Arab J. Nucl. Sci. Appl., Vol. 56, 5, 19-26 (2023)

ISSN 1110-0451

Arab Journal of Nuclear Sciences and Applications

Web site: ajnsa.journals.ekb.eg

(E S N S A)

Water and Oil Repellent Cotton Fabrics via Coating with Electron Beam Curable Formulation

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ARTICLE INFO

Article history:

Received: 28th Jan. 2023

Accepted: 26th Feb. 2023

Available online: 20th May 2023

Keywords:

Cotton fabric;

Electron beam curing;

Water repellent;

Oil repellent;

Coating.

ABSTRACT

Cotton fabric was coated with trifunctional urethane methacrylate (UMAc) under the effect of electron beam irradiation to impart high hydrophobic and oleophobic characters to be suitable for furniture and tent usage. Acetone as a solvent in the presence of castor oil as a plasticizer was added to the formulation. Electron beam irradiation was proven to give the best homogenous fast curing of the formulation. The surface morphology that was examined by scanning electron microscopy (SEM) proved core coating and thickening of the fibers. The results indicated that the crease recovery and mechanical properties of coated fabrics were increased significantly, giving a high-performance fabric. The water absorption, water vapor permeability, and hydrophobicity of the treated fabrics toward water and aqueous liquids (tea and juice) were found to decrease. Oil repellency of the treated fabrics against different oils (clove, castor, and vegetable oils) was also studied. The contact angle for water-based and oil-based solutions was increased to reach 85° and 68° for tea and clove oil, respectively. Consequently, the results indicated that the hydrophobic and oleophobic properties of cotton fabrics were enhanced abruptly after the treatment process, accompanied by a slight increase in their thermal stability.

1. INTRODUCTION

Functional fabrics were developed to help humans to stand environmental influences, as storms, heat, dust, water, or to protect against other hazards (fire, contamination, corrosive substances, etc.) [1,2]. Industrial protective clothing with specific functions has been developed, such as fire resistance [3,4], water repellency [5], oil repellency [6], self-cleaning [7], UV protection [8], chemical resistance [7], or moistness managing [3,9]. Research was also focused on the manufacturing of textiles with multiple functions for protection against various environmental factors and thermoregulation behavior [10]. On the other hand, hydrophobic surfaces have been extensively used in industrialized fields such as self-cleaning, surface water-oil isolation, and water collection, and are recognized by chemical curing to adjust the surface energy, as well as physical curing to procedure a roughness on the surface [11].

In addition, oil and water repellent are amongst the greatest obligatory purposeful properties intended for protecting cloths. The properties may be deliberated through modifying the surface of textiles and may be limited towards a thin superficial coating, to preserve the physical properties of the textile, such as flexibility, mechanical strength, softness, and breathability [12,13].

The electron beam irradiation (EB) technology, in which no catalysts or initiators and contaminants are used. It provides several requisite benefits as high rate, environmentally friendly safety, manual labor throughout usage, and constancy, in adding to fast curative [14]. Also, EB irradiation can be used in various applications such as treatment of materials and polymers modifications [15-17], aqueous purity [18], surface modification [19]. Polymers show significant changes upon the exposure to ionizing radiation, and the electron beam curing can be developed a well accepter process through a great quantity of industrial applications mostly

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DOI: 10.21608/ajnsa.2023.190166.1714

used in fields of laser print, coat, adhesive manufacture, and microelectronics [20]. Electron beam rapidly converts liquid multifunctional oligomers and monomers into solid polymers in the form of micro thickness coating on the surface of materials. Among the benefits of electron beam irradiation with respect to the traditional chemical methods of coating is the use of fewer solvents, the achievement of 100% polymerization with minimum risks and environmental pollution. The initiation of chain polymerization at low temperature and high speed, and very low energy consumption compared to conventional heat treatment due to the high return of converting electrical energy to electrons by local radiation initiation.

The electron beam irradiation that was used is efficient for treating formulation typically, which contains oligomer, unsaturated monomer, and any other additive according to the properties, which desired [21]. In this regard, unsaturated oligomers that are used in the coating process by accelerated electron beams include oligo esters, polyether acrylate, and polyurethane acrylate, as epoxy acrylate [22]. Coatings containing polyurethane and its derivatives are used in the modification of fabrics such as a finishing agents for developing permanency and water vapor penetrability and for a variety of other applications. Substantial efforts have been devoted to increasing the performance of polyurethane coatings [23,24].

