

Research Article

Development and Application of Reactive Dye Microcapsules for Cotton Fabric Dyeing

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Abstract

Reactive dyestuffs used in textile efficiency cotton dyeing are generally applied as a triple mixture. Most of these dyestuffs are supplied to HT machines in powder form without being suitable for automation. 100% of the dyestuffs used in the dyeing recipe applied in reactive dyeing cannot adhere to cotton. Maximum 70% of it adheres to cotton. If the remaining 30% is dye, the substances pass into the water phase. And the dyestuffs that pass into this water phase include substances that are insoluble in water and difficult to purify. It also increases environmental waste burdens. In project scope; By encapsulating reactive dyes with microcapsule technology, it is aimed to reduce the limits of dyes used and reduce environmental waste. And in this context; Dye saving will be achieved.

Keywords: Sustainability, Microcapsules, Eco-Dyeing, Cotton, Reactive Dyes

1. Introduction

The textile industry renews itself day by day and begins to expand its presence in the sector with different production methods and environmentally friendly processes. Although textile industries do not want to give up conventional methods, responding to user demands and increasing market share is important in today's technology. In the changing world, in order for our export customers to have an understanding not only of new products but also of clean energy, clean production, sustainable production,

minimum waste amount, minimum chemical consumption and to set an example for other sectors in the industry, the new product that will emerge from the beginning of the project is completely based on this. It is aimed to be produced according to the concepts.

The demand for textiles that encourage "well-being" will persist due to people's desire for a more fruitful and healthy way of living. With active delivery from microcapsules, textiles can "interact" with the user to promote comfort, relaxation, and a reduction in stress. The development of innovative medical textiles and performance fabrics with additional value for sports and outdoor applications has been the focus of the textile industry in the US, Japan, and Western Europe for the past ten years. Microencapsulation has the potential to contribute to this ongoing progress. For instance, it can enable the attachment of sensing chemicals to medical devices and sports apparel, which can alert users to potential risks or damage [1].

For over a century, urea-formaldehyde (UF) resin has been widely recognized as the most widely used bonding glue in the wood-based materials industry. The constant release of toxic formaldehyde, which could contaminate environments, especially indoor ones, and perhaps cause cancer and deformities, is the most significant downside of UF adhesive-based products. Thus, a considerable deal of research and development has been conducted with the goal of lowering the formaldehyde emission caused by UF adhesives [2].

Reactive colorants were developed by the industry as a substitute for traditional cellulosic fiber dyeing, and they are now the most popular for cotton fibers. Their basic idea is based on addition and substitution processes involving the fiber's hydroxyl groups. Although the system is homogeneous, alkaline, and catalyzed, the mechanisms are altered by the fibers' heterogeneity. As a result, secondary reactions take place that are roughly equal in size to the initial reactions. The alkaline environment in the solution interacts with the fiber and the water molecules. Lower wash fastness and a significant decrease in dyeing output are caused by the hydrolysis reaction with water. Applying a thin layer of polymer to a material is known as microencapsulation, and it is one way to improve the colorant's yield and protection. Microcapsules are being used more frequently and with considerable attention in the textile industry. The physical and chemical properties of the core, the interaction between polymers, and the solubility of the core determine the best encapsulation technique. The three primary categories among the numerous procedures currently in use are physical, chemical, and physical-chemical [3].

When we tried the reactive dyes on the %100 cotton towels and other household items, we have come across that during application the dyes especially red, yellow and blue have different exhaustion profiles. From operational side this caused more energy to the machines more use of water and more time spent during production. From the application side the different diffusion of dyes caused faulty dyeing. This led us to research on how to dye more accurately without compromising the quality.

Wool fabrics were dyed by LC-Ms and conventional dyeing process, respectively, using normal temperature swing dyeing testing machine (PCL-1000A, Dalin Starr Co., Ltd, Korea). The microcapsule dye bath consisted of LC-Ms, acetic acid, and sodium sulfate. Additionally, surfactant, sodium carbonate, and leveling agent were added during the traditional dyeing process. Specifically, wool fabrics were dyed at a liquor ratio of 30:1. The fabrics were placed in the dye bath at 30 °C, the pH of which was easily adjusted to four with acetic acid, and anhydrous sodium sulfate powders were added to increase the dyeing efficiency. To increase the dyeing effect of wool fabrics, surfactant, sodium carbonate and leveling agent were added to the dyeing solution using the traditional dyeing process. Then, the temperature was increased at a rate of 1 °C/min to 90 °C for microcapsule staining and 95 °C for conventional staining and remained for 50 min. After the LC-Ms dyeing process, the floating color was removed by a method called “starch dyeing”. Specifically, the dyeing residue was filtered and removed with a 220 nm filter, and the wool fabrics were continuously dyed in the remaining dye bath for 10 min under 80 °C. Since the supplied LC-Ms were significantly reduced, the dyestuffs on the surfaces of the fibers and the dyestuffs remaining in the dye bath tended to dye the inside of the fiber structure, and accordingly, the dyeing fastness was increased. In the conventional dyeing process, sodium carbonate was added to the dye bath and the temperature was reduced to 80 °C and kept for 10 minutes. Finally, the fabrics in two groups were rinsed and washed with tap water and dried overnight [4].

