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Castor oil / 2,4-toluene di-isocyanate / polyethylene glycol adducts for cotton fabric functionalization

H.M. Fahmy[1], M. Ezzat[2], A. Amr[1]*

1 Pretreatment and finishing of fabrics based on cellulose department
2, Spinning and weaving engineering department,
Textile Research and Technology Institute, National Research Centre, Dokki, Cairo, Egypt.

Abstract

Castor oil (CAO) / 2,4-toluene di-isocyanate (TDI) / polyethylene glycols (PEG, 300, 600, 1000 and 2000 Da) adducts were prepared for finishing of cotton fabric. The obtained results revealed that the optimum conditions to prepare such adducts are reacting of equimolar ratio of PEG, TDI, and CAO at temperature of 100 ^{o}C/90 min. It is observed that only the adducts that based on PEGs 1000 and 2000 Da are self-dispersable and form stable oil in water emulsions. The FTIR spectra of the synthesized CAO/TDI/PEG1000 adduct confirms the chemical structure of that adduct while the TEM analysis clarified that its emulsion particles size ranges between 10 to 80 nm. Finishing of cotton fabric samples with bathes containing the produced CAO/TDI/PEG1000 or CAO/TDI/PEG2000 adducts emulsions leads to an enhancement in whiteness index, tensile work done, softness, and antibacterial properties parallel with a reduction in extents of the resiliency, wettability, in addition to the bending rigidity characteristics of treated fabric in a comparison to a control sample. Combining of such synthesized adducts emulsions with silver nano-particles in the finishing bathes significantly increases the antibacterial characteristics of the finished fabric. The treated fabric SEM as well as EDX images were investigated.

Key words: Castor oil; Polyethylene glycol; 2, 4-Toluene di-isocyanate; Textile softener; functional finishing.
Corresponding author: A. Amr, Email:amr1631970@gmail.com.

1. Introduction

Castor oil is natural oil extracted from castor seeds of Ricinus communis. The production of castor oil has increased in the last decades due to its easy availability, non-food competition, low cost, high viscosity, high boiling point, as well as other environmental considerations [1-7]. Castor oil consists mainly of ricinoleic (about 90%), linoleic, oleic, linolenic, and stearic fatty acids. The presence of hydroxyl and carboxylic groups, double bonds, in addition to the long hydrocarbon chains in the castor oil chemical structure offer many possibilities to modify it resulting in versatile products. Castor oil is commonly used in a lot of industrial applications like cosmetics, lubricants, emulsifiers, paints, varnishes, soaps, coatings, textile dyes, plastics, inks, etc….. [1-7].

On the other hand, cellulosic fabrics such as cotton and linen have excellent wearing properties including softness, high absorptivity and breathability put they are easily wrinkled, soiled, attacked by microbes, as well as of low protection against the harmful sun radiation. The matter that drives the R&D institutions as well as the textile manufacturers to develop the cellulosic fabrics to be have durable functional properties like softness, anti-UV radiation, anti-bacterial, self-cleaning, water repellency, and anti-crease properties [6-31].

Textile softeners are commonly used because of their merits of making harsh, pre-treated, colored fabrics comfortable to wear. In general, softeners can be classified as nonionic, anionic, cationic, and reactive. Nonionic softeners are recognized with their excellent compatibility with the other finishing bath constituents, effective lubricating effect and high resistance to yellowing. However, they have the limitation of low affinity for textiles and therefore they are normally applied using the padding technique. Moreover, they cannot form covalent bonds with the textile fibers which consequently limit their durability to washing [32-34].

The present work aims to:

*Corresponding author e-mail: amr1631970@gmail.com.
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a) obtain the proper conditions for reacting equimolar ratio of castor oil (CAO), polyethylene glycols (PEG, 300, 600, 1000 and 2000 Da), and 2,4-toluene di-isocyanate (TDI) to obtain CAO/TDI/PEG adducts,
b) characterize the obtained adducts as well as their water in oil emulsions, and
c) Apply the prepared adducts emulsions as softeners for cotton fabric finishing to provide the treated fabric with softness and antibacterial characteristics.