In this work, electron beam irradiation is used as a curing agent for the trifunctional urethane methacrylate (UMAc) oligomer. The formulations contain the reactive oligomer, plasticizer, and solvent was applied on cotton fabrics, to satisfy the multifunctional properties of oil and water repellency. Moreover, the effect of the coating procedures on the thermal stability, mechanical properties, surface morphology, and aqueous liquid and oil-repellent of the treated fabric is investigated.

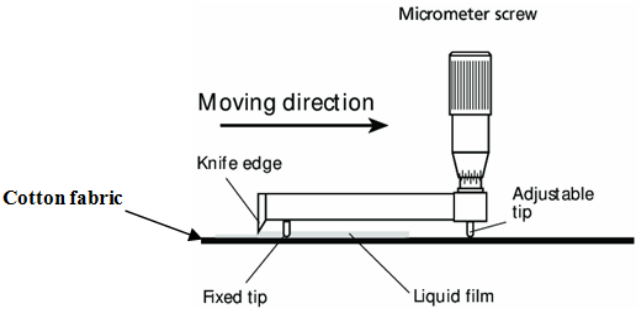
2. MATERIALS AND METHODS

2.1. Materials

Cotton fabric used in this work was kindly provided by El Nasr Spinning, Knitting, and Weaving Comp., Egypt. Before use, it was treated, with a solution comprising 5 g/L of Na_2CO_3 and 1 g/L of detergent at 60°C for 60 min to eliminate any undesired substances. Laboratory-grade trifunctional aromatic urethane methacrylate oligomer (UMAc) was purchased from Bomar Specialties Co., Hong Kong. Castor oil and acetone were purchased from El-Gomhoria Company, Egypt.

2.2. Coating of cotton fabrics with (UMAc) oligomer formulation

The UMAc) oligomer was first dissolved in acetone in the presence of castor oil as a plasticizer, with vigorous stirring to reach homogeneous solution. The cotton fabric was treated by coating the formulation with a film applicator using a thickness of 25 µ as shown in Scheme 1. The treated cotton fabric was then exposed to electron beams irradiation (2.5 MeV) utilizing an electron beam accelerator at National Center for Radiation Research and Technology. Required dose was obtained through regulating the conveyor speed and electrons beam factors.



Scheme (1): Procedure of coating on cotton fabrics using a formulation (UMAc) oligomer.

2.3. Thermogravimetric analysis

Thermogravimetric analysis (TGA) was performed by the TG-50 from Shimadzu (Japan) at a heating system rate of 10°C/min up to 600°C according to ASTM E1641-07. Measurements were carried out under a flowing nitrogen gas at 20 ml/min.

2.4. Scanning electron microscopy (SEM)

The SEM was used to examine the morphology of the cotton fabrics before and after coating. The SEM micrographs were taken with a JEOL JSM-5400 (Japan). A sputter coater was used to recoat conductive gold onto the fracture exteriors at 15 kV.

2.5. Mechanical properties

The tensile strength (TS) and elongation to the breaking point (E %) were established at room temperature. Fabrics sample was cut into strips of 100 mm long and 10 mm wide. The recorded data were the average of five readings. A Mecmesin tester, Mimesis Limited, UK, was used at a crosshead speed of 50 mm/min.

2.6. Crease recovery measurement

The crease recovery of fabric was tested in the dry state according the standard ASTM D1295-67. A tester model METEFEM (Type FF-07, Hungary) applying a weight of 1 kg for 5 min at room temperature. The recorded data of the crease recovery angles were the average of five measurements.

2.7. Water absorption properties

Water absorption was measured according to the standard method of the American Association of Textile Chemists and Colorists; test method 21-972. In this procedure, cotton samples (W_o) were soaked in a container of distilled water for 20 min, after which fabric was removed then reweighed (W_s). Water absorption percent is calculated agreeing to the following equation (1):

$$\textbf{Water absorption (\%)} = [(W_s - W_o)/W_o] \times 100 \qquad (1)$$

2.8. Water vapor permeability

Water vapor permeability was determined according to the standard ASTM E96 cup test, USA. Distilled water of fixed volume was placed in an Erlenmeyer flask and then weighted the flask (F_b). The flask was covered closely by the treated fabrics. This experiment was made at room temperature for 24 hrs or at a temperature of 70°C for 2 hrs, by using oven. After removing the sample, the flask was weighed again (**F_a**). The Water vapor permeability percent of the fabrics was calculated according to the following equation (2):

$$\textbf{Water vapor permeability (\%)} = [(F_b - F_a)/F_b] \times 100 \qquad (2)$$

