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Optimization of Salt-Free Reactive Dyeing Process for Cotton with Coconut Oil/Water Dual-Phase Dyeing System

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In this study, the coconut oil/water dual-phase dyeing system for developing salt-free reactive dyeing process for cotton was established. The selected dyes in this work were hot-dyeing reactive dyes, including C.I. Reactive Red 243, C.I. Reactive Blue 214 and C.I. Reactive Orange 70. A dual-phase dyeing system was performed under various coconut oil to water ratios and a comparative study was conducted on the conventional aqueous-based reactive dyeing in the presence and absence of salt. The results showed that the oil to water ratio of 3:1 imparted the highest colour yields (K/S values) and achieved a superior colour yield to the conventional aqueous-based dyeing. The results also pointed out that better dye fixation could be attained by dyeing cotton with the coconut oil/water dual-phase dyeing process under the optimum conditions. A comparable degree of dye exhaustion was observed for the aqueous-based and the dual-phase dyeing processes, however, the dye fixation was higher for the dual-phase dyeing, indicating less dye hydrolysis in this dyeing system. Consequently, superior colour fastnesses to washing and rubbing were obtained, less dye staining was observed in the case of dual-phase dyeing. A study on reusability of the coconut oil recovered from the spent dyebath for another reactive dyeing cycle was also conducted. The oil from the spent dyebath was directly taken to use without any further purification as a dual-phase medium along with water and the obtained result indicated a reusability potential of coconut oil. From this research, it infers that cleaner reactive dyeing process with the coconut oil/water dual-phase system for cotton could be developed.

Keywords: Coconut oil, Reactive dye, Salt-free dyeing, Dual-phase system, Cotton.

INTRODUCTION

Textile industries especially in the textile dyeing process has been placed a great concern as a major source of environmental pollution. Reactive dye is most widely used for dyeing cellulosic fibers, especially cotton, owing to its wide variety of shade and outstanding wet fastness performance. However, a large scale of reactive dyes exploited industrially for textile dyeing brings about a serious environmental pollution issue, in particular, the effluent released containing chemicals left over after the dyeing process [1]. The conventional aqueous-based reactive dyeing generates large quantities of wastewater containing high concentrations of chemicals, *e.g.* salt, alkali, unfixed dyes and others dyeing auxiliaries, which in turn cause high COD value, leading to a serious effect to human and aqualic life.

Because of increasing pollutant emissions from reactive dyeing process, various technologies have been developed with the purpose of reducing hazardous effluent discharge and improving dyeing process. Development of reactive dyeing process focuses primarily on the increment of dye fixation and decrease of chemical auxiliaries used, including salt and alkali. A huge amount of salt approximately 200,000-250,00 tons per year from a reactive dyeing process discharged in the textile effluent led to increased salinity in the river, disturbing marine life and creating infertility of soil [2]. Therefore, the salt-free reactive dyeing has been developed toward a benign reactive dyeing process. For instance, modification of reactive dye structure and cationization of cotton with cationic agents have been developed in order to eliminate the requirement of salt [3]. A cationic reactive dye offers possible environmental benefits over a conventional reactive dye due to its inherent dyeing ability in the absence of salt [4], but still it is not commercially released [5]. Modification of cotton by a cationization process was later developed and was found as an effective method for reactive dyeing in a salt-free condition. Nevertheless, this technique has yet to be affirmed by industry due to time-consuming process [6].

