

The untreated/treated fabrics with the preceding colloidal solutions were quantitatively assessed before and after 10 washing cycles to see how durable the treated fabrics were against the microbiological activity. Warm water with a 0.5 g/l

non-ionic wetting agent was also used for washing. Each wash takes about 30 minutes and is followed by a  $70^{\circ}$ C dry.

## 2.3.5. Cytotoxicity assessment

This was done using the method given by Nada et al. and a co-worker [23, 24, 46]. Cells were kept in Dulbecco's modified eagle medium (DMEM): F12 Medium (nutrient mixture)/10 percent fetal bovine serum (FBS) and incubated at 37°C in 5% CO<sub>2</sub> and 95% humidity in a usual method. Cells from the skin normal human cell line (BJ-1) cells were seeded at a density of 30000 cells per well in a 96-well plate. After a 48-hour incubation period, the culture medium was replaced with extracted media containing various concentrations of the tested compounds (ranging from 20000 to 100 ppm (g/ml)). Before extraction, samples were sterilized in a laminar flow for 20 minutes under ultraviolet (UV) light. As a control, cell culture media without any extra reagents was utilized. The dish was then incubated for another 24 hours. The MTT assay was used to assess the number of live cells. The culture medium was removed and 40 ul of MTT solution (2.5 mg/ml) was added in its stead. The solution was then incubated at 37°C for 4 hours. To dissolve the formazan crystals, 200 µl of 10% sodium dodecyl sulfate (SDS) in deionized water was added to each well and incubated overnight at 37°C. The absorbance was measured at 595 nm using a microplate multi-well reader. The following equation was used to calculate cell viability:

viability of cells =  $\frac{average(X)}{average(NC)} \times 100$ 

where: NC: absorbance of negative control, X: absorbance of the sample. the absorbance was measured at 595 nm.

## 2.3.6. Mechanical properties of the treated fabric

Tensile strength and elongation must be measured at a temperature of 25°C and relative humidity of 65 % using the tensile strength apparatus FMCW 500 (VebThuringerIndustrieWerkRauenste in 11/2612 Germany) following ASTM test method D5035. [57]The crease recovery angle (CRA) was assessed using the AATCC test method 66 - 2014. [58] The ASTM test system D 7127–13 was used to measure the roughness of the treated textiles using the surface roughness instrument SE 1700.[59] A cantilever device was used to evaluate the stiffness of the treated textiles following ASTM test D 1388-14e1. [60, 61]

#### 3. Results and Discussion

Linolenic acid (found in plant oils), eicosapentaenoic acid, and docosahexaenoic acid (both commonly found in fish oil) are the three kinds of important fatty acids in omega-3. Glycerides of oleic acid (36–54 percent) and linoleic acid (38–49 percent) make up 83–90 percent of the unsaturated fatty acids in sesame oil. Saturated fatty acids (myristic acid, 0.1 percent or less; palmitic acid, 8– 12%; stearic acid, 3.5–7%; arachidonic acid, 0.5–1%) are other fatty acid components. **[62]** 

Both oils were encapsulated in an alginate/chitosan matrix to control their release profile to use in the medical field as a result of their antibacterial properties. The ratio between alginate and chitosan, the concentration of tween, and oil concentration have been investigated to optimize the best parameter for encapsulation and release profile.

## 3.1. Optimization the Alginate/Chitosan/Oil matrix

## 3.1.1. Effect of Alginate: Chitosan ratio

The particle size analyzer was used to measure the mean particle sizes of the produced emulsions, and the findings are shown in

**Figure 3**: Particle size diagram of prepared emulsions from alginate/chitosan/oil matrix and the data are listed in **Effect of** surfactant concentration

Over 6 hours in phosphate buffer solution (pH 7.5), the release patterns of free oils from alginate/chitosan (5:1) microparticles with various tween concentrations (1, 2, 3, and 4 %) were studied (Figure 6). In general, the amount of both oils released in phosphate buffer was decreased as the tween concentration increased. In addition, increasing the amount of tween more than 2% provided a slight decrease in the oil released from the alginate/chitosan matrix. This phenomenon may be due to increasing the amount of tween decrease the surface tension inside the polymer matrix and causing an expansion in the polymer molecules and decreasing the hydrogen bonding between oil the and alginate/chitosan, which didn't provide more encapsulation efficiency.

