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Printing of Cotton Fabric with Reactive Dye using Eco-Friendly Natural Thickener

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ABSTRACT

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*Corresponding Author: ahme2005a@gmail.com The primary objective of this research was to investigate the use of a thickening agent derived from a protein-based product and mixed with sodium alginate. Additionally, the study aimed to compare this combination with fabric printed using sodium alginate alone. Using native starch-based thickeners in various industrial applications presents significant challenges, such as low water solubility and difficulties in crosslinking with reactive colors due to the high concentration of hydroxyl groups on the molecules. The print paste was prepared with SA/GT at five different mixing ratios to evaluate the impact of these ratios on the viscosity of the printing paste and the physical qualities, color strength, and fastness properties of the printed cotton fabric. The research findings indicated an enhancement in the color strength and color fastness properties of the printed cotton fabric. However, some physical properties exhibited a slight deterioration as the gelatin content in the thickener mixture increased. Encouraging outcomes were observed when sodium alginate and a gelatin-based thickener were combined in a 50:50 ratio.

Keywords: Gelatin, Sodium alginate, Thickener, Viscosity and Reactive Dyes

1. INTRODUCTION

Reactive dye printing is the most common and traditional method of printing on cotton fabric because of its reactivity towards the cotton substrate, excellent washing fastness, vibrant colour, and range of hues. In cotton fabric printing, 75% of print items are produced using reactive dye printing techniques due to their obvious advantages, which include better colour fastness and print durability [1]. The main objective of textile printing is to create visually appealing and unique designs on fabric by strategically placing one or more colour patterns [2]. In order to control the printing paste to the appropriate area of the fabric, thickener is a crucial component. Thickener serves two main functions in the printing process. These are used to alter the viscosity or flow properties of the print paste so that colour may be applied uniformly to the appropriate region of the fabric, as well as to keep the colour in place so that colour paste can be put next to colour paste without spilling onto other places [3, 4]. When printing a fabric with many colours, each colour must be applied independently to a designated area of the fabric and in the correct alignment with respect to one another; all while being kept apart by thickener [5]. Due to the fact that they are readily available and reasonably priced, starch-based thickeners are often the most widely used and well-liked natural thickeners for reactive dye printing on cotton fabric. However, there are a number of drawbacks to using native starch-based thickeners at the industrial level, including low shear stress resistance, fast retrogradation, easy thermal breakdown, and low water solubility. Furthermore, because the starch molecule has a large number of hydroxyl groups, it reacts chemically with reactive dyes, forming complex cross-links that are difficult to remove from the fabric's surface [6]. Conversely, polysaccharide thickeners should not be used with reactive dye printing since their proper functioning requires a higher alkaline pH, which may obstruct the dye's ability to react with the fabric. Reactive dyes, on the other hand, require a slightly acidic pH for optimal reactivity. The high PH of polysaccharide thickeners may neutralize the acidity required for the dye-fibre reaction, which would lead to insufficient colour development and dye fixing. When the reactive group of the dye reacts with the right alkaline conditions in this scenario, the dye cannot move from the printing paste to the fabric during the printing process. As a result, the hydrolysed dye becomes more concentrated and is unable to react with the fibre. When the hydrolysed dye is removed through washing or other post-treatment procedures, the fabric becomes stiff and has a poor colour yield [7, 8]. In light of these considerations, numerous researchers have looked at a number of discoveries and alternative technological developments for reactive dye printing cotton fabric using natural thickeners that are environmentally acceptable. Because of this, the majority of research concentrated on altering natural starch to reduce the number of hydroxy groups. Even while modification procedures can significantly improve native starch's physicochemical and structural qualities, as well as increase its water solubility and reduce starch granule size, all of these improvements still come with a cost and lose their ability to thickener and become less effi-

cient when exposed to high temperatures or acidic environments [7]. This constraint limits its applicability in applications involving acidic substances or high-temperature processing.

