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Synthesis of castor oil/poly (N-vinyl-2-pyrrolidone)/ammonium persulfate hybrids emulsion as textile softeners

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Abstract

Two castor oil / poly (N-vinyl-2-pyrrolidone) / ammonium persulfate (CAO/PVP/APS) hybrids were synthesized via reaction of castor oil with PVP of molecular weights 40000 and 1300000 Dalton in presence of APS as an initiator. The optimum reaction conditions are: PVP/CAO weight ratio, 15%; PVP concentration, 60%; APS conc., 4% (based on weight of CAO); reaction temperature, 80 °C; reaction time, 60 min. The synthesized CAO/PVP/APS hybrids were characterized via FTIR analysis whereas their emulsions particle sizes were determined using TEM analysis. Incorporation of either of the aforementioned hybrids emulsions in the easy care finishing formulations of cotton fabric results in promoting of fabric softness, tensile strength, flexibility, and antibacterial activities of treated fabric along with reducing fabric wettabiliy and resiliency. Inclusion of Ag-NPs in the aforementioned finishing bath significantly improves extent of the antibacterial activities of treated fabric. The treated fabric morphology was examined using SEM as well as EDX analysis.

Keywords Castor oil · Poly (N-vinyl-2- pyrrolidone) · Emulsion · Textile functional finishing

Introduction

Textile finishing is usually performed as a last step to impart a textile material with functional properties such as water and oil repellency [1–5], resistance to crease [6–8], resistance to microbes [9, 10], protection from UV radiation [11, 12], self-cleaning [13], pleasant scent [14], hand building [15], and softness [16].

Textile softener is a finishing agent improves the textile handle giving rise to a pleasing touch upon applying it to the textile material. In general, the textile softener is a lubricating agent that facilitates fiber slippage inside the fabric structure. Textile softeners can be broadly classified into five main types according to their chemical structure that are silicone, cationic, anionic, nonionic, and amphoteric softeners. A wide range of finishing agents is available in the market to satisfy the consumer's demands [16–19]. The textile finishing future trend is developing textiles to have multifunctional properties that are efficient, cost effective,

A. Amr amr1631970@gmail.com durable, and safe for both the consumer and the environment [10, 12, 13, 16].

Nowadays, researchers are looking for plant oils such as castor oil, soybean oil, olive oil, and linseed oil as renewable resources for using in various industries such as cosmetics, lubricants, paints, coats, etc. [1, 20–23]. Among the plant oils, castor oil occupies unique standing due to its chemical structure containing double bonds, hydroxyl groups, and carboxyl groups of the ricinoleic acid (Fig. 1) [1, 20] which is the main component of castor oil in addition to linoleic, oleic, and stearic acids. The chemical structure of castor oil offers a wide range of applications including lubricants, varnishes, paints, inks, cosmetics, plastics, textile dyes, and soaps [1, 20–23].

Emulsions of castor oil are commonly used in cosmetics, pharmaceutical products, and food industries. The castor oil emulsions can deliver the hydrophilic and lipophilic drugs [24]. Castor oil can be emulsified by means the normal emulsifiers in addition to many gums such as Arabic gum as well as Terminalia gum [25, 26]. A formula consists of polyoxyethylene sorbitan monostearate and sorbitan monooleate can emulsify castor oil as reported by Zhang et al. [27].

In the textile field, many textile auxiliaries were synthesized using castor oil as a raw material. The Turkey red oil which is the sulphated castor oil was used as an

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$$\begin{array}{c} O & OH \\ H_{3} - O - C - (CH_{2})_{7} - CH = CH - CH_{2} - CH - (CH_{2})_{5} - CH_{3} \\ O & OH \\ H_{2} - O - C - (CH_{2})_{7} - CH = CH - CH_{2} - CH - (CH_{2})_{5} - CH_{3} \\ O & OH \\ H_{3} - O - C - (CH_{2})_{7} - CH = CH - CH_{2} - CH - (CH_{2})_{5} - CH_{3} \\ \end{array}$$

Fig. 1 Chemical structure of castor oil [20]

anionic textile softener or wetting agent depending on the sulphation degree of castor oil [28]. Nair et al. utilized the coconut Oil, vegetable Ghee, and castor oil to produce eco friendly textile softeners [29]. A new textile softener was synthesized by the thermal treatment for a mixture of poly (N-vinyl-2-pyrrolidone) (PVP) and castor oil at 150 °C for 1 h [30]. Castor oil based water repellent agent was prepared using 2,4-toluene di-isocyanate as a crosslinker linker followed by emulsification the product with stearic acid/triethanol amine emulsifying system [1].