## 3.1.2. Effect of oil concentration

The release patterns of free oils (5, 10, 20, and 30 %) from alginate/chitosan (5:1) microparticles with 2% tween concentrations were investigated over 6 hours in phosphate buffer solution (pH 7.5) and illustrated in **Figure 7.** As the oil content increased more than 10 %, the amount of both oils released in

phosphate buffer is almost the same. Furthermore, increasing the quantity of oil by more than 10% reduced the amount of oil produced from the alginate/chitosan matrix. This effect might be caused by increasing the quantity of oil in the polymer

. The particles diameter of chitosan solution at d50 is 76.79 and 840.07 $\mu$ m while the diameter of the particles of alginate solution at d50 is 630.95 and 1101.32  $\mu$ m, for encapsulated sesame oil and OMEGA 3 oil respectively.

From the obtained results, it is clear to observed that, alginate/chitosan matrix led to a decrease in the particle size of oil within the range of microemulsions. In addition, decreasing the amount of alginate in the matrix led to decreasing the particle size diameter of the produced emulsion.

The uniformity values of particle diameter in an emulsion are also given in **Effect of** surfactant concentration

Over 6 hours in phosphate buffer solution (pH 7.5), the release patterns of free oils from alginate/chitosan (5:1) microparticles with various tween concentrations (1, 2, 3, and 4 %) were studied (**Figure 6**). In general, the amount of both oils released in phosphate buffer was decreased as the tween concentration increased. In addition, increasing the amount of tween more than 2% provided a slight decrease in the oil released from the alginate/chitosan matrix. This phenomenon may be due to increasing . The uniformity of emulsion particle size decreases as the amount of chitosan increased. **[63]** 

D[4,3] is one of the designations for the volume mean diameter. When the result is presented as a volume distribution, it is simply referred to as the "mean". When the result is transformed to a surface area distribution, the surface means D[3,2], is presented as the mean value. The surface means equation is presented below (following the conventions from ASTM E 799). [64]

$$D[4,3] = \frac{\sum_{1}^{n} D_{ivi}^{4}}{\sum_{1}^{n} D_{ivi}^{3}} \qquad D[3,2] = \frac{\sum_{1}^{n} D_{ivi}^{3}}{\sum_{1}^{n} D_{ivi}^{2}}$$

Where: D = the particle diameter, D [3,2] = volume/surface mean (also called the Sauter mean), means the ratio of the d(30) & d(20) and D[4,3] = the mean diameter over volume (also called the de Brouckere mean), means the ratio of the d(40) & d(30).

From the data in Effect of surfactant concentration

Over 6 hours in phosphate buffer solution (pH 7.5), the release patterns of free oils from alginate/chitosan (5:1) microparticles with various tween concentrations (1, 2, 3, and 4 %) were studied

matrix, which causes stretching in the polymer molecules network and the hydrogen bonding between the oil and alginate/chitosan was increased, resulting in more impregnating in the network and less release to the surrounding media.

the amount of tween decrease the surface tension inside the polymer matrix and causing an expansion in the polymer molecules and decreasing the hydrogen bonding between the oil and alginate/chitosan, which didn't provide more encapsulation efficiency.

# 3.1.3. Effect of oil concentration

The release patterns of free oils (5, 10, 20, and 30 %) from alginate/chitosan (5:1) microparticles with 2% tween concentrations were investigated over 6 hours in phosphate buffer solution (pH 7.5) and illustrated in Figure 7. As the oil content increased more than 10 %, the amount of both oils released in phosphate buffer is almost the same. Furthermore, increasing the quantity of oil by more than 10% reduced the amount of oil produced from the alginate/chitosan matrix. This effect might be caused by increasing the quantity of oil in the polymer matrix, which causes stretching in the polymer molecules network and the hydrogen bonding between the oil and alginate/chitosan was increased, resulting in more impregnating in the network and less release to the surrounding media.

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# 3.1.4. Effect of oil concentration

The release patterns of free oils (5, 10, 20, and 30 %) from alginate/chitosan (5:1) microparticles with 2% tween concentrations were investigated over 6 hours in phosphate buffer solution (pH 7.5) and illustrated in **Figure 7.** As the oil content increased more than 10 %, the amount of both oils released in phosphate buffer is almost the same. Furthermore, increasing the quantity of oil by more than 10% reduced the amount of oil produced from the

alginate/chitosan matrix. This effect might be caused by increasing the quantity of oil in the polymer matrix, which causes stretching in the polymer molecules network and the hydrogen bonding , it is clear that the decrease in particle size was confirmed as the D[4,3] and the D[2,3] decreased with an increase in the specific surface area. Further notification that, alginate/chitosan/sesame oil suspension matrix provides a smaller particle size than the alginate/chitosan/OMEGA 3 oil suspension matrix.