Consequently, sodium alginate is now used in the majority of textile industries instead of starch because it prints more effectively and reacts with dyes less due to mutual anion repulsion between the carboxyl groups in the alginate and the dye, despite the fact that sodium alginate molecules contain hydroxyl groups [9]. However, sodium alginate has become scarce and expensive as a result of the rising use of reactive dyes in textile printing, limiting its application. The disadvantages of starch-based thickeners, the high price of sodium alginate, the scarcity of natural thickeners, and the increasing need for thickeners drive the search for widely available materials that may be found locally and can replace traditional thickeners [10]. In this investigation, gelatine material was used as a thickening agent because of its bio adhesive, tasteless, and odourless properties [14], [15]. Besides, it had consistency properties; even when stored at room temperature for long periods; it easily dissolved and made gels at lower temperatures [16]. Moreover, it has the capacity to act as an acid, base, or amphoteric property in a solution because it consists of carboxylic and amide groups [17], [18]. Furthermore, research conducted by Abdou et.al. [19] Investigated novel chitosan-starch blends as thickening agents and their application in the textile printing treatment of textiles with chitosan. The results indicated that the developed chitosan starch-based gelatine thickening agent was considered a multifunctional finish that not only contributes to its antimicrobial properties but also results in an enhancement of colour strength in printing. Overall, the research done so far has been limited, and therefore, there is inadequate data on the utilization of eco-friendly biodegradable thickening agents in printing practices with reduced pollution in effluent. Moreover, to the best of the authors' knowledge, there are no

studies that examine protein-based natural thickeners for the application of reactive dye textile printing. The main objective of this study was to use water-soluble gelatine and sodium alginate thickeners in the preparation of printing paste for the printing of cotton fabric with reactive dyes to improve the colour fastness and strength of the printed fabric. Then the physical and mechanical properties of the printed fabric were investigated. The structure of the gelatine is shown in Figure 1.



Fig 1. Gelatin structure [16]

The goal of the current study is to increase the color fastness and color strength of printed cotton fabric by preparing printing paste with water-soluble gelatine and sodium alginate thickeners for use with reactive dye printing. This study's primary goal was to explore and stimulate the potential use of gelatine as an organic, environmentally acceptable, and native thickening. Subsequently, the produced gelatine thickener contributes as a thickener that enhances the printed colour strength, the washability of the printed textile, and the viscosity of the printing paste, among other multifunctional textile printing applications [19]. Using biodegradable thickening agents offers a printing method that is less harmful to the environment and results in less pollution in the wastewater. Here, we describe the first eco-friendly protein-based natural thickener for use in textile printing to limit water pollution while keeping good quality prints.

2. MATERIAL AND METHODS

2.1 Overview of the study area

The methodological approach for this research contains two phases. Firstly, the protein-based gelatin printing thickener was extracted from the trimming edge of the lime hide. Secondary optimization of the extracted gelatin with sodium alginate, then, the developed thickener ratio which was applied on the printing of cotton fabric, and the physical and mechanical characteristics were investigated.

2.1 Materials utilized in the study

Cotton fabric: 100% cotton plain, scoured, and bleached fabric was purchased from a local supplier called Kombolcha Textile Share Company.

Chemical: A high-demand brand (Reactive Red 5 dye, mono-chlorotriazine C.I.) was used to prepare the printing paste. In addition, urea, sodium carbonate, sodium hydroxide, a wetting agent, sodium chloride, and acetic acid were used. Thickening agent: protein-based gelatine was synthesized and used in printing with reactive dyes on the cotton substrate.

2.2 Method applied in the study

2.2.1 Gelatin extraction procedure



Fig 2. Gelatin extraction process

The study employed thermal hydrolysis procedures in conjunction with alkaline treatment as a way of extracting gelatine. Figure 2 shows the order in which the steps are necessary to separate gelatine from collagen.

The animal hide trimming and fleshing waste was gathered from a nearby tannery in Addis Ababa, Ethiopia, and extensively cleaned with distilled water to get rid of any extraneous material and contaminants that had stuck to the hide's surface [16]. Subsequently, the hides were immersed in 1000 milliliters of water containing 0.3% (w/v) degreasing agent and 1% (w/v) sodium chloride for two hours at 30 degrees Celsius to eliminate the fat components. The skins were once more cleaned with water for 30 minutes at 40 degrees Celsius. After being sliced into tiny pieces using scissors, the hides were once more cleaned with water under high pressure. After that, the hides were pre-treated for 24 hours in 100 cc of 0.4% (w/v) sodium hydroxide. Subsequently, the gelatin was extracted from cow skins that had been pretreated using a thermal hydrolysis process using distilled water at a material liquor ratio of 1:5 (g: ml) at optimal temperatures (90°C) for optimal durations (6 hours), resulting in an optimal yield efficiency of 92.4%. To create ultra-pure gelatin, the extracted gelatin was first filtered through plain cotton fabric to remove bigger undesirable particles that were naturally present in the hides. Next, the gelatine was centrifuged using a 5810R Ultrafiltration to remove any remaining colloids and turbidity. Next, for three days at room temperature, the pure solution was dried and the water was evaporated using a blower in a conditioned environment.

