



Preparation and application of lemon peel oil (*Citrus aurantifolia*) to improve microbial resistance of wool and viscose fabrics

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Abstract

Lemon peel oil (oil), its nanoemulsion as well as encapsulated with nanoclay were prepared and applied in the treatment of wool and viscose fabrics to enhance their resistance to microbes. Gas mass spectrometry (GC/MS) oil was used to identify volatile bioactive compounds. The qualitative and quantitative phenolic compounds were determined using HPLC. Transmission Electron Microscopes (TEM) and scanning electron microscope (SEM) were used for the determination of the size, shape, and size distribution of nanoparticles of oil and nano clay respectively. The morphological changes of the treated fabrics were characterized by using (SEM) and energy dispersive X-ray analysis. The changes in its antimicrobial activity have been studied.

Results of this study showed that the volatile compounds by GC-MS analysis for oil represent (98.3%) is of cyclic monoterpene. The size of the prepared nanoencapsulation oil was about 28 nm. The antimicrobial results proved significant improvements against *Staphylococcus aureus* (G+) of the fabrics treated with oil (*Citrus aurantifolia*) and its derivatives compared to the untreated ones.

Keywords: lemon peel oil, nanoemulsion, nanoclay, encapsulated oil, wool, viscose, fabrics, and microbial resistance

1. Introduction

Wool is an elegant fiber that advantages a smooth, excellent drape, and breathability. Wool is generally utilized in underwear and sports clothes [1]. And viscose is an important regenerated cellulosic fiber mostly utilized in the textile industry. The advantages of viscose are perfect spinning, ease of wearing, and not readily generating static [2]. Otherwise, natural organic textiles like cotton and wool help to grow microorganisms easily by providing a suitable environment like moisture, oxygen, nutrients, and temperature. This leads to hateful odor, allergic responses, and other concerning diseases [3]. The utilize of chemical pesticides to overcome the bacteria to the growth of many problems like pollution of the environment and the presence of generations from

insects that are resistant to pesticides. Newly, the awareness of researchers led to the use of natural plant material [4]. Extracts of plants, like neem, sandalwood, jasmine, and eucalyptus oil have been incorporated into textiles to improve their functional properties. Moreover, the medicinal plants in Egypt are of high economic value and new favorable wealth [5]. Such as silver nanoparticles colloidal solutions (AgNPs) were designed from *Citrullus Colocynthis* which was collected from the Egypt desert, (either whole fruit or separate seeds or fruit without seeds) as reducing and stabilizing. Wool and viscose fabrics were treated with the prepared AgNPs colloidal solutions to agree to them antimicrobial against Gram-positive bacteria, Gram-negative bacteria, Yeast, and Fungi [4].

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Recently, the synthesis of *Citrullus colocynthis* (L.) extracts alone or together with certain innocuous acids are considered eco-friendly nontoxic natural materials with a promising future for multi-functionalization of natural textiles, notably cotton and wool [6]. On the other hand, Citrus plants constitute one of the essential oil main sources and they are widely cultivated all over the world. This genus comprises more than 140 genera and 1300 species. The important kind is lemon [7] which contains principal components like citric acid, ascorbic acid, minerals, flavonoids, antioxidative, anti-inflammatory, antiallergic, antiviral, antiproliferative, antimutagenic, and anticarcinogenic activities [8]. Nano kaolinite was prepared from clay, a kind of fine-grained physical soil material containing clay minerals, which are hydrous aluminum phyllosilicate minerals. it was applied to lightweight wool fabric using pad batch and exhaust process for improving its fire resistance [9]. Nano kaolin (NK) is applied to realize extra values to viscose fabrics. as an increase in tensile strength, wettability, and antimicrobial resistance to different species of bacteria and fungi [10]. Science, new technology, and specialized materials were utilized to develop the textile industry as a sonication method which is the behavior of set sound energy to motivate particles in a pattern, for various purposes like the extraction of different compounds from plants, microalgae, and seaweeds [11]. The increase in the extraction of bioactive compounds achieved using sonication is an attribute to cavitation in the solvent, a method that contains nucleation, growth, and fallout of bubbles in a fluid, driven by the passing of the ultrasonic waves [12] and can be applied to produce nanoparticles, like nanoemulsions [13]. Fabrics with antibacterial properties are become important to control the infestation by microbes especially when treated by using natural environmental productions textiles with amended functionality find a diversity of applications like health and hygiene productions, particularly the garments worn tighten to the skin and various medical usages, such as infection control and barrier material [14]. So, in this study wool and viscose fabrics treat with the oil of lemon peel and its nanoemulsion as well as encapsulated oil using nano clay in the presence and absence of dye to impart their antimicrobial property. Which is considered safe, cheap, and economical compared to chemical treatments which are harmful to health, and it is difficult to dispose of them in the sewage system.