2. Experimental

2.1. Materials

Cotton fabric (mill scoured and bleached), weight of 128 g/m^2 and count (Ne) of 40/1, was provided by Misr Spinning and Weaving Company, Egypt. The castor oil (CAO) was provided by Iso-Chem. Co.. Polyethylene glycols (PEG, 300, 600, 1000 and 2000 Da), that dried at 30 °C on molecular sieves, were used. A low formaldehyde resin namely Durapret LF (DMDHEU) (70%) was utilized as a crosslinker. Di-butyl amine, 2,4-toluene di-isocyanate (TDI), toluene, and silver nitrate were used.

2.2. Methods

2.2.1. Preparation of the CAO/TDI/PEG adduct

The CAO/TDI/PEG adduct was easily prepared using a previous method [23,24] as follows: CAO and PEG was mixed well under dry nitrogen in a stoppered glass bottle at 80 °C in an oven followed by the addition of TDI that mixed well with the above mixture. After that the reaction was proceeded for a certain time at a certain temperature.

2.2.2. The CAO/TDI/PEG adduct separation

The CAO/TDI/PEG adduct produced at optimum conditions was separated from the other components for analysis as follows: isopropanol (300 ml) was added to of the produced adduct (10 g) in stopperd separating funnel and stirred well in order to dissolve all of the unreacted PEG, CAO, and TDI, followed that by filtration. This step of purification was reduplicated for an extra five times. The produced adduct which is the precipitate was then stored in a desiccator containing $CaCl_2$ for analysis [23,24].

2.2.3. Dispersing of the prepared adducts

To disperse any of the produced adducts, hot distilled water (70 °C) of a specific volume is added to the prepared adduct and then homogenized to form homogeneous emulsion that was then left to cool at room temperature [23,24].

2.3. Fabric finishing

Samples of cotton fabric (30X30 cm^2) were padded at 100 % wet pick up in finishing bathes including a certain concentration of the adduct emulsions, DMDHEU as a crosslinker, as well as ammonium sulfate as a catalyst. The padded samples were then dried (100 ^{o}C/3 min), cured (150 °C/3 min), washed with distilled water (50 ^{o}C/10 min), rinsed and then dried.

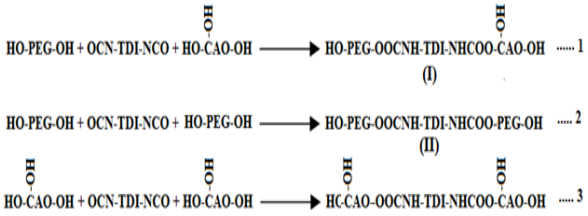
2.4. Test methods and analysis

- Fabric weight (W) was assessed according to ATSM (D 3776 – 79).
- The total conversion percentage (% TC) was evaluated using the ASTM (D 1638-T59).
- The resiliency of fabric samples (WRA) was assessed using the ASTM (D-1296-98).
- The surface roughness (SMD), bending rigidity (BR), and tensile work done (WT) of treated fabric was measured via Surface tester KES-FB4-A (Kawabata system), Japan.
- The fabric wettability was evaluated via the AATCC (39-1980).
- The UltraScan PRO - HunterLab was used to evaluate the fabric Whiteness Index.
- The antibacterial activities were estimated by the count method as mentioned elsewhere [2,3] against Staphylococcus aureus (Gram-positive bacteria) and Escherichia coli (Gram-negative bacteria).
- The FTIR analysis was performed using the spectrometer FT/IR-4700 FTIR.
- The particles size of adduct emulsion particles were determined through TEM analysis by using the electron microscope JEOL, JEM 2100 F.
- The SEM images of fabric samples were obtained using SEM Model Quanta 250 FEG attached with EDX Unit.