2.2.2 Yield percentage of extracted Gelatin

The extracted gelatin yield was calculated as percentage by using the following equation.

Yield (%)=
$$\frac{\text{weight of extracted gelatin}}{\text{initial weight of hide}} * 100\%$$
 (1)

2.2.3 Preparation of printing pastes

The thickener was immersed in water at 45 degrees Celsius before the printing paste preparation. Subsequently, the dye was combined with urea and a small quantity of water, and the combination was stirred to guarantee the dyes were thoroughly dissolved. Then, to create consistent printing pastes, add all the materials to a beaker that contains a mixture of reactive dye with urea, sodium carbonate, wetting agent, and sequestering agent in the amounts indicated in Table 1. Stir continuously for 10 minutes. Following the preparation of uniform printing pastes, the entire bleached cotton fabric was printed on, dried for three minutes at 100 degrees Celsius, and the printing paste was fixed using the thermosfixation method for three minutes in a mini dryer at different temperatures (130, 140, 150, and 160 degrees Celsius). The printed sample was then taken for one cold wash, followed by two hot washes and one cold wash, and dried at 100 degrees Celsius in the mini dryer.

1	[a]	bl	e 1.	F	Formu	lati	ons	of	printing	paste	com	binat	ions
1		~ -		-	01110		0	· ·	Printing	pase	• • • • • •		

Component for MCT printing	Weight (gm)			
SA:GT	100:0,75:25,50:50 &25:75			
Urea	2g			
Sodium bicarbonate	2g			
MCT	1%			
Sequestering agent	0.2g			
Total Solution	100g			

2.2.4 Printing procedures

The printing pastes were applied to cotton cloth using the conventional manual screen-printing technique. Table 1 details the printing paste recipe that was used on cotton fabric samples. Using a tiny drier, the printed cotton fabric samples were dried at 100°C for three minutes. The optimal fixation temperature for three minutes was then determined by fixing the printing paste using the thermos-fixation method at various curing temperatures (130, 140, 150, and 160 degrees Celsius) for each sample. After the thermos fixing procedure, the printed samples were thoroughly cleaned in warm and cold water, soap-soaped in 2 gm/lit non-ionic soap for 10 minutes at 60°C, and allowed to air dry at room temperature.

2.3 Assessment criteria

After the printing process, samples were subjected to physical testing and colour fastness **testing** by using instrumental methods.

2.3.1 Determination of colour strength

Using an illumined D65 and a 10° observer, a spectrophotometer (Hunter Lab Colour) was used to measure the printed samples' colour strength (K/S value). The Kubelka-Munk equation, K/S = (1–R) 2 /2R, was used to determine the K/S value. R stands for the printed sample's reflectance at the wavelength of maximal absorbance, K for absorbance, and S for scattering [20].

2.4 Fastness Properties of the printed fabrics

The colour fastness properties to washing, rubbing, Light and perspiration of the cotton printed samples were evaluated.

2.4.1 Rubbing fastness testing

In accordance with the ES ISO 105-X12: 2018 standard, rubbing fastness was evaluated [3, 16]. A specimen of 22 cm by 8 cm, or the size of the crock meter's plate, was created. One 5 cm by 5 cm rubbing cloth was made, and it was moistened with water for wet rubbing. The specimen was positioned with its long direction parallel to the rubbing truck when it was mounted on the crock meter's base board. Using the included spring clip, the white rubbing cloth was flattened and placed over the crock-meter's peg end. To make sure the spring clip is not in contact with the test specimen, the finger was resting on it. The specimen was then rubbed back and forth over a straight truck for 10 complete cycles at a rate of 1 second for each cycle. Finally, the white rubbing test cloth was removed and the degree of staining on the undyed fabric was evaluated with grey scales.