2.9. Contact angle with water and oils

The contact angle for oil and water over the coated fabric was measured by applying a droplet of 5ml of different tested liquids, via the Milli-Q filtration system. Photographs of the samples were taken to evaluate the contact angle. Each sample was measured with an average of three reads.

3. RESULTS AND DISCUSSION

3.1. Water and aqueous liquid repellency of cotton fabrics

The water resistance, aqueous liquid, and oil of treated cotton fabrics are much greater than that of uncoated fabric owing to resistance of the surface to water. Table 1 lists the water contact angles (WCAs) and illustrate photos for uncoated cotton and cured coated textiles at optimum irradiation dose (50kGy) with the drops of water and aqueous liquid (juice and tea). As the cotton fabric is hydrophilic material, water, juice and tea can spread easily on it within a second. The coating by UMAc seems to resist aqueous liquids, (as shown in the side view). The UMAc coating formed a film on the surfaces of the textiles, which improved the roughness of the surface and reduce the surface energy. WCAs of UMAc coated fabrics are 75°, 70°, and 85° for water, juice, and tea, respectively, as shown in Table 1. The coating with UMAc oligomer would arrange as a film on the surface of cotton fabric; and thus enhances the roughness of and reduced the surface energy. The improvement of WCAs of cotton fabrics is due to the roughness and lower surface tension of the treat formulates [25]. Frequently, the low surface tension of the treated surface is the major role for repelling the aqueous liquefied [26]. The enhancement of WCAs is owing to the lower surface tension and improved roughness of UMAc coating; the less surface tension of the coated surface has a role for the resistance to aqueous liquids [27, 28].

Table (1): Contact angles of water and aqueous liquid droplet on uncoated and cured coated cotton fabrics at irradiation dose (50kGy) and its Photographs.

Water and aqueous liquid repellency			
Liquid	Water	Juice	Tea
Photographs of uncoated cotton fabrics			
Photographs of cured coated cotton fabrics			
WCAs (A°) of cured coated cotton fabrics	75	70	85

3.2. Oil repellency

Oil resistance depends on the surface tension of the treated surface. Untreated cotton fabrics have height surface tension that enhances the absorption of liquors and oils. The UMAc coating reduces the surface tension of treated fabrics and, therefore, enhances oil repellency [27]. The cured treated fabrics with UMAc at optimum irradiation dose (50kGy) have excellent oil repellency as shown in Table 2; the droplets of oils did not penetrate for about 600 seconds. Oils with higher surface tension, as castor oil (41.35 dyne/cm) or vegetable oil (26.8 dyne/cm) were repelled by the UMAc coating. This improvement in WCAs is owing to the surface homogeneity of coated cotton fabrics after being curative by electron beam radiation [25]. The lower surface tension of UMAc oligomer has a very pronounced beneficial effect on oil repellency.

3.3. Water absorption (WAP)

Cotton fabrics are known to have excellent wettability, as water droplets can be easily spread through the fibers. Figure 1 illustrates the effect of electron beam irradiation doses on the water absorption percent of cotton fabric before and after treatment with UMAc formulation. It can be seen that the WAP was greatly decreased after treatment with UMAc formulation regardless the irradiation dose. This due to the super hydrophobicity of the UMAc formulation, which prevents water from penetrating and spreading through the fabric. In addition, the sharp reduction of water absorption percentage for the coated fabrics might be because of the development of film as a hydrophobic layer going on the surface of the fabrics. No significant variation in (WAP) is noted by increasing irradiation dose of electrons beam [25].