3. Results and Discussion

3.1. Tentative mechanism

Previous literatures confirmed the reaction between the TDI isocyanate groups and the aliphatic compounds primary hydroxyl groups producing urethane groups. Therefore, it is predicted that the reacting of PEG, CAO, and TDI with equimolar ratios under appropriate conditions would result in different products include compounds I, II, and III (equations 1 – 3) as well as marginal amounts of un-reacted starting materials in addition to extended chains of compounds I, II and III [24,34]. The castor oil grafted with PEG, i.e. compound I, represents as a self-emulsifier for the other reaction components beside of course the un-reacted PEG and compound II.



The above reaction products will pointed out by CAO/TDI/PEG adduct whereas the produced adduct will be named according to PEG molecular weight. For example the CAO/TDI/PEG1000 adduct is due to PEG of MW 1000 Da. Moreover, dispersing of the produced adducts in water causes elimination of the un-reacted isocyanate groups toxicity of TDI as indicated by equation 4 [24,34]:

$$-NCO + H_2O \rightarrow -NH_2 + CO_2 \quad \ldots\ldots 4$$

3.2. Factors influencing the CAO/TDI/PEG adduct formation

3.2.1. Reaction time and temperature

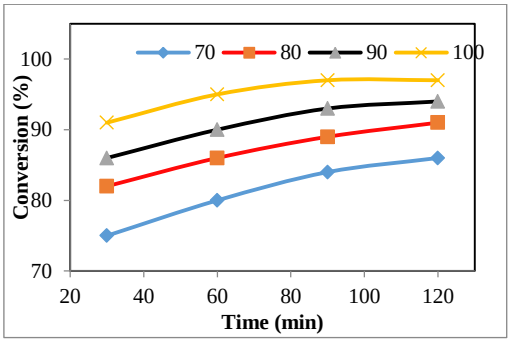


Figure 1: The impact of temperature and time on total conversion percentage of TDI.

Figure 1 shows the impact of the reaction time and temperature on percent conversion of the TDI isocyanate groups to produce urethane groups upon reaction of PEG 1000, TDI, and CAO with a molar ratio of 1:1:1 at different time intervals (30-120 min) and degrees of temperature (70 – 100 ^{o}C). It is shown that: (i) at constant temperature, the further increasing in the reaction time leads to an increasing in the conversion percentage, and (ii) at constant reaction time, raising the temperature significantly upgrades the % conversion. From Figure it can be deduced that the optimal conditions to react TDI with PEG 1000 and CAO are the temperature of 100 ^{o}C and the time of 90 min [24,34].

3.2.3. PEG molecular weight

Table 1: The impact of PEG molecular weight on TDI conversion and the adducts emulsion state.

PEG mol. wt. (Da)	Conv. (%)	Emulsion state
300	99.1	No emulsion
600	98.4	Emulsion with large oil drops
1000	97.2	Stable emulsion
2000	96.8	Stable emulsion

PEG/TDI/CAO molar ratio, 1:1:1; reaction temperature, 100 ^{O}C; reaction time, 90 min.

Table 1 reveals the impact of PEG molecular weight on TDI percent conversion and the produced CAO/TDI/PEG adducts emulsion state. It is clearly seen that among the prepared adduct, only the CAO/TDI/PEG1000 and CAO/TDI/PEG2000 adducts form stable emulsions. The un-dispersibility of CAO/TDI/PEG300 adduct and instability of CAO/TDI/PEG600

adduct emulsion may be a direct consequence for the insufficient hydrophilic moieties of compound I (Equation 1), i.e. the castor oil grafted with PEG300 or PEG600 respectively, as a self-emulsifier, the matter that renders it unable to disperse the formed adduct and form a successive emulsion [13,31,24,34]. Moreover, increasing of molecular weight of PEG leads to a lowering in percent conversion of the formed adducts, the matter that can associated with increasing of entanglement of the PEG chains and the reaction medium viscosity [13,31,24,34]. Figure 2 shows visually an image for the CAO/TDI/PEG1000 adduct emulsion.

Figure 2: An image for the CAO/TDI/PEG1000 adduct emulsion.