The solvent evaporation method was used to fabricate visual microcapsules. For this method, shell formation is the result of rapid polymerization of hydrophilic and lipophilic monomers at the interface of an oil-in-water emulsion. Figure 1a shows the fabrication process of SiO₂/PMMA/CVL visual microcapsules. 2 g of PMMA was dissolved in 60 g of dichloromethane. 0.5 g of CVL was dissolved in 4 g of phenyl acetate. CVL and PMMA solutions were ultrasonically dispersed as the oil phase by an ultrasonic disperser for two minutes. 3.2 g of PVA emulsion stabilizer was slowly dissolved in it [5].

We have compared conventional dyeing and microcapsule dyeing to see if there is a more sustainable and more compatible way to dye the fabrics. We have used the same trichromatic recipe for both conventional and microcapsule dyeing.

2. Materials and Methods

%100 cotton used for the application supplied by BURSALI TEXTILE company. Ethyl Cellulose & (Melamine-Urea-Formaldehyde) used as the outer wall material of the microcapsule. Na_2SO_4 (Salt) solution, it is used as an intermediate material for the formation of the microcapsule. Reactive Dye as used to form the color as a trichromatic recipe. Citric Acid and Acetic Acid used as binding material to form the microcapsule. Soda Solution (NaOH) or Tween 80 it is used to adjust the basic environment at the pH required for the formation of the microcapsule. Ethyl Acetate & Tween 20 & Chloroform for dissolving the dyestuff.

2.1. Recipe for the dyes

We have made 6 colors with both conventional dyeing and with microcapsules to compare the results. Microcapsules have been made with Panther 3006L machine. Colors, recipe and the production process has been shown in Table 1.

Table 1: Dyes and Recipes

Color	Trichromatic Recipe	Production Process
Light Grey	Everzol Yellow LX Everzol Red LX Everzol Blue LX	Conventional Dyeing
Light Grey	Everzol Yellow LX Everzol Red LX Everzol Blue LX	Microcapsule
Koyu Kahve	Everzol Yellow LX Everzol Red LX Everzol Blue LX	Conventional Dyeing
Koyu Kahve	Everzol Yellow LX Everzol Red LX Everzol Blue LX	Microcapsule
Mouve	Everzol Yellow LX Everzol Red LX Everzol Blue LX	Conventional Dyeing
Mouve	Everzol Yellow LX Everzol Red LX Everzol Blue LX	Microcapsule

Mor	Everzol Yellow LX Everzol Red LX Everzol Blue LX	Conventional Dyeing
Mor	Everzol Yellow LX Everzol Red LX Everzol Blue LX	Microcapsule
Power Blue	Everzol Yellow LX Everzol Red LX Everzol Blue LX	Conventional Dyeing
Power Blue	Everzol Yellow LX Everzol Red LX Everzol Blue LX	Microcapsule
Hardal	Everzol Yellow LX Everzol Red LX Everzol Blue LX	Conventional Dyeing
Hardal	Everzol Yellow LX Everzol Red LX Everzol Blue LX	Microcapsule

2.1.1. Application of the Microcapsules to the Fabric

First the water, ethyl cellulose and formaldehyde mixed to make the outer shell of the microcapsule. After that Na_2SO_4 added to make the intermediate material. Reactive trichromatic recipe added as a dye mixture. To adjust the alkalinity and pH we added soda and Tween 80 mixture. All of the application has been made at 45 degrees while mixing for 10 min after each ingredient have been added.

2.1.2. Tests of the Fabric

Conventional dyed and microcapsule dyed samples were tested for crocking fastness according to ISO-105X12, washing fastness according to ISO-105C06 option A1S. Also the spectrophotometer DE tests are made according to ISO 105 - J01.

3. Results

The spectrophotometer DE results are shown in Table 2.

Table 2: Spectrophotometer DE Results

Color	Production Process	Explanation
Light Grey	Conventional Dyeing	0.87
Light Grey	Microcapsule	
Dark Brown	Conventional Dyeing	0.71
Dark Brown	Microcapsule	
Mauve	Conventional Dyeing	0.49
Mauve	Microcapsule	
Purple	Conventional Dyeing	0.53
Purple	Microcapsule	
Power Blue	Conventional Dyeing	0.49
Power Blue	Microcapsule	
Mustard	Conventional Dyeing	0.72
Mustard	Microcapsule	

Most of results are in the reasonable area while being under 1. Mauve, purple and power blue results show us that they are well below 1 and nearly identical. All of the colors are acceptable as a microcapsule.

Washing fastness results are shown in Table 3.