On the other hand, PVP is a nontoxic, water-soluble, and synthetic biocompatible polymer. It is a suspending agent, protective colloid, clarifying agent, stabilizer, film former, viscosity modifier, and complexing agent. It is commonly introduced in many industrial applications as suspending agent, emulsion stabilizer, film former, and binder. PVP can be crosslinked through heating in presence of persulfates or at 150 °C [7, 8, 31, 32].

Keeping the above background in mind, the present work aims to emulsify castor oil using poly (N-vinyl-2pyrrolidone)/ammonium persulfate system and apply the produced emulsion for functional finishing of cotton fabrics.

Experimental

Materials

Mill scoured and bleached cotton fabric of weave structure, weight of 128 g/m² and count (Ne) of 40/1 was supplied by Misr Spinning and Weaving Co., Mahalla El—Kobra, Egypt. Castor oil (CAO) supplied by Iso-Chem. Company was used. Durapret LF (DMDHEU), low formaldehyde resin of conc. 70%, was used as a crosslinker for easy care finishing of cotton fabric. Poly (N-vinyl-2-pyrrolidone) (PVP) of molecular weights 40,000 and 1,300,000 Dalton was supplied by Alpha chemiKa. Silver nitrate, ammonium persulfate (APS) and ammonium sulfate (AS) of laboratorygrade chemicals were used.

Preparation of CAO /PVP/APS hybrid emulsion

The CAO/PVP hybrid emulsion was prepared as follows: 5 g of CAO and PVP, as 60% aqueous solution, in a specific weight ratio to CAO (0–20%) was mixed together in 150 ml beaker with stirring at different temperatures (60–90 °C). APS solution of specific concentration was then added to the reaction medium and the reaction was left to proceed under stirring for a period of time (0–90 min). After a certain time, specific volume of hot distilled water at ca 70 °C was added to the reaction medium followed by stirring using a strong homogenizer for 3 min to form a homogeneous CAO/PVP hybrid emulsion [2, 4].

Preparation of silver nano-particles (Ag-NPs)

Silver nano-particles (Ag-NPs) were prepared using trisodiumcitrate as a reductant as reported elsewhere [33].

Fabric treatment

Cotton fabric samples of $30X30 \text{ cm}^2$ were padded twice in finishing bathes containing 60 g/L of DMDHEU, ammonium sulfate as a catalyst and different concentrations of CAO/PVP hybrid emulsion (0—40 g/L), to a wet pick up of 100%. The padded samples were dried at 100 °C/3 min in Wenner Mathis AGCH-8155 oven followed by curing at 150 °C/ 3 min. The finished fabrics were then washed with distilled water at 50 °C for 10 min, thoroughly rinsed and finally dried for testing.

Analysis and test methods

- Fabric weight (W) was assessed according to ATSM (D 3776 79).
- The wrinkle recovery angle of treated fabric samples, WRA, was determined according to ASTM method D-1296–98.
- Tensile strength (TS) was tested in the warp direction according to ASTM procedure D-2256–98.
- Wettability of the finished fabric sample was assessed according to AATCC Test method 39–1980.
- The flexural rigidity test (FR) was carried out in the warp direction according to ASTM D 1388–64.
- Surface roughness (SMD) was measured using Kawabata evaluation system, Surface tester KES-FB4-A, Kato Tech Co., LTD, Japan.
- The antibacterial activity was assessed using the bacterial count method as reported elsewhere [12, 34, 35] against:

- Gram-positive bacteria: Staphylococcus aureus (SA).
- Gram-negative bacteria: Escherichia coli (EC).
- Infra-Red (IR) spectroscopy was carried out using FT/ IR-4700 FTIR Spectrometer from JASCO.
- The morphology and particles size of the hybrid emulsion was obtained by transmission electron microscope (TEM) using a JEOL, JEM 2100 F electron microscope at 200 kV.
- Scanning electron microscope (SEM) images of the treated and untreated fabric samples were obtained using SEM Model Quanta 250 FEG (Field Emission Gun) attached with EDX Unit (Energy Dispersive X-ray Analyses), with accelerating voltage 30 kV, magnification 14×up to 1,000,000 and resolution for Gun, FEI company, Netherlands.