3.3. Characterization of the adduct and its emulsion

3.3.1. FTIR

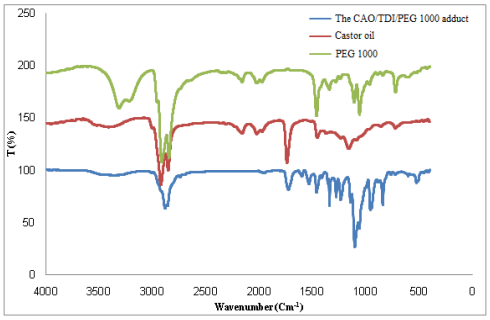


Figure 3: FTIR of CAO/TDI/PEG1000 adduct, PEG, and castor oil.

Figure 3 represents the FTIR of CAO/TDI/PEG1000 adduct, PEG, and castor oil. It is clear that the castor oil spectrum includes peaks at 3470 cm^{-1} assigned to -OH stretching vibration, 2907 and 2858 cm^{-1} assigned to -CH stretching vibration, 1746 cm^{-1} assigned to C=O stretching vibration, as well as 1464 cm^{-1} corresponding to castor oil C=C [35]. Moreover, the spectrum of PEG 1000 includes peaks at 3334 cm^{-1} due to -OH stretching, 2919 and 2853 cm^{-1} assigned to aliphatic -CH stretching vibration, 1468 cm^{-1} assigned to $-CH_2$ binding vibration, 1276 and 1355 cm^{-1} assigned to the PEG 1000 C-O and C-C bonds respectively, and 1118 cm^{-1} of C-O-C stretching vibration [3,13,24]. Furthermore, the purified CAO/TDI/PEG1000 adduct spectrum includes a broad band at 3664 - 3222 cm^{-1} assigned to -OH of either castor oil and PEG as well as –NH stretching vibrations of the urethane groups, 2991 and 2892 cm^{-1} due to PEG and castor oil -CH stretching vibration, 1741 cm^{-1} due to castor oil C=O stretching vibration, 1630 cm^{-1} assigned to –C=O of urethane groups [36], 1544 cm^{-1} assigned to –CN stretch of urethane group [36], 1108 cm^{-1}, corresponding to C-O-C stretching of PEG, 960 cm^{-1} assigned to C-H of the TDI benzene ring [3,13,24] which clearly proves the prepared CAO/TDI/PEG1000 adduct formation.

3.3.2. TEM image of CAO/TDI/PEG1000 adduct emulsion

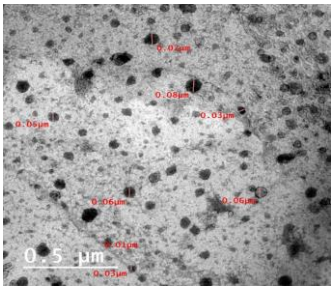


Figure 4: TEM image of CAO/TDI/PEG1000 adduct emulsion.

The CAO/TDI/PEG1000 adduct emulsion was described through TEM analysis as illustrated in Figure 4. It is well seen that the CAO/TDI/PEG adduct emulsion particles size ranges between 10 to 80 nm.

3.4. Inclusion of CAO/TDI/PEG adduct emulsion in finishing of cotton fabric

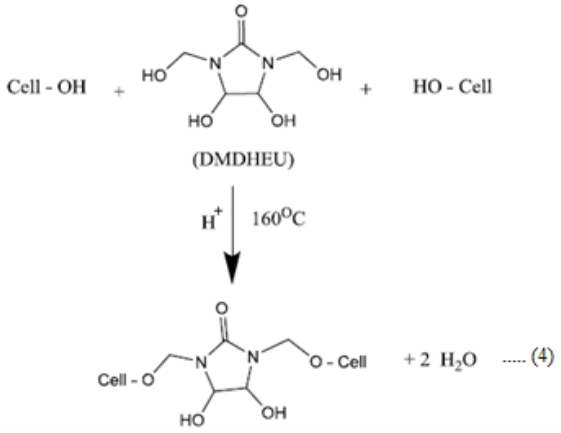
3.4.1. Performance characteristics of treated fabric

Table 2: The impact of CAO/TDI/PEG adduct emulsion concentration type and concentration on fabric performance characteristics.

