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1 **Dyeing Recycled Cotton Fibers using *Curcuma***
2 ***Longa* and *Pterocarpus Santalinus* Natural**
3 **Dyes and Bio-mordant Chitosan**
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9 **ABSTRACT**

10 Problems such as the depletion of natural resources and the increase in environmental pollution
11 brought about by industrialization, the concept of recycling gained great importance. For the textile
12 industry, which ranks second in the world by creating the most pollution after the petroleum
13 industry, studies in the field of recycling have gained momentum day by day. Natural dyes, which
14 were used in the coloring of textile products until the 19th century, have gained importance again
15 with the increase in environmental awareness. For this purpose, the dyeability of recycled cotton
16 yarns with natural dyes was examined in order to both protect natural resources and reduce the waste
17 water load in dyeing. The paper also discussed a comparison between different ways to mordant
18 cotton with different molecular weight chitosan. Although it was seen by FTIR analysis that cotton
19 fibers were dyed, the presence of chitosan could not be clearly seen. It was explained by the use of
20 low amounts of chitosan. It was observed that the mordanting processes performed with chitosan
21 contributed to the increase in the color strengths, and higher color depths were obtained with the
22 increasing chitosan concentration. Simultaneous mordanting in *Curcuma longa* dyeing, mordanting
23 before *Sandalwood* dyeing provided higher color depths than others. The washing fastness of the
24 samples dyed with *Curcuma longa* revealed the necessity of fastness improvement processes. It was
25 concluded that chitosan mordanting process was effective in terms of sustainable natural dyeing, but
26 it was necessary to improve fastness.
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31 ***Keywords:* Turmeric, sandalwood, FTIR, K/S, fastness, color**
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50 INTRODUCTION

51

52 The textile processing industry has imposed strict ecological and economic
53 restrictions on the chemicals used, including bans on certain consumer goods
54 containing synthetic agents posing challenges to sustainability issues (Ahlström et
55 al. 2005; Shahid-Ul-Islam et al. 2013). The worldwide demand for the use of
56 environmentally friendly products in the textile industry is nowadays of great
57 interest, possibly because of increasing concern about the environment, ecology,
58 and pollution control (Vankar et al. 2007; Khan et al. 2011).

59

60 It is a fact that the textile industry has grown many times during the last decades to
61 meet global and domestic demand. This tremendous growth has also led to a parallel
62 growth in environmental problems, which remained unnoticed. While any industrial
63 activity produces pollution in one form or the other, the textile industry certainly
64 released a wide spectrum of pollution into the environment. The textile
65 manufacturing process is characterized by the high consumption of resources such
66 as water, fuel and a variety of chemicals in a long process sequence, which
67 generates a significant amount of waste. The common practices of low process
68 efficiency result in substantial wastage of resources and severe damages to the
69 environment (Karthik and Gopalakrishnan 2014; Toprak and Anis 2017).

70

71 Recycling implies the breakdown of a thing into its unrefined materials with the
72 end goal that the rough material can be recuperated and used as pieces of new items.
73 On the other hand, recycle insinuates a present thing being used again inside a
74 comparable creation chain. Textile material recycling is the strategy by which old
75 pieces of clothing and diverse materials are recovered for recycle or material
76 recovery (Wang 2010). Recycling technologies have been a well-established part
77 of the textile industry since the first industrial revolution (Horrocks 1996).
78 Recycling in textile were divided into four methods and classified as: (i) recycling
79 a product into its original form; (ii) mechanical recycling of post-consumer
80 products; (iii) chemical recycling; (iv) waste burning for recovering heat/energy
81 (Vadicherla et al. 2017; Patti et al. 2021). Although the thermal method is used very
82 limited in textile, the 2nd and 3rd methods are generally used (Sandin and Peters
83 2018).