2.4.2 Washing fastness testing

For wash fastness, the ES ISO 105 C06: 2018 [3] technique was used. To create a composite specimen, a 10 X 4 cm specimen was layered between cotton and polyester fabrics and stitched on all four sides. A 5g/l soap and 2gpl sodium carbonate washing solution was added to the Launder-O-meter along with a 1:50 liquor ratio. The sample was heated to 60 degrees Celsius and rotated at a rate of 22 rotations per minute for 45 minutes. After being taken out, the specimen was washed in cold water under running water without any friction. Three sides of the stitch were opened, and room temperature drying was done. Grey scales with geometric shapes were used to assess the staining intensity and colour shift.

2.4.3 Light fastness testing

In accordance with the ES ISO 105 B02:2015 [2] standard, light fastness was evaluated. A cloth sample that has been sliced so that half of it is covered and the other half is exposed was framed and put in a testing chamber with a standard blue scale reference cloth. This blue scale wool fabric, which comes in eight hues ranging from dark blue to light blue, is used to document how a fabric's colour changes. For approximately 72 hours, the test sample is continuously subjected to xenon-arc lamp light with a radiant intensity of 5. The original unexposed sample, which is rated on blue scales from 1 to 8, was compared to the altered sample.

2.4.4 Perspiration fastness testing

Perspiration fastness was tested according to ISO 105F10 [16] standard. Specimens of 40×100 mm were attached to the same size of multifibre to form a composite fabric. The composite fabric was wetted in acidic and basic solutions and placed between acrylic-resin plates under a pressure of 12.5 kPa separately. The test

devices containing the composite specimens were placed in the oven for 4 h at $37^{\circ}C \pm 2 {}^{\circ}C$. The change in colour of each specimen and the staining of the adjacent fabrics were assessed and compared with on the grey scales.

2.5 Physical properties of the printed fabrics

2.5.1 Determination of flexural rigidity

Stiffness of fabric was tested according to ASTM D1388-2007 [21] standard method. Rectangular sample of 2.5 cm x 20 cm (template size) in warp and weft direction, respectively, was prepared. The sample was placed on the stiffness tester and moved gently until the sample coincided with an inclined indicator. The length to which the specimen moved was measured. The flex-ural rigidity was calculated using equation [21].

 $G=1/8 \times W \times L^3$ --- (2)

Where, G is flexural rigidity of fabric, W is the GSM of the fabric and L is the bending length of the fabric.

2.5.2 Determination of tear strength

The tear strength was tested according to ASTM D1423-83 standard method [22]. Tear strength is the capacity of a material to withstand the tearing force required to propagate a tear after its initiation. The test specimen was cut according to the standard template size and the required dimensions are specified in relevant test standards. Tearing strength was tested in the warp and weft direction using Digital Tear Tester with C-Type load. An initial cut about 1cm length was made part way down the centre of a strip and then the two tails thus formed are pulled apart, so that a tear proceeds through the uncut portion of the fabric. The force used to tear the fabric was measured and recorded.

2.6 Measurement of tensile strength

The fabric sample's tensile strength was ascertained through the application of the standard test procedure ASTM D5035-95 [23]. The tensile strength of the cloth was measured using a Universal Strength Tester in both

the warp and weft directions. A 100 mm by 150 mm fabric sample was made individually for the weft and warp direction tests. A 75 mm gauge length was set between the two jaws. The sample was then secured in place between the jaws. The specimen was positioned in the centre, and its length was almost exactly parallel to the direction of force exerted. Over the clamped breadth, the specimen's tension was constant. After adjusting the speed, the sample broke in 20 ± 3 seconds.

3. RESULTS AND DISCUSSION

The colour values, physical characteristics (stiffness, tear strength, and tensile strength), and fastness characteristics (sweating, washing, rubbing, and light fastness) of printed fabric samples were evaluated. The effects of duration and temperature on the collagen gelatine extraction process were investigated. Furthermore, the effects of time storage on the printing pastes' initial viscosities were analysed and compared to sodium alginate, a conventional natural textile thickening. Processed, the extracted gelatine was added to the printing pastes and used as a thickening agent.