Adduct Type	Adduct conc. (%)	WRA (w + f)°	W (S)	WI (%)	(TWD) (gf*cm/cm²)	BR (gf*cm²/cm)	SMD (µ)
Untreated Control	0 0	151 260	1 2	71.32 64.74	13.71 10.98	0.0397 0.0498	1.136 1.623
CAO/TDI/1000	2 4	245 237	4 5	68.1 68.4	11.76 12. 38	0.0459 0.0476	1.452 1.415
CAO/TDI/2000	2 4	239 230	5 6	68.6 68.9	12.47 13.01	0.0484 0.0488	1.372 1.277

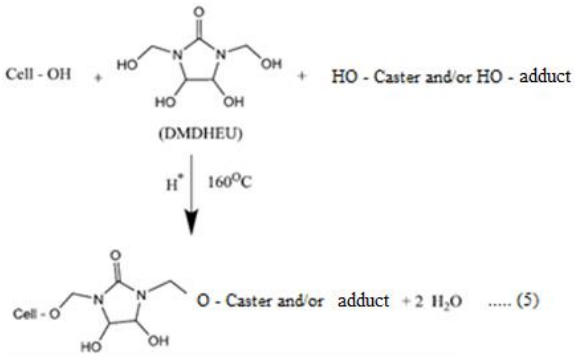
[DMDHEU], 60 g/L; [$(NH_4)_2SO_4$], 6 g/L; drying, 100 °C/3 min; curing, 160 °C/3 min; wet pick up, 100 %. Control fabric sample is a sample treated in an absence of the prepared adduct emulsion. WRA: wrinkle recovery angle (weft+warp)°; W: wettability (per seconds); WI: whiteness index; TWD: tensile work done; BR: bending rigidity; SMD: surface roughness.

Table 2 illustrates the performance characteristics of fabric samples finished with finishing bathes include 20 and 40 g/L of the CAO/TDI/PEG1000 or CAO/TDI/PEG2000 adducts emulsions. It is obvious that: i) treating of fabric samples with easy care finishing bath in absence of the prepared emulsions, i.e. the control sample, results in an enhancement in surface roughness, bending rigidity, and resiliency parallel with a reduction in wettability, tensile work done, and whiteness index characteristics of treated fabric, with respect to a blank fabric. This can be associated with crosslinking of cotton cellulose with DMDHEU as a crosslinker (equation 4) [2,3,13,19,24]:

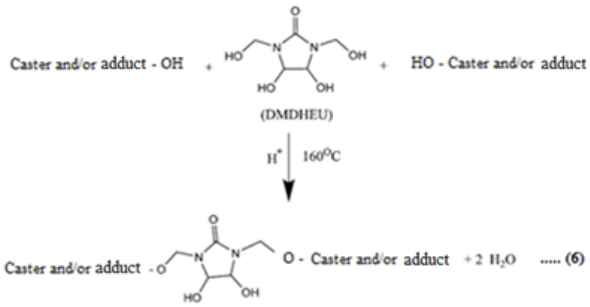


ii) introducing of the nominated adducts emulsions in the above finishing formulations results in an improvement in extents of whiteness index, tensile work done, and softness parallel to a reduction in wettability, resiliency, and bending rigidity characteristics of treated fabric in comparison to the control fabric sample reflecting the fixation of such adducts components onto/within the finished samples structures as represented by the next equations [2,3,13,19,24]:

a) Fixation of ingredients of the prepared adducts onto/within cellulose of the cotton fabric



b) Deposition of ingredients of the prepared adducts onto/within cellulose of cotton fabric



iii) increasing of emulsion concentration of such adducts in the finishing bath leads to an upgrading of extents of softness, whiteness index, tensile work done, and bending rigidity, accompanied with a lowering in extents of wettability and resiliency characteristics of treated fabric suggesting the increasing of extent of binding of the aforementioned adducts components to the finished fabric samples structures [2,3,13,19,24], and iv) increasing molecular weight of PEG of the adduct causes an improvement in extents of softness, whiteness index, tensile work done, and bending rigidity parallel to a decreasing in resiliency as well as wettability characteristics of treated fabric.