2. Materials and methods

2.1. Chemicals and dyes

Oil of lemon peel (*Citrus aurantifolia*) grown in Egypt was used in the present study. The peels collected from local Egyptian stores were rinsed and used to prepare the essential oil. Non-ionic

surfactants (tween 80 and span 20) were procured from Sigma-Aldrich Co., St. Louis, USA. Nutrient and sabouraud dextrose agar media were obtained from Merck, Darmstadt, Germany.

Clay(kaolin) was gained from Middle East Mining Investments Company (MEMCO), Cairo, Egypt. Other chemicals were used from the laboratory grade. Wool fabric (140 g/m² and yarn count Nm 70/2) and viscose fabric (110 g/m², number of warps is 375/10 cm, and number of wefts

is 320/10 cm) was purchased by Miser firm for Spinning and Weaving, Mahalla El- Kubra, Egypt. The dyes used (Figure 2) were commercial samples; C.I. Acid Red 249, Formula:

CC₂₉H₂₀C₁N₃Na₂O₁₀S₃ (Egypt Colors, (Egypt) and C.I. Reactive red 120, Formula: C₄₄H₂₄C₁₂N₁₄Na₆O₂₀S₆ (BASF Aktiengesellschaft)

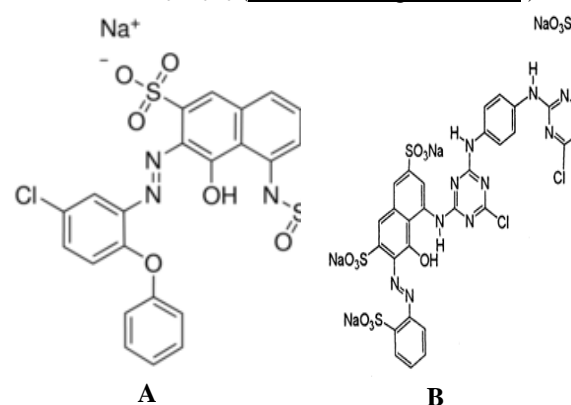


Figure 1: Chemical structure of (A) C.I. Acid Red 249 and (B) C.I. Reactive red 120

2.2. Scouring of fabrics

Wool was washed for 30 minutes in a solution containing 1 g/l sodium carbonate and 2 g/l nonionic detergents (Egyptol) at 50°C with a liquor ratio of 1:50. Also, viscose fabrics were scoured for 45 minutes with (2g/L) nonionic detergent solution at 60°C with a liquor ratio of 1:25. Fabrics were washed by warm and cold water, then dried at ambient temperature.

2.3. Preparation of lemon peel oil

Oil of Egyptian lemon (*Citrus aurantifolia*) was extracted using the hydro-distillation method for the outside fresh layer (flavedo) according to Shukla *et al* [15]

2.4. Preparation of nanoemulsion of lemon peel oil

The nanoemulsion of lemon peel oil was carried out using the oil as an internal stage in deionized water. Surfactants were mostly used to stabilize micro-emulsion systems during the preparation of microspheres. Primarily, the surfactants were mixed with deionized water, and all the mixtures were heated up to 40 °C. then the mixture was cooled to 29 °C, dropwise of oil was added to complete 50 ml of emulsion batch. The batch was activated by sonication for 30 min using an ultrasonic processor that worked at 20 kHz with a power of 750 W. Moreover, an ice bath was utilized to inhibit the temperature increase during the ultrasonication method [16].

2.5. Preparation of nanoclay

Conventional particle size reduction technique using Ball milling process (Ball milling Type: P_Q-N₂ Planetary Ball Mill, Gear Drive 4- station – planetary Ball mill, 220 V) was applied to prepare nanoparticles of clay according to Joni *et al.*, [17]. A hundred grams of the powdered sample was ball milled in a 200 ml agate vessel with 130 numbers of zirconia beads in the range from 0.5 to 1.5 mm diameter (75 beads 0.5 mm diameter, 30 beads 1.0 mm diameter, and 25 beads 1.5 mm diameter) at 4000 rpm then the dried sample powder was pulverized for 90 min to obtain a fine powder.

2.6. Preparation of encapsulation of lemon peel oil

The nanoemulsion of lemon peel oil was added to the nanoclay sample as a carrier to prepare encapsulation of lemon peel oil. The mixture was shaken for 90 minutes using a mechanical high-speed homogenization device at a speed of 24,000 rpm [18].