3.1 Optimization of extraction parameters

Table 2. Gelatin extraction conditions

S No	Temp.	Time	Weight	Yield
5.INO	(°C)	(Hr.)	(kg)	%
1	70	2	5	58
2	70	4	5	79
3	70	6	5	89
4	70	8	5	87.3
5	80	2	5	69
6	80	4	5	85
7	80	6	5	92
8	80	8	5	89.6
9	90	2	5	73.6
10	90	4	5	88
11	90	6	5	92.4
12	90	8	5	91.6
13	100	2	5	78
14	100	4	5	89.8
15	100	6	5	91
16	100	8	5	91.2

Taguchi Design version 19, a two-factor, four-level Minitab program, was used to determine the optimal conditions for gelatine extraction from collagen. The outcomes are shown below. Using methods for thermal hydrolysis, gelatine was isolated. One component was changed while keeping the other variables unchanged in order to optimize the extraction of gelatine. Five kilos of hide were extracted at a pH of five.

3.2 Effect of extraction temperature on Gelatin yield

Temperatures of 70°C, 80°C, 90°C, and 100°C were used to extract gelatine from collagen while keeping other variables constant. As it has been demonstrated on Figure 3 the yield of gelatine rose as the extraction temperature was raised, peaking at 90 °C. According to this investigation, the best percentage yield of gelatine was obtained at the ideal extraction temperature of 90°C. The percentage yield was shown to decrease at temperatures higher than 90 °C. This could be due to the consequence of the extracted gelatine being lost due to the excessive hydrolysis of the collagen.



Fig 3. Impact of temperature on Gelatin yield (%)

3.3 Impact of extraction duration on Gelatin yield

Under the same other conditions, collagen was subjected to two, four, six, and eight hours of gelatine extraction. The proportion of gelatine produced at different durations is shown in Figure 4. The yield of the gelatine extraction increased as the extraction time increased, reaching up to six hours. The highest percen age yield of 92.4% was attained after six hours. However, when the period was increased beyond six hours, the yield percentage decreased due to the intense hydrolysis of the collagen.





3.4 Measurement of printing paste viscosity

The viscosity of the printing paste was ascertained by monitoring its flow rate. Longer storage periods cause the water in the extracted paste to evaporate, which lowers the printing paste's flow rate. This is particularly true when there are larger amounts of gelatine in the paste than sodium alginate, the viscosity of the paste significantly increases as shown in Figure 5. However, if it is stored for more than eight hours, it becomes viscous and unsuitable for a printing process.



Fig 5. Impact of storage time on the viscosity of printing paste

3.5 Impact of thickener ratio on paste viscosity

he viscosity of the printing pastes increased as the gelatine content increased, as seen in Figure 6. This demonstrates that sodium alginate is not as viscous as gelatine. However, gelatine paste did not produce a consistent viscosity when it was stored alone for more than eight hours. Sodium alginate's excellent viscosity stability makes it the perfect ingredient to combine with gelatine to form a paste.

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3.6 Effect of thicker concentration on color strength

The cotton fabric sample printed using a thickening consisting of 50% sodium alginate and 50% gelatine exhibited the strongest colour, as seen in Figure 7.



Fig 7. Impact of SA:GT thickener on colour strength of printed fabric

Apart from being used as a thickening ingredient, gelatine also serves as a cationic agent, giving the fabric a positive site and intensifying the colour when printed. It is especially important for preparing print pastes for reactive dyes since it interacts with cloth rather than reactive dyes.

3.7 Impact of curing temperature on colour strength

The curing temperature was adjusted between 130 ^oC and 160 ^oC in order to examine the impact of temperature on the colour yield of monochlortriziane reactive dye printing on cotton fabric. Figure 8 demonstrated that when the curing temperature was raised, the colour strength also raised and reached its peak value at 150 ^oC. Once more, at higher curing temperatures of roughly 160^oC., a poorer colour value was observed. This could be due to reactive dye's susceptibility to higher temperatures.



Fig8. Impact of curing temperature on the colour strength of printed fabric

3.8 Impact of thickener on dry crease recovery angle



Fig 9. Impact of thickener on dry crease recovery angle

Figure 9 illustrates how increasing the amount of gelatine in the printing paste led to a slight increase increase recovery angle in both the weft and warp directions, improving the crosslinking property of the printed cotton fabric.