Results and discussion

Tentative mechanism

It was reported that heating of APS aqueous medium causes the decomposition of APS to free radical species (SO_4^{-1} and HO) [1, 4, 7, 36, 37]. Thus, it is anticipated that introducing of PVP as well as castor oil in the previous reaction may cause the following reactions:

$$S_2 O_8^{2-} \to 2 S O_4^{--} \tag{1}$$

$$SO_4^- + H_2O \rightarrow HSO_4^- + OH^-$$
 (2)

$$\begin{array}{ccc} \overset{H}{\leftarrow} & \overset{H}{\leftarrow$$

$$\frac{\text{CAO} + \text{R}^{\circ}}{(\text{Castor oil})} \rightarrow \frac{\text{CAO}^{\circ}}{(\text{Castor oil radicals})} + \text{RH}$$
(4)

$$\begin{array}{ccc} CAO^{\bullet} & + & (CH - \bullet)_{n} & \longrightarrow & CAO - PVP \\ & & & & \\ & & & & \\ & & & \\ & & & \\ & & & \\ & & & \\ & & & \\ & & & \\ & & & & \\ &$$

PVP – PVP

(5)

$$PVP' + PVP' \rightarrow (Crosslinked PVP)$$
(6)

The aforementioned reaction contains a mixture of unreacted castor oil and PVP, castor oil grafted with PVP, crosslinked PVP in addition to other oxidized species; all in state of entanglement and will be donated by CAO/PVP/APS

Table 1 Effect of APS concentration on CAO/PVP/APS hybrid emulsion state

APS/CAO (%)	Emulsion state		
1	No emulsion		
2	Emulsion with large oil drops		
3	Emulsion with tinny oil drops		
4	Stable emulsion		
5	Stable emulsion		

PVP MW, 40000 (Dalton); PVP/CAO weight ratio, 20%; PVP conc., 60%; reaction time, 60 min; reaction temp., 80 °C

hybrid. Indeed, the hydroxyl groups as well as double bonds of castor oil act as grafting centers [38–40]. Moreover, the castor oil grafted with PVP may serve as a self emulsifier for the unreacted CAO whereas the crosslinked PVP act as a stabilizer for the resulted emulsion [40]. The major factors affecting that hybrid formation will be studied as follows.

Factors affecting the CAO/PVP/APS hybrid emulsion formation

APS concentration

Table 1 illustrates the impact of APS/CAO weight ratio on the CAO/PVP/APS hybrid emulsion state. It is obvious that, increasing APS/CAO weight ratio from 1 and up to 4% is accompanied with a gradual improvement in state of the hybrid emulsion which could be attributed to the gradual increasing in magnitude of the free radical species capable to form CAO/PVP species (Eq. 5) responsible for emulsification of the remaining CAO in the reaction medium [2–4]. Increase of the APS/CAO ratio in the reaction medium, within the range studied, does not affect the emulsion state, suggesting a rapid rate of termination due to the faster rate of initiation at the higher concentrations of APS [36, 37].

PVP/CAO weight ratio

Table 2 shows the effect of the PVP/CAO weight ratio, keeping the PVP concentration at 60%, on state of the CAO/PVP/APS hybrid emulsion. It is clear that increasing

 Table 2
 Effect of PVP/CAO weight ratio on CAO/PVP/APS hybrid emulsion state

PVP/CAO weight ratio	Emulsion state		
5	Emulsion with large oil drops		
10	Emulsion with tinny oil drops		
15	Stable emulsion		
20	Stable emulsion		

PVP MW, 40000 (Dalton); PVP conc., 60%; APS conc., 4% (based on weight of CAO); reaction time, 60 min; reaction temp., 80 °C

of the PVP/CAO ratio to 15% in the reaction medium results in a progressive improvement in the emulsion state which may be associated with the enhancement in CAO/ PVP species formation capable to emulsify that hybrid [2–4]. The further increasing in such ratio, i.e. at 20%, does not change the hybrid emulsion state.