Figure 3: Particle size diagram of prepared emulsions from alginate/chitosan/oil matrix

The existence of synthesized encapsulated oil in Alginate/chitosan matrix with different ratios was

between the oil and alginate/chitosan was increased, resulting in more impregnating in the network and less release to the surrounding media.

verified by transmission electron microscope (TEM), which revealed the morphology of typical Sesame oil-loaded Alginate/chitosan. Particles were found to be spherical, distinct, and regular using a transmission electron microscope (**Figure 4**), with particle diameters ranging from 20 to 50 nm depending on the ratio between alginate and chitosan **[65, 66]**. On the other hand, when the ratio between alginate and chitosan becomes (5:1), the particles had a fluffy look rather than a smooth surface with good distribution.

Free oils (10 %) release patterns from alginate/chitosan microparticles with the different ratio in presence of tween (2%) were measured over 6 hours in phosphate buffer solution (pH 7.5) (**Figure 5**). In general, the amount of OMEGA 3 oil released was greater than sesame oil released in phosphate buffer. This phenomenon might be explained by the fact that the OMEG 3 oil particles size is greater than sesame oil. Which allows the polymer matrix to more expanded, allowing the OMEGA 3 to dissociate.

In phosphate buffer, however, the ionic composition of the surrounding media caused the sponge-like structure to collapse, preventing the components from being released further. An anionic medication was placed into a polycationic–polyanionic polymer matrix to create microparticles. Because particulate systems have a high ionic content, the polymeric structure is likely to pack into a more compact shape in settings with greater ionic strengths.

	D [4, 3] -	D [4, 3] - Specific		D [3, 2] -			1		
	Volume	Uniformity	surface	Surface	d (0.1)	d (0.5)	d (0.9)		
	weighted mean	-	area	weighted mean					
Sesame Oil									
Chitosan	176.06	1.98	0.37	16.31	5.63	76.79	477.34		
01:05	7.44	1.96	2.58	2.33	1.07	2.94	13.57		
02:04	21.36	5.04	2.34	2.56	0.98	3.88	59.81		
03:03	14.40	3.50	2.46	2.44	1.03	3.41	36.69		
04:02	36.48	0.56	0.32	19.05	10.51	31.89	68.96		
05:01	312.30	0.66	0.03	214.61	139.32	217.82	412.97		
Alginate	657.65	0.74	0.54	112.06	145.32	630.95	1031.24		
OMEGA 3 oil									
Chitosan	703.71	1.49	0.33	18.12	8.57	840.07	1281.40		
01:05	98.04	1.22	0.42	14.35	7.28	26.16	115.71		
02:04	135.47	1.84	0.23	26.49	13.30	58.57	361.12		
03:03	338.36	9.87	0.60	10.01	3.77	82.80	971.82		
04:02	312.30	0.66	0.03	214.61	139.32	217.82	412.97		
05:01	258.86	0.64	0.51	11.69	3.82	283.64	534.31		
Alginate	1128.46	0.21	0.01	1059.08	787.04	1101.32	1509.49		

Table 1: Hydrodynamic diameter of prepared emulsions from alginate/chitosan/oil matrix

Therefore, it is clear that the release profile for both oils in chitosan or alginate only provides a high oil release at the first hour and almost completely release after 3 hours, while encapsulation of both oils in the Alginate/chitosan matrix provides a slow release for oil from the matrix. In addition, alginate/chitosan with a 5: 1 ratio provides the low release profile for both oil which confirmed the good encapsulation of oil inside this matrix than the others.