2.7. Treatment and dyeing of wool and viscose fabrics

Wool and viscose fabrics were flooded using the pad-dry-cure technique with a concentration of 1 % (o.w.f) of prepared oil and its derivatives, at pick up 90%, L.R: 1:50, dried at room temperature, cured at 140 °C for 4 min. On the other hand, the exhaustion method using a laboratory shaking device (Julabo- Germany) was

applied to dyed wool and viscose fabrics with 1% C.I. Acid Red 249 and C.I. Reactive Red 84 respectively, and treated fabrics in one step with a concentration of 1 % (o.w.f) oil nanoemulsion and 1 % (o.w.f) nanoclay, then the solution was adapted to pH 4:4.5 for acid dye and 4.5-5 for reactive dye, liquor ratio 1: 50 at 85 C° for an hour. The dyed treated samples were washed with hot and cold water and air-dried

3. Measurements and analysis

3.1. Gas chromatographic-mass spectrometric analysis for lemon peel oil

The volatile compounds in lemon oil were analyzed using coupled gas chromatography Hewlett-Packard pattern (5890) / mass spectrometry Hewlett-Packard-MS (5970) (GS/MS). Ionization voltage is 70 eV, mass zone m/z 39-400 a.m. Results for the peaks were matched to the known

constants of the mass spectra (National Institute of Standard and Technology, NIST) and approached with those of main compounds. The quantitative account has been examined based on crest region integration [19].

3.2. High-performance liquid chromatography analysis (HPLC)

High-performance liquid chromatography analysis was carried out to determine the polyphenolic compounds by an Agilent 1260 spectrum. Eclipse C18 column (4.6 mm x 250 mm i.d., 5 µm) was used to separate the compounds. The mobile phase contains water (A) and 0.05% trifluoroacetic acid in acetonitrile (B) at a flow rate of 1 ml/min. The mobile phase was designed sequentially in a

Linear-gradient as follows: 0 min (82% A); 0–5 min (80% A); 5-8 min (60% A); 8-12 min (60% A); 12-15 min (82% A) and 15-16 min (82% A). The multi-wavelength detector was observed at 280 nm. The injection volume appreciates 10 µl for each of the sample solutions. The column temperature was kept at 35 °C.

3.3. Determination of Antioxidant Activity

a- ABTS test

The total antioxidant activity was carried out by the improved casino-bis (ethylbenzothiazoline6-sulfonic acid) radical scavenging (ABTS). For the 2, 2- azino bis (3- ethylbenzothiazoline - 6-

sulfonic acid) (ABTS) test, the procedure used the style of Thipong [20]. Results were shown in mM Trolox equivalents (TE)/ ml extract. More dilution was necessary if the ABTS value measured was above the linear zone of the standard curve.

b- FRAP test

The method of Thipong was utilized to determine the ferric reducing antioxidant power (FRAP) [20]. Results were expressed in mM TE/ml extract. It should increase the dilution, if the FRAP value showed was over the linear range of the standard curve.

c- DPPH test

The antioxidant activity was measured by using the 2,2-diphenyl-1-picrylhydrazyl free radical (DPPH) test, which was carried out as reported by Thipong, [20]. It was determined using a calibration curve prepared with ascorbic acid and expressed as mg of ascorbic acid equivalent (AAE) per ml of sample.

3.4. Transmission Electron Microscopy (TEM)

The shape and size of the prepared nanoemulsion of oil of lemon peel solutions were practically determined using TEM; JEOL-JEM-1200-Japan. Samples for TEM measurements were intended by placing a drop of colloidal solution on 400 mesh copper grids coated by an amorphous carbon film and evaporating the solvent in the air at room temperature. The prepared nanoemulsion solutions' average diameter is specified from the diameter of 100 nanoparticles found in several arbitrarily chosen areas in enlarged microphotographs.

3.5. Antimicrobial activity

3.5.1. Antimicrobial activity for lemon peel oil and its derivatives

Evaluation of the antimicrobial activity for lemon peel oil and its derivatives was achieved towards *Staphylococcus aureus* ATCC 6538 P 6538, *Escherichia coli* ATCC 8739, and *Candida albicans* ATCC 10231 utilizing the completely spread process [21], twenty milliliters of either nutrient or sabouraud dextrose agar media were poured into Petri dishes. After solidification, the nutrient agar plates were inoculated by 200 μ L of each microbial suspension (matching a 0.5 McFarland standard solution) of either *Staphylococcus aureus* or *E. coli*, while sabouraud dextrose agar was inoculated by *Candida albicans* suspension, (appropriate a 1 McFarland standard liquid. The suspension is equally diffusion utilizing a sterile swab over the surface of the medium. Wells of 7mm in diameter were synthesized in the agar dishes and 0.1mL of the sample solution

was put into the wells and kept at 37°C for 24 h. The diameter of the inhibition zone created around the wells in mm. was used to measure the antimicrobial activity.