Reaction time

Table 3 reveals the state of the CAO/PVP/APS hybrid emulsion as a function of reaction time. It is clearly seen that increasing the reaction time up to 60 min brings about a gradual promotion in emulsion state of the hybrid, the matter that can be explained by the enhancement in APS decomposition and the subsequent increasing in CAO/PVP species formation that ultimately promotes the hybrid emulsion state. Beyond 60 and up to 90 min, the hybrid emulsion remains stable suggesting a depletion in both the PVP and/or APS concentrations [2–4, 36, 37].

Reaction temperature

The effect of the reaction temperature on emulsion state of the CAO/PVP/APS hybrid is shown in Table 4. It is well seen that raising the reaction temperature from 60 to 80 °C results in an upgrading the state of the hybrid emulsion, which can be associated with the enhancement in the initiator decomposition and the subsequent increasing in the free radicals number as well as increasing of mobility of the reactants molecules that subsequently enhances the CAO/PVP species formation responsible for emulsification of the unreacted CAO [2–4, 36, 37]. Moreover, increasing of the reaction temperature from 80 to 90 °C does not affect state of that hybrid emulsion suggesting a faster rate of termination at higher temperatures [36, 37].

 Table 3
 Effect of reaction time on CAO/PVP/APS hybrid emulsion state

Emulsion state		
No emulsion		
Emulsion with large oil drops		
Emulsion with tinny oil drops		
Stable emulsion		
Stable emulsion		

PVP MW, 40000 (Dalton); PVP/CAO weight ratio, 15%; PVP conc., 60%; APS conc., 4% (based on weight of CAO); reaction temp., 80 °C

Table 4	Effect	of	reaction	temperature	on	CAO/PVP/APS	hybrid
emulsio	n state						

Reaction temperature (°C)	Emulsion state		
60	Emulsion with large oil drops		
70	Emulsion with tinny oil drops		
80	Stable emulsion		
90	Stable emulsion		

PVP MW, 40000 (Dalton); PVP/CAO weight ratio, 15%; PVP conc., 60%; APS conc., 4% (based on weight of CAO); reaction time, 60 min

PVP Molecular weight

The emulsion state of the CAO/PVP/APS hybrids prepared using PVP of molecular weights 40000 and 1300000 Dalton is shown in Table 5. It is clear that increasing of the PVP molecular weight over the range of 40000 to 1300000 Dalton results in formation of CAO/PVP/APS hybrids having stable emulsions. Moreover, upon storing both the hybrids emulsions for studying their emulsions stability, it was noticed that after about two weeks, tinny oil drops appeared on the CAO/PVP40000/APS hybrid emulsion surface which by shaking the emulsion, such oily drops disappeared and stable emulsion was obtained again. On the other hand, storing the CAO/PVP1300000/ APS hybrid emulsion for one month does not change its visual appearance reflecting its higher stability compared to the CAO/PVP40000/APS hybrid emulsion.

Characterization of the of CAO/PVP/APS hybrids and their emulsions

IR of the CAO/PVP/APS hybrid

Figure 2 shows the FTIR analysis of CAO/PVP40000/APS hybrid, castor oil, and PVP. It is clear that the hybrid spectrum shows the following peaks:

1. Peaks resembles to that of PVP spectrum that are [2, 4, 7, 8]:

 Table 5
 Effect of PVP molecular weight on CAO/PVP/APS hybrid emulsion state

PVP molecular weight (Dalton)	Emulsion state	
40000	Stable emulsion	
1300000	Stable emulsion	

PVP/CAO weight ratio, 15%; PVP concentration, 60%; APS conc., 4% (based on weight of CAO); reaction temperature, 80 °C; reaction time, 60 min