**Figure 4**: TEM image of Sesame oil in Alginate/Chitosan matrix a) Chitosan, b) Alginate/Chitosan (1:5), c) Alginate/Chitosan (2:4), d) Alginate/Chitosan (3:3), e) Alginate/Chitosan (4:2) f) Alginate/Chitosan (5:1), g) alginate



Figure 5: The release profiles of sesame oil and OMEGA 3 from Alginate/Chitosan matrix with different ratio

#### 3.1.5. Effect of surfactant concentration

Over 6 hours in phosphate buffer solution (pH 7.5), the release patterns of free oils from alginate/chitosan (5:1) microparticles with various tween concentrations (1, 2, 3, and 4 %) were studied (**Figure 6**). In general, the amount of both oils released in phosphate buffer was decreased as the tween concentration increased. In addition, increasing the amount of tween more than 2% provided a slight decrease in the oil released from the alginate/chitosan matrix. This phenomenon may be due to increasing the amount of tween decrease the surface tension inside the polymer matrix and causing an expansion

in the polymer molecules and decreasing the hydrogen bonding between the oil and alginate/chitosan, which didn't provide more encapsulation efficiency.

# 3.1.6. Effect of oil concentration

The release patterns of free oils (5, 10, 20, and 30 %) from alginate/chitosan (5:1) microparticles with 2% tween concentrations were investigated over 6 hours in phosphate buffer solution (pH 7.5) and illustrated in **Figure 7**. As the oil content increased more than 10 %, the amount of both oils released in phosphate buffer is almost the same. Furthermore, increasing the quantity of oil by more than 10%

reduced the amount of oil produced from the alginate/chitosan matrix. This effect might be caused by increasing the quantity of oil in the polymer matrix, which causes stretching in the polymer



**Figure 6**: The release profiles of Sesame oil and OMEGA 3 from Alginate/Chitosan matrix with different tween concentration

#### 3.2. Characterization of the treatment technique

The gauze cotton fabric was treated with three distinct techniques: pad-wet, pad-dry, and pad-dry cure. **Figure 8** shows that treated gauze cotton textiles using the pad-dry-cure approach produce a homogeneous coated layer from alginate/chitosan/ oil on the fabric's surface better than the other two techniques. This is due to the presence of a crosslinker in the treatment formula, which works well to form a cross-linkage network between the fabric and the alginate/chitosan composite during the curing step, allowing chemical bonding to form and

molecules network and the hydrogen bonding between the oil and alginate/chitosan was increased, resulting in more impregnating in the network and less release to the surrounding media.

providing well-distributed oil within a chemically bonded network. Furthermore, the pad wet method provides an agglomeration of the composite on the surface of the fabric, whereas the pad-dry method provides an incomplete film on the surface due to the incomplete reaction between BTCA and composite in the presence of SHP, which requires 140°C for an excellent working according to the suggested mechanism in **Figure 9**.



**Figure 7**: The release profiles of Sesame oil and OMEGA 3 with different oil concentrations from Alginate/Chitosan matrix



Figure 8: SEM image for treated gauze cotton fabric with encapsulated Sesame oil and OMEGA 3 oil using different techniques in presence of BTCA as a crosslinker



c) treated with sesame oil using pad-dry

b) treated with OMEGA 3 oil using pad wetd) treated with OMEGA 3 oil using pad-dryf) treated with OMEGA 3 oil using pad-dry-cure



Figure 9: Suggesting mechanism for the reaction between the cotton fabric and alginate/chitosan

# *3.3. Evaluation of the treated gauze cotton fabric using the pad-dry-cure technique*

## 3.3.1. Morphological behavior

The alginate/chitosan layer on the cellulose surface showed a homogeneous structure and revealed a uniform distribution of the components on the fabric surface as predicted in **Figure 10a**. Sesame oil and omega 3 oil droplets generated in the alginate/chitosan matrix and coated on the cellulose surface, both films appeared homogeneous, as observed in **Figure 10b and c**. Presence of chitosan may lower network density by causing significantly entangled porous sizes and disrupting stable threedimensional polymer networks [67].

In contrast to treated fabric using Alginate/Chitosan/Sesame oil matrix, we found that treated fabric using Alginate/Chitosan/OMEGA 3 oil matrix had superior droplet size distribution. Probably because the films included more OMEGA 3 oil, because lowering the size of droplets lowers the interfacial tension between the oil (nonpolar) and the Alginate/Chitosan chains (polar). **[68]** 

e) treated with sesame oil using pad-dry-cure



Figure 10: SEM image of treated fabric with Alginate/Chitosan/oil a) treated fabric with Alginate/Chitosan b) treated fabric with Alginate/Chitosan/Sesame oil matrix c) treated fabric with Alginate/Chitosan/OMEGA 3 matrix

#### 3.3.2. Physical and Mechanical properties

Despite the differences in the size of the oil droplets visible in films, adding either oil to the basic polymer matrix reduces the homogeneity of the component distribution. However, this shift in component distribution might impart features that contribute to the final formed film on the surface.