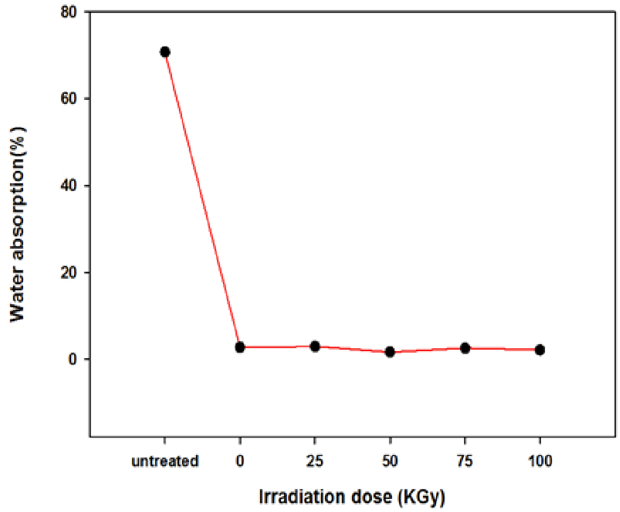
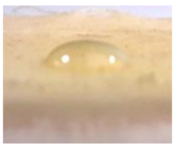
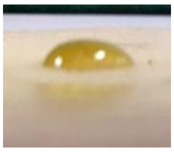


Fig. (1): The effect of electron beam irradiation doses on the water absorption percent of cotton fabric before and after treatment with UMAc formulation

Table (2): Contact angles and camera photos of different oil droplets (clove, caster, and vegetable oils) over uncoated and cured coated cotton fabric at irradiation dose (50kGy).

Oil Repellency			
Oils	Clove oil	Castor oil	Vegetable oil
Photographs of uncoated cotton fabrics			
Photographs of cured coated cotton fabrics			
WCAs (A°) of cured coated cotton fabrics	68°	65°	37°

3.4. Water vapor permeability

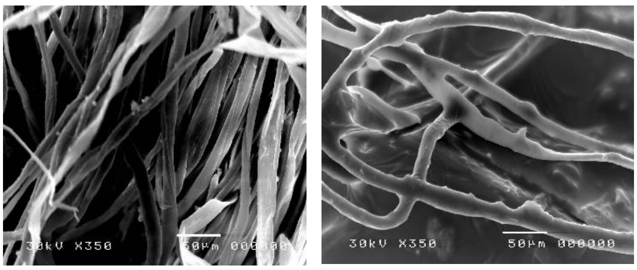
Water vapor permeability (WVP) is defined as the capability to permit vapor of water to pass over it. It is also definite as per “grade which the fabric, coat or lamination permits vapor of water to pass over that one building” [29]. Also the fabric (WVP) significantly affects the performance of fabrics. The WVP is a clear indication of the void blocking after coating of fabric treatments could have a minimal effect on vapor transfer [30]. As shown in Table 3, the uncoated fabric displayed a high WVP value while cotton fabrics coated with UMAc and curing at dose of 50 kGy results in the formation of blocking on the surface of fabrics and hence the vapor transfer is hindered and the resistance of water vapor for the coated textiles is high.

Table (3): The percentage of water vapor permeability for uncoated and cured coated cotton fabric at irradiation dose (50kGy).

Sample	Water vapor permeability (%)
Uncoated cotton	0.16
Coated cotton	0.07

3.5. Surface morphology

Figure 2 shows the scanning electron micrographs of cotton fabrics before and after coating with EB curable formulation. It can be seen that the coated fabrics appear in bulky form, in which the formulation appeared to diffuse and cover the fibers and the thickness of the fiber was increased compared to the uncoated fabric.



(a) **(b)**

Fig. (2): Scanning electron micrographs of: (a) uncoated fabric and (b) coated fabric with EB curable formulation at irradiation dose (50kGy).

3.6. Mechanical properties

The study of the mechanical properties is important because it affects the final application of polymers and polymer-coated substrates [31]. The effect of the UMAc coating on tensile strength (TS) and elongation (E%) of the cotton fabric was investigated as shown in Figure 3 and Table 4. It is clear that the value of TS was found to increase by increasing of irradiation dose, to reach a maximum value at 50 kGy due to the effect of crosslinking. Further increase in the irradiation dose up to 75 kGy, was accompanied with a decrease in the TS. This is attributed to the occurrence of degradation due to the effect of irradiation-induced chain scission. Radiation is known to cause either chain scission or crosslinking, according to overall conditions of radiation [32]. On other hand, E% for all the treated cotton fabrics was found to decrease by increasing the irradiation dose. This might be ascribed with the increased density of crosslinking, which hinders the movement of the molecular chain, resulting in an overall decrease in E%.