3.4.2. The antibacterial characteristics of CAO/TDI/PEG adducts emulsions finished fabric

Table 3: The antibacterial characteristics of CAO/TDI/PEG adducts emulsions finished fabric.

Easy care finishing formulation	Reduction (%)	
	G -ve	G+ve
Untreated	0	0
CAO/TDI/PEG1000 emulsion (20 g/L)	52(45)	58(50)
CAO/TDI/PEG1000 emulsion (40 g/L)	61(54)	67(59)
CAO/TDI/PEG2000 emulsion (40 g/L)	81(73)	85(76)
CAO/TDI/PEG2000 emulsion (40 g/L)/Ag-NPs (1%)	89(80)	97(87)

[DMDHEU], 60 g/L; [$(NH_4)_2SO_4$], 6 g/L; wet pick up, 100 %; drying, 100 ^oC /3 min; curing, 160 ^oC /3 min. Values in parentheses reveal the antibacterial characteristics of treated fabric after 15 washing cycles.

Table 3 illustrates the antibacterial characteristics of cotton samples treated with easy care finishing formulations include different concentrations of the CAO/TDI/PEG1000 and CAO/TDI/PEG1000 adducts emulsions in presence or absence Ag-NPs. It is clear that: i) treating cotton samples with either of the CAO/TDI/PEG1000 or CAO/TDI/PEG1000 adducts emulsions, regardless of their concentration, acquires the treated fabric antibacterial characteristics against E. coli (G-ve) and S. aureus (G+V) bacteria reflecting the castor oil [1-3], PEG 1000 [37,38], and PEG 2000 [37,38] antibacterial properties ii) the antibacterial characteristics of cotton sample finished with CAO/TDI/PEG2000 adduct emulsion is of higher extent than that treated with the CAO/TDI/PEG1000 adduct emulsion, iii) increasing the adducts concentration, irrespective of the adduct type, enhances the antibacterial characteristics of treated fabric, suggesting increasing of fixation of such adducts components

onto/within the treated samples structures, iv) introducing of Ag-NPs in the above finishing bathes remarkably promotes the antibacterial characteristics of treated fabric reflecting the positive impact of Ag-NPs as a bio-additive, in addition to the castor oil species, on harming the bacterial membrane leading to cell death, formation of Ag^+ ions, in the presence of moisture, that can bind to the bacterial DNA causing their inactivation according to equation (7):

$$O_2 + 4H_3O^+ + 4Ag \rightarrow 4Ag^+ + 6H_2O \quad \ldots\ldots (7)$$

and/or formation of radicals of oxygen which oxidize the bacterial molecular structure as clear from equation (8) [39-44]:

$$H_2O + (1/2)O_2 \xrightarrow{Ag^+} H_2O_2 \rightarrow H_2O + (O) \quad \ldots\ldots (8)$$

, and v) the antibacterial characteristics of finished fabric are durable for 15 washing cycles.

3.4.3. SEM and EDX images of the CAO/TDI/PEG1000 adduct emulsion finished fabric

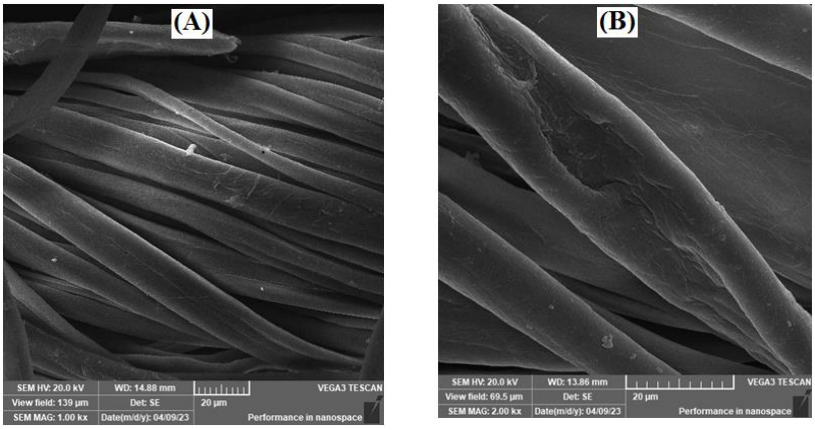


Figure 5: SEM images of untreated fabric sample (A) and CAO/TDI/PEG1000 adduct emulsion finished fabric sample (B).