3.9 Tensile strength of printed fabric

As the amount of gelatine in the printing paste was increased, the tensile strength of the printed fabric decreased both warp-wise and weft-wise, as shown in Figure 10. The tensile strength may have declined because of the formation of cross-links by the hydroxyl groups in the cellulose, which stop the cellulose chains from sliding. Compared to the printed sample with unprinted, the unprinted samples had a higher tensile strength.



Fig 10. Impact of thickening agent concentration on tensile strength

3.10 Tear strength of printed fabric



Fig 11. Impact of thickening agent concentration on tear strength

The warp and weft tear strengths of the printed cotton samples declined as the amount of gelatine in the printing paste increased, as Figure 11 illustrates. This could be the outcome of increased gelatine cross-linking with the substrate, which increases fabric flexural rigidity, reduces tear strength, and lowers yarn slippage.

3.11 Estimating flexural rigidity

The result of a study implies how two thickeners at various mixing ratios affected the flexural rigidity (bending stiffness) of printed cotton fabrics are displayed in Table 3. The stiffness of the printed samples was found to increase with an increase in the proportion of gelatine in the printing paste, possibly as a result of the increased crosslinking of the fabric

Table 3. Flexural rigidity of printed	fabric
before washing	

Thick ener	Ber lengt	nding h (cm)	Flexural rigidity (mg-cm)			
ratio					(
	Warp Direction	Weft Direction	Warp Direction	Weft Direction	Overall (Warp+weft	
100:0	4.01	3.97	121.00	117.32	119.16	
75:25	4.09	4.05	128.28	124.56	126.42	
50:50	4.18	4.15	136.94	134.00	135.47	
25:75	4.27	4.22	145.97	14092	143.45	

Table 4. Flexural rigidity of printed fabric after washing

	Ben Lengt	ding h (cm)	Flexural Rigidity (mg- cm)					
Thick- ener ratio	Warp Direction	Weft Direction	Warp Direction	Weft Direction	Overall (Warp+weft)			
100:0	3.01	3.07	51.13	54.25	52.69			
75:25	2.87 2.95		44.32	48.14	46.23			
50:50	2.53	2.75	30.36	38.99	34.68			
25:75	2.27	2.32	21.93	23.41	22.67			

Tables 3 and 4 demonstrate that washing significantly affects the printed materials' bending length and flexural rigidity. This indicates that the thickener used in this study is quite washable when cleaned after printing.

		Colou	ır fastne	ss prope	erty of the	e printed	fabric	
Thickeners/	Types of dyes	Washing fastness	Rubbing fastness		Perspiration		Light fast-	
			Dry	wet	Acidic	alka- line	ness	
100:0/St.	MCT	4/5	5	4	4	4/5	6	
75:25	MCT	4	4/5	4	4	4	6	
50:50	MCT	4	4/5	4	4	4	6	
25:75	MCT	4	4/5	4	4	4	6 -7	

Table 5. Fastness properties of the printed fabric

3.12 Colour fastness properties of printed samples

The results of numerous colour fastness tests, such as exposure to light, washing, perspiration, and crocking were conducted on all printed samples as shown in Table 5. In order to examine the colour variations of the printed samples, samples printed with different concentrations of gelatine extracted from hide were compared with a control sample consisting solely of sodium alginate. Consequently, the samples' light and rubbing fastness ranged from very good to excellent.

4. CONCLUSION

The traditional thickener used in reactive printing raises the cost of printing. The protein-based thickener used in this study was extracted from cow hide and combined with sodium alginate in different proportions. When printing reactive dye on cotton fabric at different curing temperatures, the optimal printing conditions were obtained by utilizing different ratios of sodium alginate and gelatine as a thickening. The results indicate that increasing the ratio of gelatine to sodium alginate as a thickening agent enhanced the depth of the printed cloth. When used alone, gelatine has a stronger thickening effect; but, if it is left out for more than eight hours, it becomes unstable in viscosity. Mixing gelatine with sodium alginate is preferred because it has a high viscosity power and is less expensive, while sodium alginate has strong viscosity stability. In terms of colour values, colour fastness properties, and physical qualities of the cotton fabric samples printed with reactive dye, t

using combinations of gelatine and sodium alginate as a thickener were compared with sodium alginate alone. In terms of colour values, the thickener combination produced superior printing outcomes than sodium alginate alone. Nonetheless, each sample's colour fastness characteristics were similar.

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