3.5.2. Antimicrobial activity for the treated fabric

The antimicrobial activity of the treated tissues was estimated with the same microbes mentioned previously. Initially, a single colony of the tested bacteria and fungi was cultured in 30mL of nutrient broth then incubated at 37°C and 180rpm for 18h. The treated fabrics (1cm²) has been dipped in 10mL of 0.05M phosphate buffer (pH 7) in 50mL screw-capped bottles that were inoculated by 200 μ L of the previously cultured bacteria then incubated at 37°C and 150rpm for 24h. After vigorous shaking to detach the fabric adherent cells, the optical density (OD) of the sample solution was determined at 600nm. The cultured bottle without the addition of any fabric was the control (100% growth).

3.6. Washing fastness

The fabrics were washed using 5 g/l nonionic detergents (o.w.f.), for 45 minutes, at a 50C °, liquor ratio of 1:50 [22].

3.7. Mechanical properties

Mechanical properties of treated fabrics as well as untreated fabrics using an Instron Tensile Tester (USA) according to ASTM D 76 Standard Specification for Textile Testing Machines to measure the tensile and elongation for untreated and treated fabrics.

3.8-Scanning Electron Microscope (SEM) and Energy Dispersive X-ray Analysis (EDXA)

The distribution of raw clay, its nanoparticles, and encapsulated with nano oil of lemon peel were observed under (SEM) by (type QUANTA FEG 250, HOLLANDE) as well as untreated and treated dyed fabrics with oil nanoemulsion and nano clay. In addition, EDXA was used to show the presence of new elements in dyed treated fabrics compared to untreated fabrics.

4. Results and discussions

4.1. Gas chromatographic-mass spectrometric analysis for lemon peel oil

In the current study, two compounds that represented 99.7% of the oil were identified. As illustrated in Table (1), limonene was the main constituent as it

represented 98.3 % and citral was the second compound that represented 1.4 %. Lemon oil is generally monoterpenoid. Limonene, is a cyclic monoterpene, has a hydrophobic nature, and exists as oil in citrus peel. This mixture, as a natural preservative, could be very helpful to resist microbial, fungal, and parasites [23].

Table (1): GC/MS analysis for lemon peel oil

| Peak | RT | Name | Formula | Area Sum % |
|------|--------|------------|-----------------------------------|------------|
| 1 | 11.323 | D-Limonene | C ₁₀ H ₁₆ | 98.3 |
| 2 | 20.158 | Z-Citral | C ₁₀ H ₁₆ O | 1.4 |

4.2. Total phenolic and flavonoid contents for lemon peel oil

The total phenolic content was calculated as gallic acid equivalent, and the result indicated that the total phenolic content of the extracted lime oil was 0.02 mg GAE/mL while the total flavonoid content analysis indicated the presence of 0.01 mg CE/mL. The polyphenolic compounds for lemon peel oil were evaluated by HPLC analysis, the results were illustrated in the table.

Table (2): Identified polyphenolic compounds for lemon peel oil by HPLC analysis

| Compound | Conc. (µg/g) |
|------------------|--------------|
| Gallic acid | 0.97 |
| Chlorogenic acid | 0.99 |
| Syringic acid | 3.34 |
| Pyro catechol | 2.31 |
| Rutin | 11.73 |
| Ellagic acid | 0.96 |
| Ferulic acid | 4.08 |
| Naringenin | 0.30 |
| Taxifolin | 1.96 |
| Cinnamic acid | 0.08 |

4.3. Transmission electron microscopy of nanoemulsion of lemon peel oil

Nanoemulsion is a heterogeneous style consisting of one immiscible liquid sparse as droplets within another liquid. These droplets of nanoemulsion measure between 20 to 500 nm. The diameters and surface properties of droplets of nanoemulsion perform a significant role in the biological manner of the formulation. Small droplet sizes appear as transparent emulsions so that product appearance is not changed by the addition of an oil phase [24]. In the

current study, the droplet size of the prepared nano-emulsion was examined under TEM and the result indicated that it was in a range between 8.96 and 13.06 nm as shown in figure (2).

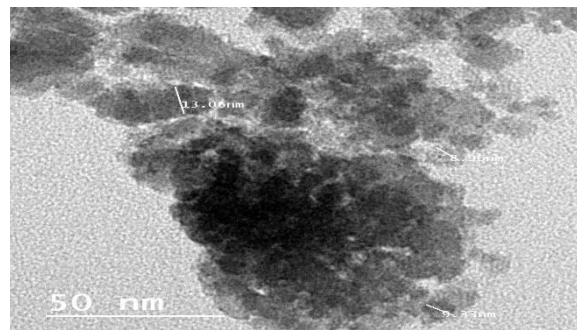


Figure (2): Transmission electron microscope of nano-emulsion of lemon peels oil

4.4. SEM of the prepared nano clay and encapsulation of lemon peel oil nanoemulsion

The morphological characteristics of clay samples were examined under SEM and results are shown in figure (3) (A, B) indicated that the particles size of raw clay was approximately 12.8 mm while prepared nanoparticles were in the range between 26.22 and 27.17 nm. In addition, by examining encapsulated nano-emulsion, it had been indicated that its size was about 28.22 nm (Figure 3C).