84 In the literature, many papers discussed cotton recycling in different ways. These
85 works could be classified in two groups as: (i) reclaiming of waste (Jeihanipour et
86 al. 2010; Ratanakamnuan et al. 2012; Liu et al. 2019; Wagaye et al. 2020) and (ii)
87 its characterization (Wanassi et al. 2015, 2016; Gun et al. 2016; Haule et al. 2016;
88 Zhang et al. 2016; Dhanapal and Dhanakodi 2020; Vasanth Kumar and Raja 2021).
89

90 Color management with recycled waste textiles is an important issue. However, it
91 still has not received the necessary attention in the literature. Approaches on this
92 subject; (i) removal of color, (ii) separation by color and (iii) the most common of
93 which is to carelessly mix the colors of the wastes to use gray tones or to dye these
94 mixtures in dark tones. (Haslinger et al. 2019; Määttänen et al. 2019). The most
95 preferred approach creates a significant constraint on color, which is the most
96 important reason for preference of textile products.
97

98 Natural dyes with a few exceptions are non-substantive and hence must be used
99 in conjunction with mordants. Mordant is a chemical, which can fix itself on the
100 fiber, and it also combines with the dyestuff. The challenge before the natural dyers
101 in application of natural color is the necessity to chemical mordants which
102 themselves are pollutant and harmful. Due to the environmental hazard caused by
103 metallic mordant while dyeing of textile fabric, dyers are always looking for safe
104 natural mordant for natural dyes (Teli et al. 2013; Chakraborty 2014).
105

106 Chitin is the second most abundant biopolymer in nature after cellulose. It is
107 presented in the outer skeleton of shrimps, lobsters, crabs, and insects (Huang et al.
108 2018; Moattari et al. 2018). Chitosan in the structure of 2-amino-2-deoxy- β -D
109 glucan is obtained by *N*-deacetylation of chitin (Mourya and Inamdar 2008; Kasaai
110 2010). Chitosan is a hydrophilic polymer (Massella et al. 2019) and attracted great
111 interest in textile applications; from fabric pre-mordantings to finishing processes.
112 Some example applications of chitosan use were sizing agent (Stegmaier et al.
113 2008), dyeing auxiliaries (Yen 2001; Ramadan et al. 2011; Kaliyamoorthi and
114 Thangavelu 2015), binder for printing (Bahmani et al. 2000), anti-shrinkage (Huang
115 et al. 2008), anti-static (Abdel-Halim et al. 2010), anti-microbial (Lim and Hudson
116 2003; Alonso et al. 2009), flame-retardant (Leistner et al. 2015) and anti-odour
117 (Hasebe et al. 2001) agents. The applications of chitosan for different applications

118 in textiles are reported, but the application of such functional biopolymer as a
119 mordant in natural dyeing has been quite rare in the literature. Teli and Sheikh
120 (2012) used chitosan extracted from waste shrimp shells as a mordant in
121 simultaneous natural dye printing and anti-bacterial finishing of cotton in
122 comparison with commonly used metal mordants. The efficiency of chitosan as an
123 eco-friendly mordant and anti-bacterial finish has been studied (Teli and Sheikh
124 2012).

125

126 The scope of this research was to create an ecological dyeing process to the fibers,
127 which were already recycled. For this reason, we investigated the effects of using
128 different molecular weight chitosans as pre- and meta-mordants in the dyeing of
129 recycled cotton fibers with two different natural dyes. Recycled cotton fibers were
130 treated by two methods as following: pre-mordanting+dyeing (pre-mordanting with
131 chitosan - drying - dyeing) and meta-mordanting+dyeing (dyeing+simultaneous
132 mordanting with chitosan - drying). In these methods chitosan was used by different
133 concentrations before or together dyeing with natural dyes (*Curcuma longa* and
134 *Sandalwood*). The effects of these various independent variables on dyeing
135 behaviour of the recycled yarns were studied with color measurements (color
136 coordinates and color strength values), and washing fastness. Control samples were
137 obtained by natural dyeing without the use of mordant chitosan. In addition, FT-IR
138 analyses were performed in order to see chemical properties of yarns.

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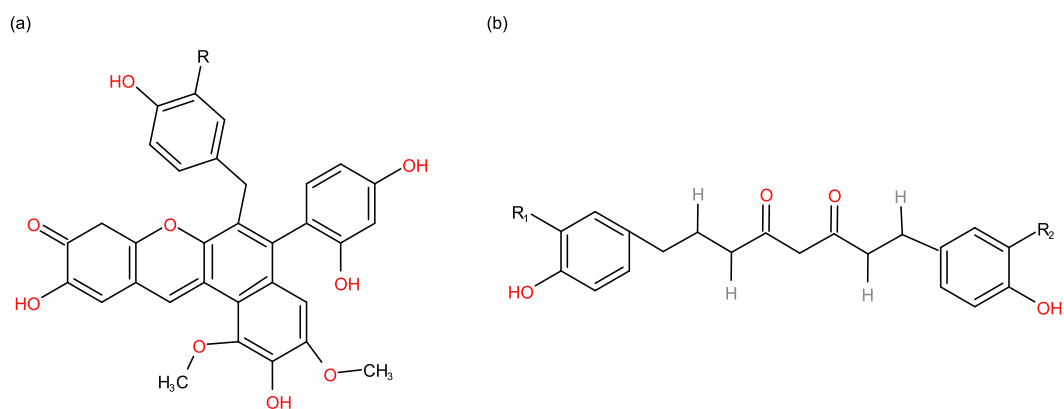
141 **MATERIALS AND METHODS**