Later, alternative reactive dyeing systems, such as a dual-phase process, have been developed by changing the dyeing medium from a conventional aqueous-based one to a salt-free non-aqueous/aqueous system. In a dual-phase system, the initial non-aqueous media, including hydrocarbon solvent, *e.g.* hexane, cyclohexane and iso-octyl silane, were studied. However, these solvents are environmentally unfriendly and harmful to human health [7]. Consequently, greener non-aqueous dyeing media have been considered as an option to promote more eco-friendly dual-phase reactive dyeing process. Vegetable oils such as cottonseed oil [8,9], soybean oil [10], spent coffee ground oil [11] and spent cooking oil [12,13] have been studied as a dyeing medium for the oil/water dual-phase dyeing system due to their non-toxicity, biodegradability and low volatility [14]. Moreover, a type of synthetic oils, including liquid paraffin, was also investigated and displayed a promising potential to be a benign dyeing medium for the oil/water dual-phase system [15,16]. Apart from reducing chemicals used in reactive dyeing, *i.e.* salt, the dual-phase dyeing system could also enhance a comparable dye fixation as compared with the conventional aqueous-based dyeing. In addition, spent-bath oil could potentially be recovered and used for another dyeing cycle. In this study, coconut oil was chosen as a non-aqueous medium for the oil/water dual-phase system. Owing to its light yellow in colour, coconut oil would not affect the shade of dyed fabrics obtained. High saturated fatty acid content of coconut oil results in a good thermal stability that enables coconut oil to use at elevated temperatures. In addition, its high flash point and biodegradability [17,18] could provide safety for hot-dyeing applications and environment, respectively, when it is applied as a dyeing medium for the dual-phase reactive dyeing.

In this work, coconut oil was used as a dyeing medium in the oil/water dual-phase reactive dyeing on cotton. The hot-dyeing reactive dyes, including C.I. Reactive Red 243, C.I. Reactive Blue 214 and C.I. Reactive Orange 70, were examined. Optimum ratio of coconut oil/water for a dual-phase dyeing process was investigated in terms of colour yield of the dyes obtained on cotton. The exhaustion and fixation behaviours of the selected reactive dyes in the dual-phase process were investigated on cotton. Moreover, the build-up properties of dyes were also studied as compared with the conventional aqueous-based dyeing. The colour fastnesses to washing and rubbing of the dyed cotton fabrics were assessed. Finally, reusability of coconut oil from the spent dyebath was conducted.

EXPERIMENTAL

Coconut cooking oil (Naturel brand) from Lam Soon Public Co., Ltd., Thailand was used. The azo-based, *bis*-monochlorotriazinyl reactive dyes selected for this study were Drimaren Red X-6BN, Drimaren Navy X-GN and Drimaren Yellow X-4RN from Archroma, Co., Ltd, Thailand. The details of each dye used are shown in Table-1. Plain weave cotton fabric (144 g/m^2) was scoured before use. A commercial wetting agent, Sera Wash C-NEC, non-ionic wetting agent was kindly supplied by DyStar, Thailand. All chemicals used are of reagent grade.

Optimum coconut oil to water ratio for dual-phase dyeing: Cotton fabrics (2 g) were dyed with the selected reactive dyes at 2%owf in the coconut oil/water dual-phase dyebath. The dual-phase dyeing system was established using coconut oil and water at volume ratios of 1:3, 1:2, 1:1, 2:1 and 3:1 in a Daelim Starlet infrared dyeing machine at a liquor ratio of 20:1. The dyeing process was first heated to 80 ºC at a heating rate of 2 ºC/min. Sodium carbonate, acting as an alkali for dye fixation, was added at 15 g/L after 10 min dyeing at 80 ºC and the dyeing was proceeded for another 20 min before cooling the dyebath down to 30 ºC. The dual-phase dyeing procedure is shown in Fig. 1.

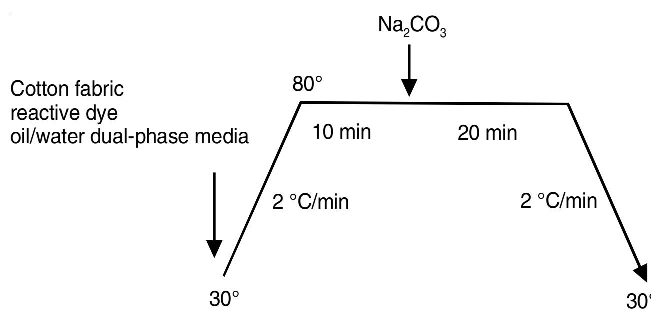
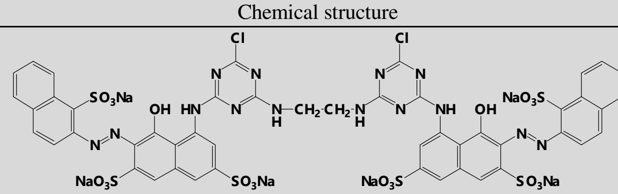
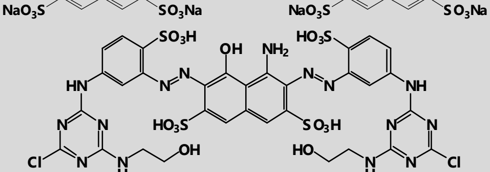