The mechanical and physical properties such as tensile strength, elongation at break, air permeability, crease recovery angle, and surface roughness, of treated gauze cotton fabrics with alginate/chitosan and both oils in the presence and absence of BTCA as a crosslinker, were measured and recorded in **Error! Reference source not found.** 

The crease recovery angle (CRA) increases substantially when the surface roughness, air permeability, tensile strength, and elongation at break decrease following each treatment, as shown in **Error! Reference source not found.** This indicates that the studied alginate/chitosan matrix was thoroughly absorbed into the microstructure of cotton fiber and that a thin film from the biopolymer employed, which formed on the cotton's surface, was responsible for these changes. **[25, 69-71]**The mechanical characteristics of the deposited film, such as roughness, air permeability, tensile strength, and elongation at break values, are all reduced.

A crosslinker butane tetracarboxylic acid (BTCA) and sodium hypophosphite in the treatment bath was also important in evaluating the usefulness of the above-mentioned characteristics (SHP). During this pre-treatment, the development of covalent crosslinking connections among adjacent cellulose chains would add stiffness to the cotton structure. Meanwhile, the BTCA acid and reaction would destroy the cotton fiber's cellulose chain chemically.

The crease recovery angle for treated gauze cotton fabrics in both the warp and weft directions was investigated further, and the results show that the crease recovery angle of all treated fabrics in the presence of biopolymers has increased values when compared to untreated fabrics or treated fabrics in the absence of biopolymer materials. These findings confirm that biopolymer materials play an important role in enhancing the mechanical or physical properties of fabrics. The development of an extreme cotton structure network densely cross-linked by covalent chemical connections between the cellulose chain and biopolymers may have played a major role in improving the crease recovery angle. **[25, 69-71]** 

Oil	Crosslinker	R (µm)	Tensile strength	Elongation at a break (%)	CRA (W+F°)	Air permeability (cm <sup>3</sup> /cm <sup>2</sup> /s)	
Blank fabric		21.45	152.12	39.21	198.65	221.87	
Control fabric without oil		21.41	147.77	38.46	204.52	221.30	
Sesame oil	without BTCA	21.37	143.42	37.70	210.39	220.73	
	with BTCA	21.29	134.49	36.10	216.95	219.89	
OMEG 3 oil	without BTCA	21.33	138.95	36.90	223.52	219.06	
	with BTCA	21.42	138.39	35.20	215.28	221.22	

**Table 2**: Physical and mechanical properties for treated fabrics with sesame oil and OMEGA 3 oil in the presence and absence of crosslinker

#### 3.3.3. Release study

A bandage from gauze-treated fabric with and without crosslinker (BTCA) has been produced with a core of treated cotton filament. The oil-releasing performance of the final produced bandage was examined.

**Figure 11** illustrates the cumulative release curves of both oils (sesame oil and OMEGA 3 oil) encapsulated in alginate/chitosan (5:1) from treated cotton gauze fabric in the presence and absence of butane tetracarboxylic acid (BTCA) as a crosslinker over 6 hours in phosphate buffer solution (pH 7.5). After one hour, both oils were released from cotton gauze fabric with 58.61 and 31.44 % in absence of BTCA, while in presence of BTCA the released oil percent was decreased to be 48.52 and 17.35 % for sesame oil and OMEGA 3 oil respectively as illustrated in **Error! Reference source not found.**. After 6 hours, the released oils were increased to be 77.87 and 71.30 %% in absence of BTCA, while in presence of BTCA the released oil percent was

**Table 4** summarizes the quantitative antibacterial findings before and after the 10 washing cycles. Fabrics treated with alginate/chitosan (control sample) without both examined oils exhibit antimicrobial resistance for examined microbes E. Coli, St. Aureus, and C. Albicans better than blank fabric.