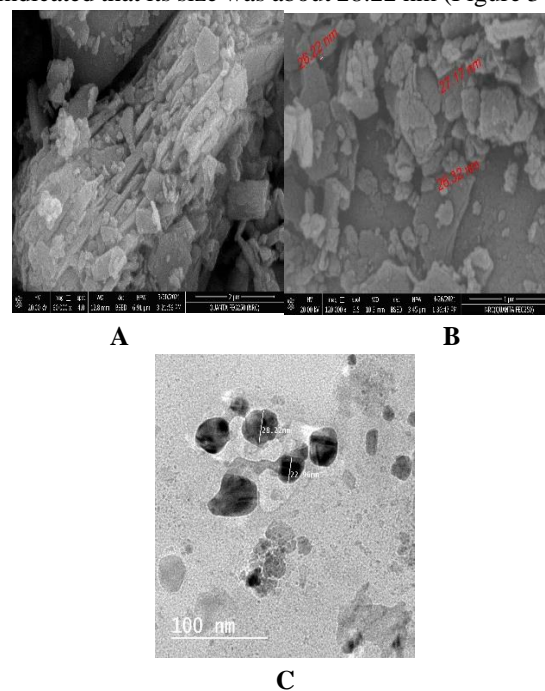


Figure 3: SEM:(A) raw clay and (B) nano clay, TEM: (C) encapsulation of lemon peel oil nanoemulsion

4.5. Antioxidant activity

The antioxidant activity of essential oils is generally related to their main components and their possible synergistic effects [25]. In the current study, the antioxidant activity of the prepared nanoemulsion oil, as well as the encapsulated form, was determined by three complementary techniques and the results illustrated in the table (3) indicated no significant difference.

Table (3): Antioxidant activity for nanoemulsion oil and Encapsulated form

| Sample | DPP (% inhibition) | ABTS (mmol TE /ml) | FRAP (mmol Fe ⁺² / ml) |
|-------------------|--------------------|--------------------|-----------------------------------|
| Nanoemulsion | 12% | 0.61±0.001 | 0.33±0.02 |
| Encapsulated form | 20% | 0.66±0.001 | 0.52±0.019 |

4.6. Antimicrobial activity

4.6.1. Antimicrobial activity for lemon peel oil

A stock solution (10% w/v in DMSO) of oil was prepared to examine its activity against microbes. The result shown in figure (5) indicated that the prepared oil exhibited antibacterial activity against the examined strains with an inhibition zone of 18 and 25mm against *Staphylococcus aureus* (G+) and *Escherichia coli* (G-) (*E. coli*) respectively. Additionally, it possessed an antifungal activity against *Candida albicans* with an inhibition zone of 18mm. This result agrees with that reported by Kamal, et al [26] that the major components of lemon essential oil from monoterpene hydrocarbons and their oxygenated derivatives increase its antimicrobial activity.

4.6.2. Antimicrobial activity for oil nanoemulsion and its encapsulation

The results of the antimicrobial activity for oil nanoemulsion and its encapsulation as shown in table (4). It is illustrated that increased by increasing the percentage of the extracted oil but encapsulation with nano clay adversely affected antimicrobial activity. This is consistent with what was proposed by previous investigations that kaolinite dissolution is encouraged by three kinds of low-molecular-weight organic acid, i.e., citric, oxalic, and malic acids [27]. In addition, Zhang et al., (2017) [28] reported that the kaolinite composition was slightly broken by citric acid as it changed their activity.

Table (4): Antimicrobial activity for lemon peel oil and its derivatives in which the inhibition zone was measured in millimeters.

| Sample | <i>S. aureus</i> (G+) | <i>E. coli</i> (G-) | <i>Candida albicans</i> |
|---|--------------------------|---------------------|-------------------------|
| | Antimicrobial activity % | | |
| Oil nanoemulsion (5 %) (w/v) + 0.25 % (w/v) surfactant | 30 | - | 40 |
| Oil nanoemulsion (15 %) (w/v) + 0.5 % (w/v) surfactant | 36 | 10 | 44 |
| Oil nanoemulsion (30 %) (w/v) + 1 % (w/v) surfactant | 40 | 11 | 48 |
| Oil nanoemulsion (50 %) (w/v) + 3 % (w/v) surfactant | 44 | 18 | 50 |
| Oil nanoemulsion (50 %) (w/v) + 3 % (w/v) surfactant + nanoclay | 13 | - | 18 |
| Amoxicillin (10mg/mL) | 45 | 28 | Nd |
| Diflucan (10mg/mL) | Nd | Nd | - |