Fig. 1. Dyeing profile of salt-free, dual-phase dyeing process

TABLE-1
HOT-DYEING REACTIVE DYES USED

Reactive dye	C.I. Number	Chemical structure	m.w.
Drimaren Red X-6BN	C.I. Reactive Red 243		1449.99
Drimaren Navy X-GN	C.I. Reactive Blue 214		990.76
Drimaren Yellow X-4RN	C.I. Reactive Orange 70	N/A	N/A

After dyeing, the fabrics were rinsed with running water and a subsequent washing-off process was conducted in 5 g/L wetting agent at 70 ºC for 20 min. Then, the fabrics were rinsed and dried at room temperature. The colour yields (K/S values) of the dyed fabrics were measured with a Datacolor 550 spectrophotometer. The optimum oil to water ratio was studied.

A comparative study was conducted by dyeing cotton fabrics with a conventional aqueous-based reactive dyeing system. The input of selected reactive dyes was 2%owf at a liquor ratio of 20:1 with similar dyeing profile as described for a dual-phase dyeing in Fig. 1, but an aqueous medium was used instead and salt was also added. The influence of Na_2SO_4 concentrations was studied at 50 and 100 g/L and the alkali (Na_2CO_3) used was at 15 g/L. In addition, the dyeing with no salt addition was also compared. Table-2 shows the dyeing conditions used in this study.

TABLE-2
REACTIVE DYEING CONDITIONS STUDIED

Dyeing system	Na_2SO_4 (g/L)	Na_2CO_3 (g/L)	Oil to water ratio
Aqueous-based	0, 50 and 100	15	–
Dual-phase	0	15	1:3, 1:2, 1:1, 2:1 and 3:1

Exhaustion and fixation behaviours: The dye exhaustion in aqueous-based and dual-phase dyeing systems with 2%owf reactive dyes was determined by measuring the absorbance of the dyebath solution before and after dyeing with a Specord 250 UV/Vis spectrophotometer at the wavelengths at maximum absorption of 440, 560 and 620 nm for Reactive Orange 70, Reactive Red 243 and Reactive Blue 214, respectively. The exhaustion percentages were calculated from eqn. 1.

$$\text{Exhaustion (\%)} = \left(\frac{A_0 - A_1}{A_0}\right) \times 100 \qquad (1)$$

where A_0 and A_1 are the absorbances of the dyebath solution before and after dyeing, respectively.

The fixation percentages of the dyes applied at 2%owf in both dyeing systems were calculated from the K/S values of the dyed fabrics (eqn. 2).

$$\text{Fixation (\%)} = \frac{(K/S)_2}{(K/S)_1} \times 100 \qquad (2)$$

where $(K/S)_1$ is the colour yield of the dyed fabrics after rinsing with water and air dried and $(K/S)_2$ is the colour yield of the dyed fabrics after being exposed to rinsing and subsequent three consecutive washing-off cycles (with virtually no dye staining in washing bath) and air dried.

Build-up properties of the reactive dyes on cotton: In aqueous-based and dual-phase reactive dyeing systems, the build-up properties of selected reactive dyes were investigated on cotton fabrics. The dyes were applied onto the cotton fabrics at the concentrations of 0.5, 1, 2, 3 and 5%owf. The colour yields of dyed cotton fabrics were also measured with a Datacolor 550 spectrophotometer.