Furthermore, fabrics treated with both oils encapsulated in alginate/chitosan matrix in presence of BTCA as crosslinker exhibit antimicrobial resistance with good values with OMEGA 3 oil than sesame oil for examined microbes E. Coli, St. Aureus, and C. Albicans than in absence of BTCA. This was also clear by investigating the microbial activity after 10 washing cycles which provide highly decrease in the reduction percent in the case of treated fabric in absence of BTCA. As a result of the temporary deposit of the alginate/chitosan/oil matrix without chemical combination with the fabric decreased to be 73.56 and 65.18 % for sesame oil and OMEGA 3 oil respectively.

As indicated in the release curve, The release of sesame oil from alginate/chitosan is much faster than that of OMEGA 3 oil incubated in phosphate buffer (pH 7.5). The release results appear to indicate that a significant amount of oil remained on the alginate/chitosan surfaces due to weak interactions forces between polyelectrolytes and oil.

On the other hand, the oil contained in the matrix had to overcome an extra physical barrier. The decreased electrostatic interactions between the polysaccharide-based polyion complexes and oil at this crosslinker are more likely to blame for the slow release of oil from the alginate/chitosan matrix.

#### 3.3.4. Antimicrobial activity

The antimicrobial activity of gauze cotton-treated textiles was tested against three microbes: Escherichia coli (E. Coli), Staphylococcus aureus (S. Aureus), and Candida albicans (C. Albicans).



Figure 11: The release profiles of treated gauze fabric with Alginate/Chitosan/oil matrix in presence and absence of BTCA

DICH							
Time (min)	Released oil (%)						
	Sesame oil	Sesame oil/BTCA	OMEGA 3	OMEGA 3/BTCA			
0	0	0	0	0			
15	34.52	21.36	9.99	3.32			
30	47.05	36.47	11.88	6.63			
60	58.61	48.52	31.44	17.35			
120	63.42	56.70	53.18	38.00			
180	68.24	61.52	60.43	52.50			
240	73.06	66.34	64.05	57.93			
300	77.87	71.16	67.68	61.55			
360	77.87	73.56	71.30	65.18			

**Table 3**: The release profiles of treated gauze fabric with Alginate/Chitosan/oil matrix in presence and absence of BTCA

Furthermore, because all the bacteria examined had different cell wall structures, the treated textiles are more effective for Gram-positive bacteria than Gram-negative bacteria.

The inhibitory effect of polyphenolic chemicals in microbial RNA and DNA has been linked to antimicrobial action. The cytoplasmic microbial membrane can also be depolarized. Because ergosterol inhibits a critical component in fungi's cell membrane, the chemicals also have antifungal properties. [39, 41, 61, 72-78]

Fabric treated with composites based on alginate/chitosan/OMEGA 3 oil matrix, on the other hand, shows higher reduction percent values than fabric treated with alginate/chitosan/sesame oil matrix, owing to the presence of chitosan in the matrix, which successfully interacts with bacterial cells during the coating process. [28-30, 55]

After 10 washing cycles, the bacteria reduction percent of treated textiles was studied, and the microbial count decreased. These findings indicated that treated textiles have a high level of microbial resistance, which is reduced by washing but still inhibits bacteria growth. Additional proof that these treated textiles will produce favorable results in the **Table 4** and **Figure 12**.

The two types of bacteria and fungi examined have no effect on untreated gauze cotton fabric (blank). In contrast, fabric treated with alginate/chitosan only without oil provide an antimicrobial activity with  $35.23\pm0.59$ ,  $45.19\pm3.88$ , and  $51.94\pm0.92$  mm<sup>2</sup> for E. Coli, S. aureus, and C. Albicans, respectively. Furthermore, fabrics treated with both alginate/chitosan/oil matrix shows a medical area.

Because of the amine groups, which have a high bactericidal impact because of their positive charge, the addition of chitosan in a composite has also been demonstrated to offer a greater bacterial decrease, demonstrating the antimicrobial action of the treated textiles. This is because the interaction of amines with gram-negative cell surface lipopolysaccharides, which are more anionic, serves to inhibit nutrition transfer into the cells. **[76-79]** 

Furthermore, this amino group increases the lipophilic characteristics of the compounds. The antibacterial effect of the treated materials was therefore predicted to have a lower influence on the presence of OMEGA 3 oil in the amino group of sesame oil than on the presence of OMEGA 3 oil in the amino group of sesame oil. As a result, the bacterial cytoplasmic membrane's contact mechanism was disrupted. **[80]** 

The antimicrobial activity of treated gauze cotton fabric was evaluated using the inhibition zone system (as a qualitative method) against Escherichia Coli (E. Coli; ATCC 25922), Staphylococcus aureus (S. Aureus; ATCC 29213), and Candida Albicans (C. Albicans; ATCC 10231) as a fungus as depicted in significant improvement in antibacterial activity with good efficiency against the three types of microbes tested (bacteria and fungi).