Nd: not detected

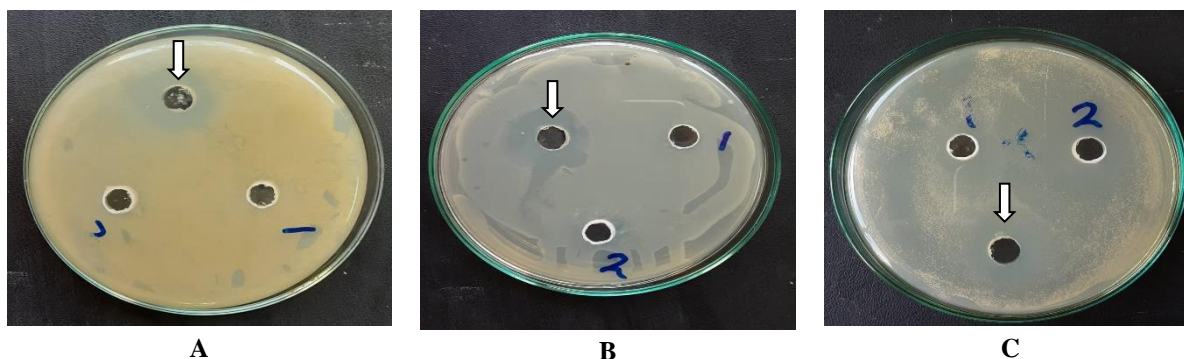


Figure 4: Antimicrobial activity for lemon peel oil against (A) *Staphylococcus aureus* (B) *E. coli* and (C) *Candida albicans*

4.6.3. Antimicrobial activity for the treated fabric

The treated fabrics showed a negative result against *Escherichia coli* and *Candida albicans*

However, the best results of the inhibition zone in millimeter of the growth rate for *Staphylococcus aureus* (G+) for wool and viscose fabric which was treated at concentration 1% (o.w.f) for each of the prepared nanoencapsulation of lemon peels oil (waste) and nano clay in presences of 3% surfactant which can be done as detergents, wetting agents, emulsifiers, foaming agents, or dispersants and dyed in one bath with (1 % C.I. Acid Red 249 and 1 % C.I. Reactive red 120) is 85.2% and 77.1 respectively compared to other untreated, dyed and treated fabrics as shown in table 6, 7. *Staphylococcus aureus* (G+) is a main human pathogen that causes an enormous range of clinical infections in the region of 30% of human people [29]. This result is consistent and confirms the ability of lemon which contains phenolics and flavonoids to dissolve the minerals in the clay [30]. which in turn can bind to the active groups in the wool and viscose

fabric through the dye that acts as a crosslinker between them. But the result of the inhibition zone in millimeter of the growth rate of *Staphylococcus aureus* (G+) for wool and viscose fabrics that were treated with the prepared concentration 1% (o.w.f) nanoemulsion of lemon peels oil which contains ingredients that have bioactivities. It could improve the result and dyed in one bath with (1% C.I. Acid Red 249 and 1% C.I. Reactive red 120) is 93.5 % and 90 % respectively parallel to other treated and dyed. Moreover, there is no enhancement for the inhibition zone in millimeters of the growth rate of *Staphylococcus aureus* (G+) to wool fabrics treated with the prepared concentration of 1% (o.w.f) of oil and nanoemulsion of lemon peels oil because it's volatile and needs to be attached to the fabrics by a crosslinker. On the other hand, the results of the inhibition zone in millimeters of the growth rate of *Staphylococcus aureus* (G+) to viscose fabrics treated with the prepared concentration of 1% (o.w.f) of oil and nanoemulsion of lemon peel oil enhancement than untreated and dyed fabrics. On the other hand, the inhibition zone in millimeters of the growth rate of *Staphylococcus aureus* (G+) for wool and viscose fabrics treated with the prepared concentration of 1% (o.w.f) nanoclay is the same as untreated fabrics. This may be attributed to the nanoclay structure consisting of two silicon tetrahedra and one aluminum or

magnesium octahedron sandwiched between two tetrahedra, which in turn changes the functional polypeptide groups in the case of wool and were incorporated into the basic structure of viscose fabrics.

Table (5): The growth rate of *Staphylococcus aureus* (G+) of treated wool fabric after washing for five cycles

| Sample | The growth rate of <i>Staphylococcus aureus</i> (G+) ATCC 6538 (%) |
|--|--|
| Untreated wool | 100 |
| Wool dyed with 1% (o.w.f) C.I. Acid Red 249 | 100 |
| Wool treated with 1% oil (o.w.f) | 100 |
| Wool treated with 1% oil nanoemulsion (o.w.f) | 100 |
| Wool treated with nanoclay 1% (o.w.f) | 100 |
| Wool treated with encapsulation 1% oil (o.w.f) | 100 |
| Wool dyed with 1% C.I. Acid Red 249 in presences of 1% nanoemulsion of oil (o.w.f) | 93.5 |
| Wool dyed with 1% C.I. Acid Red 249 in presences of 1% nanoemulsion of oil and 1% (o.w.f) nanoclay | 85.2 |