Colour fastness of dyed cotton fabrics: The samples used in this testing were cotton fabrics dyed to the same level of colour yield (K/S ≈ 13). Colour fastnesses to washing and rubbing of the dyed fabrics were performed according to the ISO/C06 standard method No. C2S and the BS 1006 X 12 105 test method, respectively.

Reusability of spent coconut oil: For reusability evaluation, coconut oil was collected from the spent dyebath and reused as a dyeing medium along with water in a dual-phase process for one additional dyeing cycle with 2%owf Reactive Blue 214 at a liquor ratio of 20:1. The degree of dye exhaustion in a reused oil/water dual-phase dyeing system and the colour yield obtained were examined in comparison with that dyed in a virgin bath of coconut oil/water media.

RESULTS AND DISCUSSION

Optimum coconut oil to water ratio for a dual-phase reactive dyeing system: The K/S values of the cotton fabrics dyed with Reactive Red 243, Reactive Blue 214 and Reactive Orange 70 in aqueous-based and coconut oil/water dual-phase reactive dyeing systems are compared in Table-3. In aqueous-based dyeing without salt, the three dyes displayed poor dye uptake as observed from the K/S values. The dye exhaustion was improved when Na_2SO_4 was added into the dyebath, as a result of dye-fiber substantivity enhancement [19]. This result also indicates salt sensitivity of the dyes as observed from significant increase of the K/S values when salt concentration was increased. In case of a salt-free, coconut oil/water dual-phase system, the dyes exhibited superior colour yields (higher K/S values) to the conventional aqueous dyeing system with no salt addition, indicating the effect of coconut oil in raising the dye uptake onto cotton. K/S values of the dyed fabrics in a dual-phase system were higher when the ratios of coconut oil in the dyebath were increased and highest colour yield was attained when 3:1 coconut oil to water ratio was employed. In a dual-phase medium, an oil/water emulsion is formed. Stability of this emulsion was controlled by changing the mixing ratio of oil to water. Moreover, the rate of dye movement from the dyebath to fabrics was affected by the emulsion stability. In highly stable emulsion, the dyes preferably stay in the emulsion rather than moving onto cotton fibers [9]. From the above results, it elucidates that as coconut oil to water ratio increases, the emulsion stability is lowered and thus accelerate the penetration of the dyes onto cotton, resulting in the increase of dye uptake. The ratio of coconut oil to water used in the dual-phase dyeing plays an important role in manipulating dyeing ability of these *bis*-monochlorotriazinyl reactive dyes on cotton. From this study, the optimum coconut oil to water ratio for the salt-free, dual-phase dyeing is 3:1. Moreover, dyeing under such conditions impart a superior colour yield on cotton to the conventional aqueous-based dyeing with 100 g/L Na_2SO_4. The increment of oil to water ratios results in the decrease of emulsion stability, leading to demulsification of dye solution in oil and rapid exhaustion of the dyes to fibers. Therefore, the optimized coconut oil/water dual-phase dyeing system could be feasible to develop a salt-free reactive dyeing process for cotton (as compared with a conventional aqueous dyeing with salt). Furthermore, it helps reduce pollutant discharge to environment.

TABLE-3
K/S VALUES OF THE DYED COTTON UNDER DIFFERENT DYEING SYSTEMS

Reactive dyeing system	Dyeing auxiliaries	K/S		
		Red 243	Blue 214	Orange 70
Aqueous-based	No salt	3.73 ± 0.12	4.13 ± 0.10	3.69 ± 0.08
	Salt (50 g/L)	9.06 ± 0.67	8.28 ± 0.37	8.38 ± 0.10
	Salt (100 g/L)	11.30 ± 0.40	10.74 ± 0.24	10.20 ± 0.27
Dual-phase	1:3 oil/water	5.07 ± 0.02	5.68 ± 0.54	5.41 ± 0.40
	1:2 oil/water	6.75 ± 0.23	6.70 ± 0.59	6.17 ± 0.14
	1:1 oil/water	7.00 ± 0.11	7.70 ± 0.39	7.55 ± 0.16
	2:1 oil/water	10.10 ± 0.83	10.60 ± 0.23	9.51 ± 0.41
	3:1 oil/water	12.29 ± 0.32	11.29 ± 0.54	11.76 ± 0.35