The presence of sesame oil or OMEGA 3 oil in the alginate/chitosan matrix is responsible for the high activity. Furthermore, treated fabrics are more effective against Gram-positive bacteria than Gram-negative bacteria, which is due to the differences in cell wall construction between the two bacteria strains studied.



Figure 12: antibacterial activity of untreated and treated cotton gauze fabrics with alginate/chitosan/oil matrix in the presence and absence of crosslinker against three microbes after washing

a) E. Coli, b) S. aureus, and c) C. Albicans

- 1) untreated fabric2) treated fabrics with the crosslinker
- 3) treated fabrics with alginate/chitosan/sesame oil matrix in presence of a crosslinker
- 4) treated fabrics with alginate/chitosan/sesame oil matrix in absence of a crosslinker
- 5) treated fabrics with alginate/chitosan/OMEGA 3 oil matrix in presence of a crosslinker 6) treated fabrics with alginate/chitosan/OMEGA 3 oil matrix in absence of a crosslinker

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	Crosslinker	Antimicrobial Activity								
Oil		E. coli (ATCC 25922)			S. Aureus (ATCC 29213)			C. Albicans (ATCC 10231)		
			Bacteria Reduction			Bacteria Reduction			Bacteria Reduction	
		ZI (mm <sup>2</sup> )	before washing	after 10 washing cycles	ZI (mm <sup>2</sup> )	before washing	after 10 washing cycles	ZI (mm <sup>2</sup> )	before washing	after 10 washing cycles
Blank fabric		0	0	0	0	0	0	0	0	0
Control fabric without oil		35.23±0.59	21.41	11.43	45.19±3.88	25.92	13.82	51.94±0.92	19.24	10.27
Sesame oil	without BTCA	87.14±1.88	42.82	22.85	133.85±4.59	51.85	27.65	58.79±0.11	38.48	20.54
	with BTCA	91.54±2.69	51.99	47.73	78.83±5.49	58.28	41.11	89.78±0.87	46.66	44.9
OMEG 3 oil	without BTCA	83.33±0.06	54.57	27.29	183.11±952	56.85	39.79	75.59±0.16	40.65	28.35
	with BTCA	92.92±3.09	56.53	45.04	120.49±4.35	61.85	53.06	98.67±0.47	50.79	45.66

Table 4:microbial reduction % for treated fabrics with sesame oil and OMEGA 3 oil in the presence and absence of crosslinker



Figure 13: cell viability (%) with treated gauze cotton fabrics alginate/chitosan with both oils in the presence and absence of  ${\rm BTCA}$ 

#### 4. Conclusion

The healing of burn wounds can be helped by using a medical dressing treated with sesame oil or omega oil by encapsulation due to their properties in treating burns because they contain many bioactive compounds. Capsules of alginate-chitosan loaded with sesame oil or omega oil were formed by emulsion method, and the size of the capsules ranged from 20 to 50 nm. The capsules were formed at different ratios between alginate and chitosan, then they were formed at different percentages of surfactant concentration and after that, it was formed at different percentages of oil concentration. The best parameters for capsule formation were as the following: the ratio between alginates to chitosan 5 to 1 and the concentration of surfactant was 2% and the oil concentration was 10%. Three different ways were used to treat the fabric with treatment solution and the best way was pad-dry-cure. It gave a homogeneous coated layer from capsules on the fabric's surface. The results suggest that the morphology of both sesame and omega oil-loaded capsules was spherical. The sesame oil-loaded capsules were smaller than omega oil-loaded capsules. The results show that the oil release was slow and sustained release at pH 7.5 for 6 hours. The release of omega oil was faster than the sesame oil. Oil release of both oils is affected by the presence of cross linking agents, as the oil release was faster in

the absence of crosslinking factors than its presence. The treated fabrics show accepted mechanical and physical properties in addition to having antimicrobial activity. The effectiveness of fabrics treated with omega oil on inhibiting microbes is better than fabrics treated with sesame oil. The effectiveness of fabrics for antimicrobial activity is affected by cross linkers, whereas it gives better results in their presence than in their absence.

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