Table (4): Tensile strength and elongation at break (%) of cotton fabrics before and after coating with UMAc at different doses of EB irradiation.

Sample	radiation dose (kGy)	Tensile strength (MPa)	Elongation (%)
Untreated	----	3.63	64.86
Treated	0	15.46	68.54
	25	25.04	49.10
	50	57.42	42.09
	75	22.88	46.83
	100	20.19	37.96

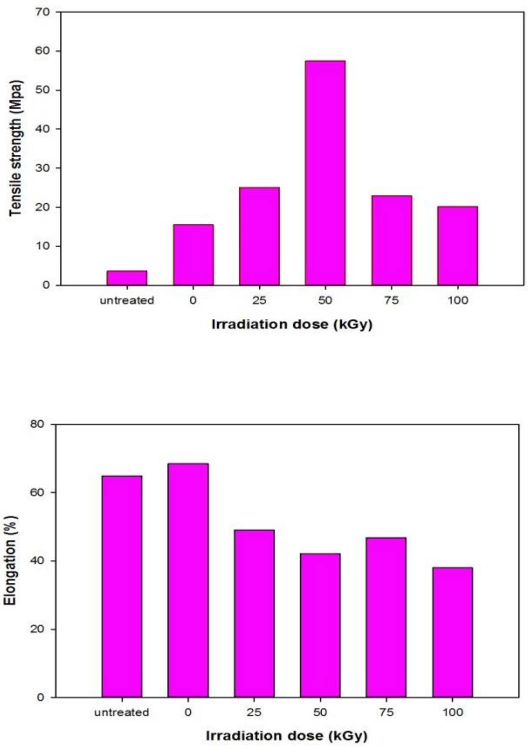


Fig. (3): Effect of electron beam irradiation dose on the tensile strength and elongation at break (%) of cotton fabrics before and after coating with UMAc.

3.7. Crease recovery properties

The crease recovery is defined as the capability of the fabrics to resist and recover from the deformation produced through the loading on the surface. Crease recovery can be determined via the angle between the two pre-folded halves, which is termed the angle of crease recovery [25]. Figure 4 shows the effect of electron beam irradiation doses on the crease recovery angle of cotton fabrics, before and after they had been treated by coating with UMAc oligomer. It can be seen that the crease recovery angle of the treated cotton fabrics was higher than untreated fabrics as shown in Figure 4. It was noticed that, the increasing of irradiation doses up to 25 kGy give a slight increase in the crease recovery angle of the treated cotton fabrics. Any further increase of irradiation doses up to 100 kGy has no effect on the crease recovery angle. The improvement in the crease recovery properties can be attributed to the increasing in elasticity and flexibility resulting from the occurrence of crosslinking of the UMAc formulation [33]. The slight decrease in the crease recovery angles of coated cotton with increasing irradiation dose up to 100 kGy is in accordance with previous reports [34]. At higher doses, degradation would occur and so the decrease in elasticity, which causes the decrease increase recovery [35].

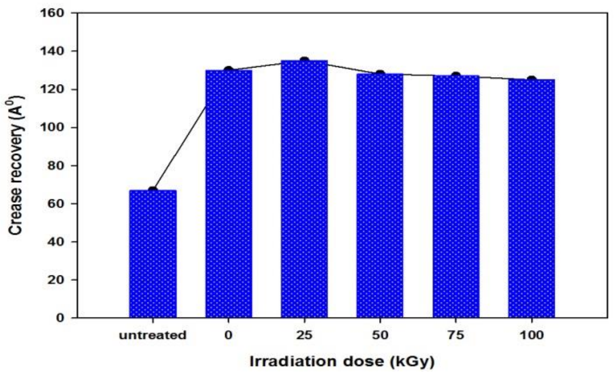


Fig. (4): Effect of electron beam irradiation doses on the crease recovery angle of cotton fabrics, before and after treatment by coating with UMAc oligomer.