Dye exhaustion and fixation behaviours: In this study, the exhaustion and fixation behaviour of Reactive Red 243, Reactive Blue 214 and Reactive Orange 70 in a conventional aqueous-based and the dual-phase systems with a 3:1 coconut oil to water ratio were determined. The %exhaustion of these three dyes in an aqueous dyeing in the presence of salt increased with increasing salt concentrations (Table-4). This is explained by an ability of salt to assist dye exhaustion onto cotton by reducing electrical repulsion between anions of dyes and fibers [20]. However, by dyeing in the oil/water dual-phase bath, a comparable degree of dye exhaustion could be achieved with no need of salt addition. When the %fixation was considered, it was observed that salt could also affect the dye fixation. Higher dye exhaustion onto the fiber driven by salt could help reduce dye loss due to hydrolysis. On the contrary, a 3:1 oil/water dual-phase dyeing in the absence of salt could render even better dye fixation with less hydrolyzed dyes, especially in the case of Reactive Blue 214. From the dye fixation results, it seems that coconut oil used in a dual-phase system significantly involves in achieving better dye-fiber fixation rate, hence, reducing dye hydrolysis.

Build-up properties: The build-up curves of Reactive Red 243, Reactive Blue 214 and Reactive Orange 70 in the conventional aqueous-based and 3:1 oil/water dual-phase dyeing systems are shown in Fig. 2. In this study, the dyeing was carried out at various dye concentrations. For the aqueous-based system, dyeing was carried out with no-salt, 50 and 100 g/L salt additions. These dyes showed excellent build-up properties in the oil/water dual-phase system. A poor build-up of three dyes was found in the aqueous dyeing system without salt. By adding salt into the dyebath, the dyes exhibited better build-up properties, particularly at 100 g/L salt. However, this could not compete with the dye build-up obtained from the salt-free, dual-phase dyeing. Higher build-up profile was clearly observed for Reactive Blue 214 and Reactive Orange 70 while Reactive Red 243 exhibited a slightly better build-up curve as compared with those of aqueous-based dyeing. This is in line with the dye fixation results reported in the previous section.

The results agree with the work reported by An *et al.* [15] that the build-up properties in a liquid paraffin dyeing system were higher than in the conventional reactive dyeing both in the presence and absence of salt.

Colour fastness properties: Table-5 compares the colour fastness of the cotton fabrics dyed with three reactive dyes from the conventional aqueous dyeing at 50 and 100 g/L salt

TABLE-4
DYE EXHAUSTION AND FIXATION OF THE CONVENTIONAL AQUEOUS DYEING AND
DUAL-PHASE SYSTEM WITH 3:1 COCONUT OIL TO WATER RATIO

Reactive dyeing system	Red 243			Blue 214			Orange 70		
	Exhaustion (%)	Fixation (%)	Hydrolyzed dye (%)	Exhaustion (%)	Fixation (%)	Hydrolyzed dye (%)	Exhaustion (%)	Fixation (%)	Hydrolyzed dye (%)
Aqueous/salt (50 g/L)	92.09±0.21	65.83±1.45	26.26±1.47	90.32±0.56	58.17±2.40	32.15±1.89	95.45±0.22	64.64±3.22	30.81±3.41
Aqueous/salt (100 g/L)	97.30±0.48	71.97±2.51	25.33±2.37	94.87±0.30	66.59±1.56	28.28±1.77	98.59±0.19	69.84±0.58	28.75±0.74
Dual-phase	96.74±0.50	74.34±0.76	22.40±0.81	93.60±0.44	73.82±2.01	19.78±1.57	98.52±0.09	75.51±2.70	23.01±2.73