Fig. 2 FTIR spectra of CAO/PVP/APS hybrid, castor oil, and PVP

- A peak at 2923 cm⁻¹ due to stretching vibration of -CH of PVP,
- A peak at 1276 cm⁻¹ due to C–N bond of PVP, and
- A peak at 1666 cm⁻¹ due to the stretching vibration the C=O group of both PVP and the crosslinked PVP.
- 2. Peaks resembles to that of castor oil spectrum that are [1, 41–44]:
 - A broad stretching vibration band at 3397 cm⁻¹ due to OH group,
 - A stretching vibration band at 3007 cm⁻¹ due to C=C,
 - symmetric and asymmetric stretching vibration bands at 2923 and 2857 cm⁻¹ respectively due to the of CH₂,
 - A stretching band at 1739 cm^{-1} due to C = O,
 - Asymmetric and symmetric vibrations of -CH₃ groups at 1450 and 1369 cm⁻¹ respectively,
 - Stretching vibration bands at 1238 and 1160 cm⁻¹ due to C–O of ester group, and

TEM images of the CAO/PVP/APS hybrids emulsions

Figure 3(A) and (B) shows the particles size of CAO/PVP40000/ APS and CAO/PVP1300000/APS hybrids emulsions respectively determined by the transmission electron microscope (TEM). It is clear that the particles size of CAO/PVP40000/ APS hybrid emulsion ranges approximately from 15 to 50 nm whereas that of CAO/PVP1300000/APS hybrid emulsion ranges from 270 to 350 nm suggesting that the higher the PVP molecular weight, the larger is the hybrid emulsion particle size.

Inclusion of the CAO/PVP/APS hybrids emulsion in easy care finishing of cotton fabric

Performance properties of treated fabric

Table 6 depicts the impact of inclusion of CAO/PVP40000/ APS and CAO/PVP1300000/APS hybrids emulsions in easy care finishing bath of cotton fabric on some performance properties of treated fabric. It is well seen that crosslinking of cotton fabric samples in absence of that hybrids emulsions gives rise to an enhancement in resiliency accompanied with a reduction in tensile strength, softness degree, flexibility, i.e. higher rigidity, and wettability of treated fabric, compared with the untreated fabric. The matter that could be attributed to crosslinking of cotton cellulose with DMDHEU as a crosslinker and the subsequent molecular degradation in the cellulose structure [2–5].

Meanwhile, inclusion of either of the aforementioned hybrids emulsions in the above mentioned finishing bath brings about a fixation of such hybrids ingredients onto/ within the treated fabric structure, via the labile hydrogen atom of the castor oil or its hybrid hydroxyl groups, [2–6] which subsequently results in promoting fabric softness, tensile strength and flexibility due the lubricating effect of the hybrid ingredients as well as reducing fabric wettabiliy and resiliency as a direct consequence for the decreasing in

Fig. 3 TEM images of A CAO/ PVP40000/APS and B CAO/ PVP1300000/APS hybrids emulsions



Table 6Some performanceproperties of the CAO/PVP/APS hybrids emulsion treatedcotton fabric

PVP MW (Dalton)	Soft. conc (%)	WRA (W+F) ^O	TS (Kg)	W (S)	SMD (µ)	FR (µN.m)
Untreated	0	138	50	2	1.799	4.2
Control	0	220	32	3	1.927	5.1
40000	3	190	35	8	1.813	3.4
	5	187	37	11	1.711	3.6
1300000	3	183	37	12	1.788	3.6
	5	174	38	16	1.620	3.9

Fabric finishing conditions: The fabric samples were padded in finishing bath containing 60 g/L of DMDHEU, 4 g/L of AS and a specific conc. of the prepared emulsions to a wet pick up of 100%; drying at 100 $^{\circ}$ C /3 min; curing, 160 $^{\circ}$ C /3 min. Control sample is a sample treated with an easy care finishing bath in absence of the prepared emulsions

WRA wrinkle recovery angle, TS tensile strength, W wettability, SMD surface roughness, FR, flexural rigidity

extent of crosslinking with the DMDHEU [6]. On the other hand and regardless of the hybrid type, increasing of the hybrid emulsion concentration in the aforementioned finishing bath leads to an enhancement in extents of the above mentioned properties suggesting the increasing in extent of fixation of such hybrids ingredients onto/within the treated fabric structure. Moreover, replacing the CAO/PVP40000/ APS hybrid emulsion with the CAO/PVP1300000/APS hybrid emulsion in the finishing bath significantly enhances softness, tensile strength along with decreasing resiliency, wettability, and flexibility of treated fabric compared with the CAO/PVP40000/APS hybrid emulsion treated fabric which can be attributed to the difference between such hybrids in the PVP molecular weight [4].