3.8. Thermal stability

Figure 5 and Table 5 show the TGA thermograms and the corresponding rate of thermal decomposition reaction curves of cotton fabrics, before and after they had coat with UMAc formulation under the effect of electron beam irradiation. Overall, the heating temperature up to ~ 450ºC, the untreated cotton fabrics displayed higher thermal stability than the treated fabrics according to the weight loss (%). However, within higher temperatures up to 600ºC, the treated cotton fabric showed a relatively higher thermal stability. For untreated or treated cotton fabrics, the thermal decomposition goes through three stages. The first stage up to 255°C, there is nearly no difference of the thermal stability of either untreated or treated cotton fabrics. The main thermal decomposition occurs within the temperature range from 255-400ºC, which is attributed to cellulose dehydration [36]. While there is no difference in the weight loss (%) of 50 % between untreated and treated cotton fabrics, at 80% weight loss, the untreated cotton fabrics showed a higher thermal stability than treated fabrics.

Although, the rate of thermal decomposition curves showed the same behavior, the temperature of the maximum rate of reaction (T_{max}) differs from one substrate to another. In this regard, the T_{max} of untreated cotton was observed at ~320ºC, whereas the T_{max} of the treated cotton fabric was seen at ~350ºC. This finding indicates that the treated cotton fabrics is thermally higher than untreated cotton fabrics due to the occurrence of crosslinking of the UMAc coating under the effect of electron beam irradiation. The coated fabrics is thermally stable as result for the density of crosslinking and therefore the increased rigidity of the system [37].

Table (5): Temperatures at different weight loss (%) and that at maximum decomposition of cotton fabrics before and after electron beam irradiation coating with UMAc formulation at dose (50kGy).

Fabric samples	$T_{50\%}$ (ºC)	$T_{80\%}$ (ºC)	T_{decom} (°C)
Untreated cotton	341.6	421.8	510.0
Treated cotton	341.2	395.8	560.0

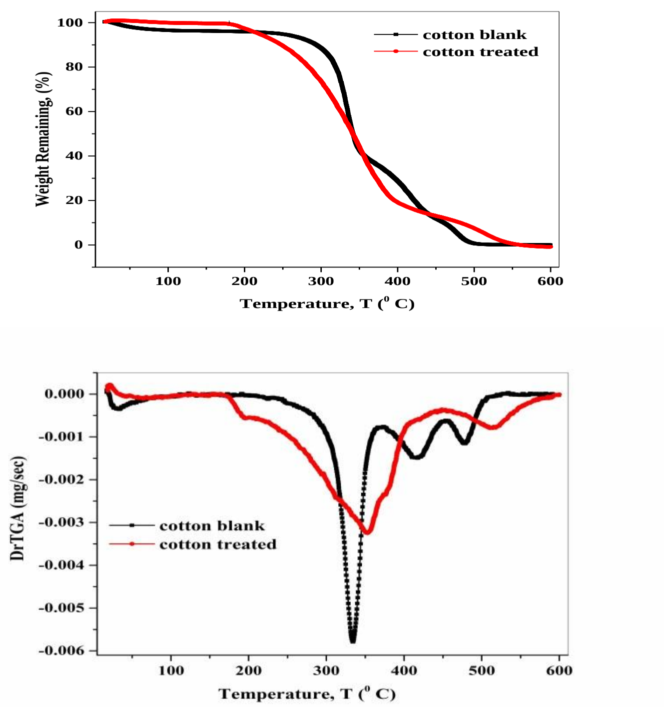


Fig. (5): TGA thermograms and the corresponding rate of thermal decomposition reaction curves of cotton fabrics, before and after coating with UMAc formulation under the effect of electron beam irradiation at dose (50kGy).

4. CONCLUSION

In this work, coating of cotton fabrics under the effect of electron beam curable UMAc formulations was performed to impart oil and water repellency for variable end-user applications. The thermal, mechanical properties, and water, aqueous liquid, and oil repellency were studied. The liquid repellency and thermal stability of treated cotton fabric are improved through coating process. The crease recovery and water vapor permeability of the coated fabric were also improved. The effect of EB irradiation dose on physical and mechanical properties has been studied. The TS values were increased up to 50 kGy of radiation dose and then tended to decrease, while the (E%) values of the treated cotton fabric were reduced with increase radiation doses. The water absorbance percent of treated fabrics was slightly decreased with increasing the radiation dose. The treated cotton fabrics display excellent water, aqueous liquids, and oils repellence; thereby they could be used in the furniture industry and tent materials.

ACKNOWLEDGMENT

The authors gratefully recognize the considerable financial attention for Egyptian Atomic Energy Authority (EAEA). This work stayed strengthened over National Centre for Radiation Research and Technology (NCRRT).

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