Table 3: Washing Fastness Results

Color	Production Process	Washing Fastness					
		Wool	Acrylic	Polyester	Polyamide	Cotton	Acetate
Light Grey	Conventional Dyeing	4/5	4/5	4/5	4/5	4	4/5
Light Grey	Microcapsule	4/5	4/5	4/5	4/5	3/4	4/5
Dark Brown	Conventional Dyeing	4/5	4/5	4/5	4/5	4	4/5
Dark Brown	Microcapsule	4/5	4/5	4/5	4/5	3/4	4/5
Mauve	Conventional Dyeing	4/5	4/5	4/5	4/5	4	4/5
Mauve	Microcapsule	4/5	4/5	4/5	4/5	3/4	4/5
Purple	Conventional Dyeing	4/5	4/5	4/5	4/5	3/4	4/5
Purple	Microcapsule	4/5	4/5	4/5	4/5	3/4	4/5
Power Blue	Conventional Dyeing	4/5	4/5	4/5	4/5	3/4	4/5
Power Blue	Microcapsule	4/5	4/5	4/5	4/5	3/4	4/5
Mustard	Conventional Dyeing	4/5	4/5	4/5	4/5	4	4/5
Mustard	Microcapsule	4/5	4/5	4/5	4/5	3/4	4/5

Washing fastness results shows us that there is a slight decrease in staining performance with cotton, other than that the results are identical and well in the acceptable area. Color change for all dyeings resulted as 4/5

Color fastness to rubbing results are shown in Table 4.

Table 4: Color Fastness to Rubbing Results

Color	Production Process	Rubbing Fastness	
		Dry	Wet
Light Grey	Conventional Dyeing	4/5	4/5
Light Grey	Microcapsule	4/5	4/5
Dark Brown	Conventional Dyeing	4/5	4/5
Dark Brown	Microcapsule	4/5	4/5
Mauve	Conventional Dyeing	4/5	4/5
Mauve	Microcapsule	4/5	4/5
Purple	Conventional Dyeing	4/5	4/5
Purple	Microcapsule	4/5	4/5
Power Blue	Conventional Dyeing	4/5	4/5
Power Blue	Microcapsule	4/5	4/5

Mustard	Conventional Dyeing	4/5	4/5
Mustard	Microcapsule	4/5	4/5

Results show us nearly perfect scores. All of the colors that undergo the test for both conventional and microcapsule is identical. This shows us that the microcapsules are very well exhausted into the fiber

4. Discussion and Conclusion

In this novel study the complicated conventional dyeing for household items have been tried to change with microcapsule method. The microcapsules have been made with the Panther3006L machine and been compared with the conventional dyed samples. Both the conventional dye recipe and the microcapsules recipe has the same reactive trichromatic recipe.

The samples have been tested according to washing fastness, color fastness to rubbing and their color change (DE) by a spectrophotometer. According to the results for color change with micro capsules we have all the results under 1, which is acceptable and for mauve, purple and power blue below 0.5 value which is nearly identical with the original sample. For washing fastness, the results are identical only showing a slight reduction in cotton for microcapsules which stays in a reasonable area for washing fastness. Lastly for color fastness to rubbing it shows us identical and nearly perfect results.

The results show us that there is a potential for work and more colors and recipes could be tried. But the potential shows us that the microcapsules could give us a more sustainable method for dyeing and there could be more growing potential.

5. Acknowledge

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References

- [1] Gordon Nelson, Application of microencapsulation in textiles, *International Journal of Pharmaceutics*, Volume 242, Issues 1–2, 2002, Pages 55-62, ISSN 0378-5173, [https://doi.org/10.1016/S0378-5173\(02\)00141-2](https://doi.org/10.1016/S0378-5173(02)00141-2).
- [2] Hongyun Duan, Teng Qiu, Longhai Guo, Jun Ye, Xiaoyu Li, The microcapsule-type formaldehyde scavenger: The preparation and the application in urea-formaldehyde adhesives, *Journal of Hazardous Materials*, Volume 293, 2015, Pages 46-53, ISSN 0304-3894, <https://doi.org/10.1016/j.jhazmat.2015.03.037>.
- [3] Dutra, F. [et al.]. Microencapsulation of C.I. reactive orange 122 via solvent evaporation. A: World Textile Conference. "AUTEX2019: 19th World Textile Conference on Textiles at the Crossroads: Ghent, Belgium: 11-15 June, 2019: proceedings". 2019, p. 1-6.
- [4] Zhao, F., Rao, B., Xue, W. *et al.* The Development of Eco-Friendly Dye Microcapsules for Wool Fabric Dyeing Application. *J Polym Environ* **27**, 1202–1211 (2019). <https://doi.org/10.1007/s10924-019-01423-0>
- [5] Zheng, Xu & Wang, Qing & Li, Yao & Xu, Shuangshuang & Li, Yunfeng. (2020). Fabrication of self-reactive microcapsules as color visual sensing for damage reporting. *Journal of Materials Science*. 55. 10.1007/s10853-020-04668-6.