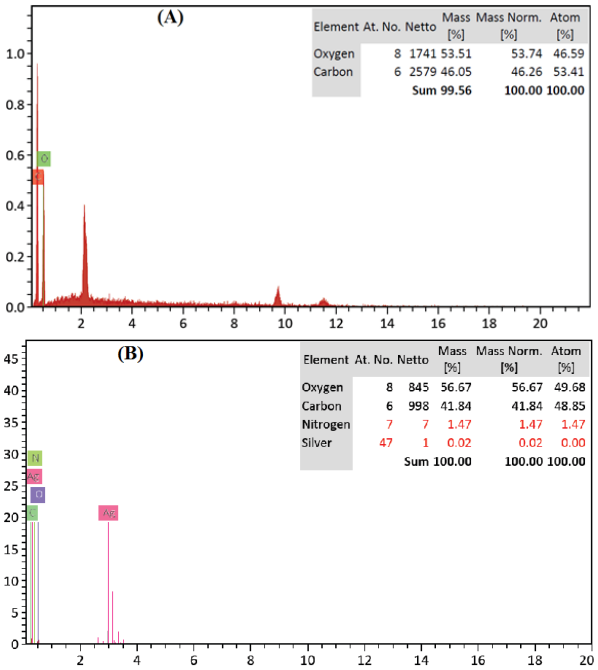


Figure 6: EDX images of untreated fabric sample (A) and CAO/TDI/PEG1000 adduct emulsion finished fabric sample (B).

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Figure 5 indicates the SEM images of untreated fabric sample and cotton fabric finished with a finishing bath includes DMDHEU (60 g/l), ammonium sulfate (6 g/L), the prepared emulsion (40 g/L) and Ag-NPs nanoparticles (2 g/L). It is observed that the SEM of the finished sample confirms the deposition of a film of the CAO/TDI/PEG1000 adduct ingredients on surface of the finished fabric which is not present in SEM image of the untreated sample. On the other hand, Figure 6

represents the EDX images of such fabric samples. It is clearly seen that, the EDX image of the finished fabric includes the elements of O, N, C, and Ag while that of the untreated sample includes only the elements of O and C [45-50].

3.5. Storage stability of the adduct emulsion

To examine the storage stability of the CAO/TDI/PEG1000 adduct emulsion, 300 ml of the emulsion was kept in a stoppered 500 ml conical flask under the normal lab conditions for one month. After, about three weeks, the emulsion starts to separate into two layers, the upper layer was the adduct emulsion whereas the bottom was turbid water. Upon shaking, a homogeneous emulsion was formed again.

4. Conclusions

- The finest conditions to prepare the CAO/TDI/PEG adducts are reacting equimolr of CAO, TDI and PEG at 100 ^{o}C/90 min.
- Among the prepared adducts, only the adducts containing PEG of molecular weight 1000 or 2000 Da are self dispersable and form stable oil in water emulsions.
- The synthesized adduct chemical structure was assured via the FTIR analysis.
- The TEM image showed that the CAO/TDI/PEG1000 adduct emulsion particles size is between 10 to 80 nm.
- Finishing of cotton fabric with the aforementioned adducts emulsions increases whiteness index, tensile work done, softness, and antibacterial characteristics in addition to a decreasing in wettability, resiliency, and bending rigidity characteristics of the fabric.
- Introducing of Ag-NPs as well as the adduct emulsion in the finishing bath increases the antibacterial characteristics of the finished fabric.

Data availability

The datasets used and/or analyzed during the current study are available from the corresponding author on reasonable request

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Author contributions Conceptualization
All authors have read and agreed to the published version of the manuscript

Competing interests
The authors declare no conflicts of interest.

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