Table (6): The growth rate of *Staphylococcus aureus* in the presence of viscose fabric after washing for five cycles

| Sample | The growth rate of <i>Staphylococcus aureus</i> (G+) ATCC 6538 (%) |
|---|--|
| Untreated viscose | 100 |
| Viscose dyed with 1% (o.w.f) C.I. Reactive red 120 | 100 |
| Viscose treated with 1% oil (o.w.f) | 86.9 |
| Viscose treated with 1% nanoemulsion oil (o.w.f) | 95 |
| Viscose treated with 1% nanoclay (o.w.f) | 98 |
| Viscose treated with 1% encapsulation oil (o.w.f) | 100 |
| Viscose dyed with 1% C.I. Reactive red 120 in presences of 1% nanoemulsion of oil (o.w.f) | 90 |
| Viscose dyed with 1% C.I. Reactive red 120 in presences of 1% nanoemulsion of 1% oil and nanoclay (o.w.f) | 77.1 |

4.7. Tensile Strength and Elongation

Table 7 shows the results of tensile strength and elongation of wool and viscose fabrics before and after dyed in the presence of the encapsulated oil and nanoclay in one bath. it is illustrated that a little enhancement of the tensile strength of the treated fiber than the untreated one. But it is found that decrease in elongation for treated fabrics compared with untreated fabrics. This may be attributed to the presence of Al and Si elements that play an important role in this change.

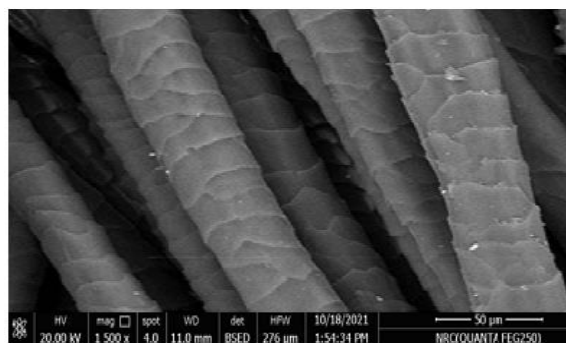
Table 7: Tensile strength and Elongation % of untreated and treated fabrics

| Samples | Tensile strength (kg/mm ²) | Elongation % |
|---|--|--------------|
| Untreated wool | 29.1 | 15.21 |
| Wool dyed with 1% C.I. Acid Red 249 in presences of 1% nanoemulsion of oil and 1% nanoclay (o.w.f) | 34.7 | 1.3 |
| Untreated viscose | 18.7 | 11.8 |
| Viscose dyed with 1% C.I. Reactive red 120 in presences of 1% nanoemulsion of oil and 1% nanoclay (o.w.f) | 21.1 | 9.0 |

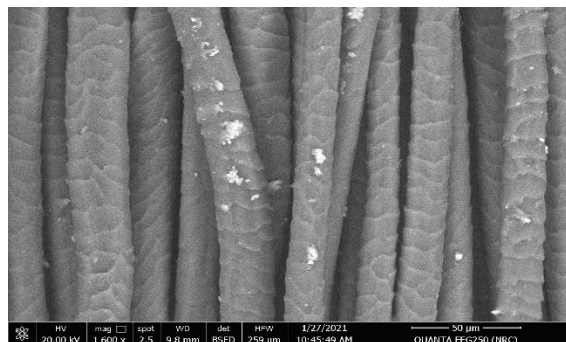
4.8. SEM and EDXR Analysis

The results of SEM for untreated and treated wool and viscose fabrics with concentration 1% (o.w.f) from oil nanoemulsion, nanoclay, and dye (C.I. Acid Red 249, Reactive red 120 respectively in one bath as shown in figure (6). It is illustrated that nanoparticle distribution on the surface of the dyed treated fabrics than the untreated one.

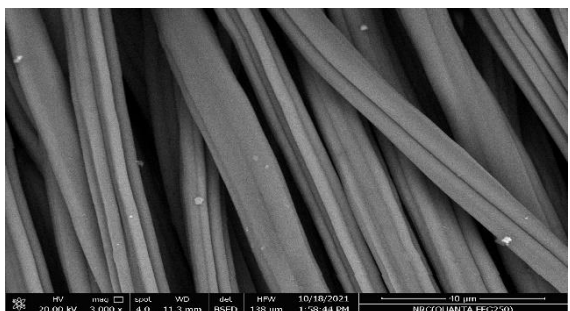
The results of EDXA analysis for untreated and treated wool and viscose fabrics with concentration 1% (o.w.f) from oil nanoemulsion, nanoclay, and dye (C.I. Acid Red 249, Reactive red 120 respectively in one bath as shown in figure (6,7). it is indicated that the presence of Al and Si elements for dyed treated samples as compared to the untreated samples that materials with positive charges possess strong antibacterial properties, bacterial surfaces charge change, and consequently, bacterial cells autolyzed. This is in harmony with opinions scientific previously,