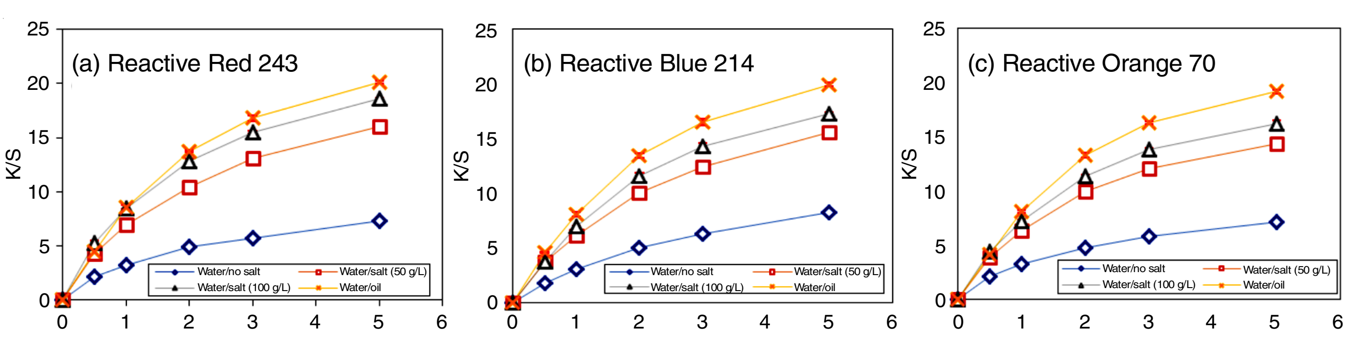


Fig. 2. Build-up curves of the reactive dyes on cotton fabrics dyed under salt-free, dual-phase process and conventional aqueous-based dyeing at various salt concentrations

TABLE-5
COLOUR FASTNESS OF THE DYED FABRICS FROM THE CONVENTIONAL AQUEOUS DYEING AND THE DUAL-PHASE PROCESSES

Reactive dyeing system	Wash fastness						Rubbing fastness					
	Colour change			Staining on cotton			Dry			Wet		
	Red 243	Blue 214	Orange 70	Red 243	Blue 214	Orange 70	Red 243	Blue 214	Orange 70	Red 243	Blue 214	Orange 70
Aqueous/salt (50 g/L)	5	5	5	3	3	3/4	4	4/5	4	3/4	4	4
Aqueous/salt (100 g/L)	5	5	5	3	3	3/4	4	4/5	4	4	4	4/5
Dual-phase	5	5	5	3/4	3/4	4	4/5	5	5	4	4	4/5

and the oil/water dual-phase system. The wash fastness results informed that no colour changes was found on the tested samples of these dyes for both conventional aqueous dyeing in the presence of salt and dual-phase dyeing systems. Lower degree of dye staining on multifiber adjacent fabrics of dyed fabrics from the dual-phase system indicates a better colour fastness to washing as compared with those from the conventional dyeing at 50 and 100 g/L salt. The results of dry rubbing fastness were also in line with washing fastness with samples from the dual-phase system being rated at higher levels on the grey scale of staining as compared with the aqueous-based dyeing. While the wet rubbing of the dyed samples from both dyeing systems was nearly similar with about 0.5 unit difference on the grey scale for the samples of 50 g/L salt as compared with the others. Inferior colour fastness level in the case of aqueous-based dyeing with salt addition implies a lower degree of dye fixation and more hydrolyzed dyes (Table-4), when compared with dual-phase system. Owing to dye hydrolysis, in addition, aqueous-based dyed fabrics would lose dye molecules easily, when compared with dual-phase dyed fabric, resulting in poor staining properties [21]. Furthermore, the wet rubbing fastness of both conventional aqueous-based and oil/water dual-phase dyeing systems displayed poorer degree of staining than the dry rubbing fastness.