Antibacterial activities of treated fabric

Table 7 shows the impact of the finishing bath formulation on the antibacterial properties of CAO/PVP40000/APS hybrid emulsion treated fabric. It is clear that incorporation of such hybrid emulsion gives rise to an enhancement in antibacterial bacterial properties of that hybrid emulsion treated fabric reflecting of the antibacterial properties of castor oil. Moreover, incorporation of Ag-NPs in the aforementioned finishing bath significantly improves extent of the antibacterial activities due to [32, 45, 46]:

 (i) Creation of Ag⁺, in a moisture presence, that binds to the bacterial DNA giving rise to its inactivation (Eq. 7), and/or

$$O_{2(aq)} + 4H_3O^+ + 4Ag_{(s)} \rightarrow 4Ag^+_{(aq)} + 6H_2O$$
 (7)

(ii) Genaration of oxygen radicals which subsequently oxidize the bacterial molecular structure (Eq. 8):

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

Furthermore, Table 7 depicts that such antibacterial properties of treated fabric are durable for 15 washing cycles reflecting the crosslinker role in fixation of castor oil via its hydroxyl groups onto/within the finished fabric structure.

SEM and EDX images

Figure 4 shows the SEM images of untreated fabric and CAO/PVP40000/APS hybrid emulsion treated fabric. It is obvious the treated sample shows a deposited film of CAO/PVP40000/APS hybrid ingredients on its surface compared to the untreated sample image. On the other hand, Fig. 5 loading the treated fabric with Ag-NPs of a content 0.09% (w/w).

 Table 7
 The antibacterial properties of CAO/PVP40000/APS hybrid emulsion treated fabric

PVP MW	Ag-NPs (%)	Reduction (%)		
(Dalton)		G + ve	G-ve	
Untreated	0	0	0	
Control	0	0	0	
CAO/PVP40000/APS	0	93.3 (90.2)	90.9 (86.3)	
hybrid emulsion	1	100 (96.9)	99.7 (93.1)	

Fabric finishing conditions: The fabric samples were padded in finishing bath containing 60 g/L of DMDHEU, 4 g/L of AS and a specific conc. Of the prepared emulsions to a wet pick up of 100%; drying at 100 °C/3 min; curing, 160 °C/3 min. Control sample is a sample treated with an easy care finishing bath in absence of the prepared emulsions. Values in parentheses indicate durability of treated fabric to washing after 15 washing cycles. WRA: G + ve: St. aurous; G - ve: E. coli

Fig. 4 SEM images of

sion treated fabric

A untreated fabric and **B** CAO/ PVP40000/APS hybrid emul-





Fig. 5 EDX image of cotton fabric treated with easy care finishing bath containing the CAO/PVP40000/APS hybrid emulsion and Ag-NPs

Conclusions

- The optimum reaction conditions to synthesize the CAO/PVP/APS hybrids are: PVP/CAO weight ratio, 15%; PVP concentration, 60%; APS conc., 4% (based on weight of CAO); reaction temperature, 80 °C; reaction time, 60 min.
- The FTIR analysis confirmed synthesis of the CAO/PVP/ APS hybrids.
- The TEM analysis proved that the particles of the CAO/ PVP1300000/APS hybrid emulsion are larger than that of the CAO/PVP1300000/APS hybrid emulsion.
- Incorporation of either of the aforementioned hybrids emulsions in the easy care finishing formulations of cot-

ton fabric results in promoting fabric softness, tensile strength, flexibility, and antibacterial activities of treated fabric along with reducing fabric wettabiliy and resiliency.

- Inclusion of Ag-NPs in the aforementioned finishing bath significantly improves extent of the antibacterial activities of treated fabric.
- The treated fabric was examined using SEM as well as EDX analysis.

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Declarations

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