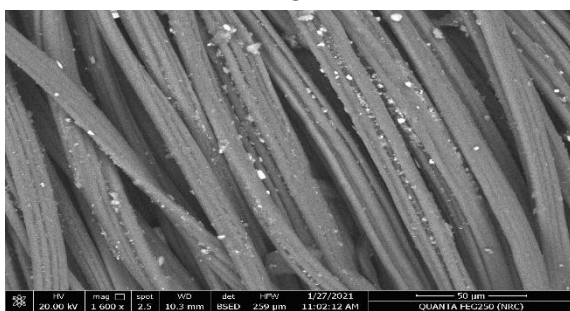
A



B

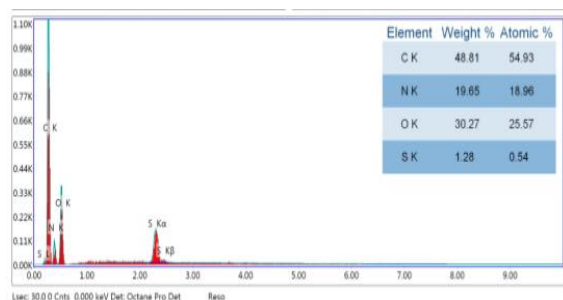


C

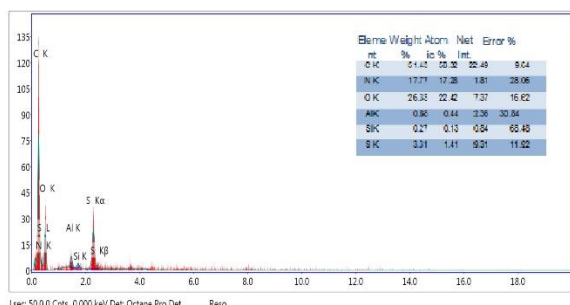


D

Figure 5: (A) Scanning electron microscope for untreated wool fabric, (B) dyed treated wool fabric (C), untreated viscose fabric, (D) dyed treated viscose fabric

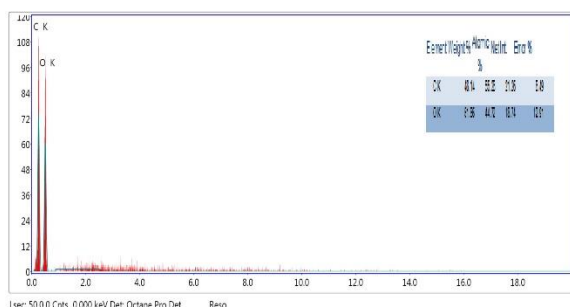


A

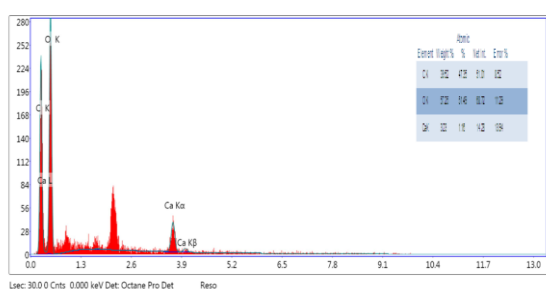


B

Figure 6: EDXA analysis of (A) untreated wool fabric and (B) dyed treated wool fabric



C



D

Figure 7: EDXA analysis of (C) untreated viscose fabric and (D) dyed treated viscose fabric

8. Conclusion

The preparation of oil from the peel of *Citrus aurantifolia* and its nanoemulsion as well as encapsulated with nano has been achieved. GC/MS analysis of the prepared oil indicated that limonene

and citral were the main constituents as they represented 99.7% of the oil. The antimicrobial activity of lemon peel oil and its derivatives was in vitro estimated against *Staphylococcus aureus* (G+), *E. coli*, and *Candida albicans*. The particle size of oil nanoemulsion was measured in the range between 22 and 28 nm using the TEM device. The treated fabrics showed a negative result against *Escherichia coli* and *Candida albicans*. It also turned out that the best results for inhibition of *Staphylococcus aureus* (G+) growth for dyed treated fabrics in one bath with nanoemulsion oil and nanoclay after 5 washing cycles using the exhaustion method.

The results of tensile strength and elongation to untreated and dyed treated wool and viscose fabrics showed a little improvement of the tensile strength and decrease in elongation for dyed treated fabrics than the untreated one. The results of EDX analysis indicate the presence of Al and Si elements for dyed treated fabrics than untreated ones which improves the effectiveness of fabrics for the microbe especially *Staphylococcus aureus* (G+).

Conflict of Interest

The authors declare no conflict of interest.

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