Reusability of coconut oil for reactive dyeing: Reusability study of coconut oil from the spent dyebath for reactive dyeing on cotton was performed with Reactive Blue 214 at 2%owf. Coconut oil recovered from spent dyebath was relatively turbid as compared with the oil in its virgin state (Fig. 3). The dyeability of Reactive Blue 214 in the reused oil/water medium is compared with those of virgin oil in the dual-phase dyeing process in terms of %exhaustion and K/S values (Table-6). The results demonstrated that dye exhaustion and K/S values of the reused coconut oil/water dual-phase dyeing were slightly lower than the process exploiting fresh coconut oil. This slightly inferior dyeability in the reused oil/water dyebath may be explained by the influence of oil hydrolysis products, *i.e.* free fatty acids and glycerol, generated in the former dyeing cycle. Fatty acids have an adverse effect on alkalinity required for dye fixation as they consume alkali in neutralization, thereby lowering dyebath alkalinity. Besides, glycerol may compete with cotton to react with reactive dyes [8,12].

The results infer a reusability potential of coconut oil recovered from the spent dyebath of a dual-phase dyeing process. A direct reuse of the spent oil gave a slightly lower dye uptake on cotton. Therefore, purification prior to further reactive dyeing is recommended for efficient utilization of spent coconut oil in the dual-phase dyeing.

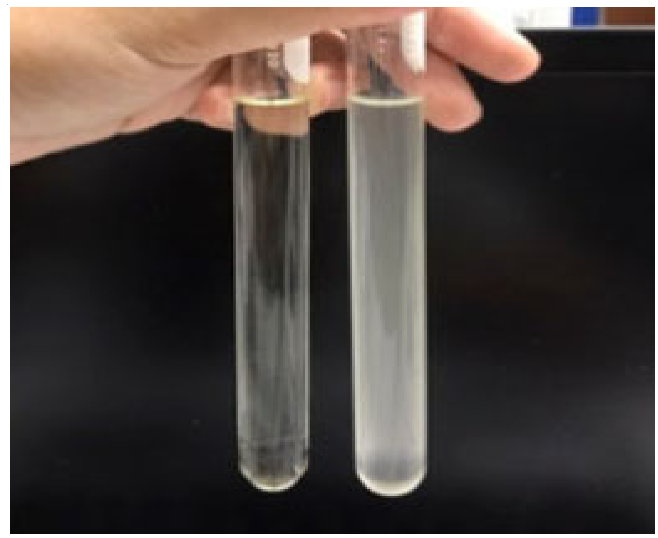


Fig. 3. Coconut oil before (left) and after (right) exposed to dyeing process

TABLE-6
DYE EXHAUSTION PERCENTAGES AND COLOUR YIELDS (K/S VALUES) OF BLUE 214 IN OIL AND REUSED OIL/WATER DUAL-PHASE DYEING SYSTEMS

Dyeing system	Exhaustion (%)	K/S
Oil/water	93.60 ± 0.44	13.44 ± 0.19
Reused oil/water	89.77 ± 0.42	12.51 ± 0.10

Conclusion

From this work, it is concluded that the coconut oil could potentially be utilized for salt-free, reactive dyeing on cotton. The optimal oil and water ratio in dual-phase system of Reactive Red 243, Reactive Blue 214 and Reactive Orange 70 was 3:1. The dye-ability of the three dyes in the dual-phase medium of coconut oil and water was superior to the conventional aqueous dyeing, being observed from degrees of dye fixation and colour yield obtained. It may be said that reactive dyeing in this dual-phase medium at an appropriate oil to water ratio could enhance dye exhaustion onto cotton fiber with lessening chance of dye hydrolysis, hence the dyes are better fixed onto the fiber than in the aqueous-based process even those with salt addition. As expected, the colour fastnesses to washing and rubbing of the dyed fabrics from the dual-phase process are also better than from the aqueous-based counterparts as a consequence of less hydrolyzed dyes. Furthermore, reusability of the coconut oil from the spent dyebath could be possible. This study elucidates that the coconut oil/water dual-phase dyeing system could be developed for cleaner reactive dyeing and reducing pollutant emission from a textile dyeing process.

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CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this article.

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