142

143 In this study recycled 100% cotton yarns were used. These undyed and recycled
144 cotton fibers were obtained from fiber and clothing waste. Cotton yarns count,
145 which could contain small part of other cellulosic fibers, was Ne 20/1. For chitosan
146 mordanting Sigma-Aldrich brand low molecular weight (LMW) and medium
147 molecular weight (MMW) chitosan bio-polymers were used. The chitosan solution
148 was prepared using soft water and 80% acetic acid of technical grade. *Curcuma*
149 *longa* (Cu) (C.I. Natural Yellow 3) and *Sandalwood-Pterocarpus santalinus* (S)
150 (C.I. Natural Red 22) (Chakraborty 2014) natural dyes used for dyeing. The

151 structure of the dyes are given in Figure 1. The Genclear RWM1 obtained from
152 GenKim was used for washing.

153



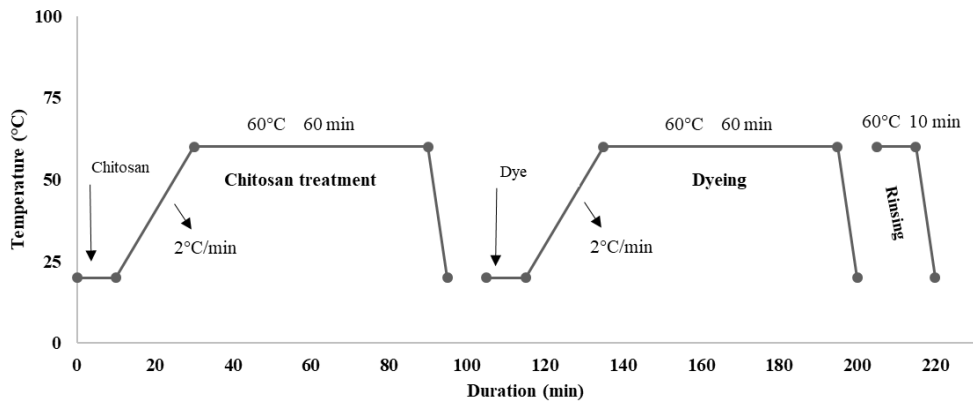
155 **Figure 1.** Structure of (a) *Pterocarpus santalinus*, (b) *Curcuma longa*

156

157 Prepared three different solutions with concentrations of 1%, 3% and 5% (o.w.f.)
158 from two types of chitosan (LMW and MMW). Chitosan was dissolved by the aid
159 of acetic acid at pH 3-4. Chitosan LMW solution prepared during 30 minutes by
160 magnetic stirrer at 1.000 rpm in 24°C. With the same parameters chitosan MMW
161 prepared for 24 hours. Two different natural dyes used by taking the same
162 concentration (10% owf) for each test. Naturel dye solutions prepared by dissolving
163 just in water at neutral pH.

164

165 Chitosan and natural dye solutions were used with two different mordanting called
166 pre- and meta-. In the pre-mordanting, dyeing was performed after the chitosan
167 mordanting, while in the meta-mordanting, chitosan mordanting and dyeing were
168 carried out simultaneously in the same bath. Chitosan solution and dyeing baths
169 liquor ratio was 40:1 (o.w.f.) for wet treatments. The treatments were conducted in
170 sample dyeing machine during 60 minutes at 60°C. These mordanting and dyeing
171 processes started at 20°C and baths heated up to 60°C in 20 minutes by increasing
172 2°C/minute. After dyeing, samples washed with water at 60°C with 4g L⁻¹
173 detergent. All experiments repeated three times for avoid mistakes. The time-
174 temperature diagrams of the processes are given in Figure 2 and Figure 3.

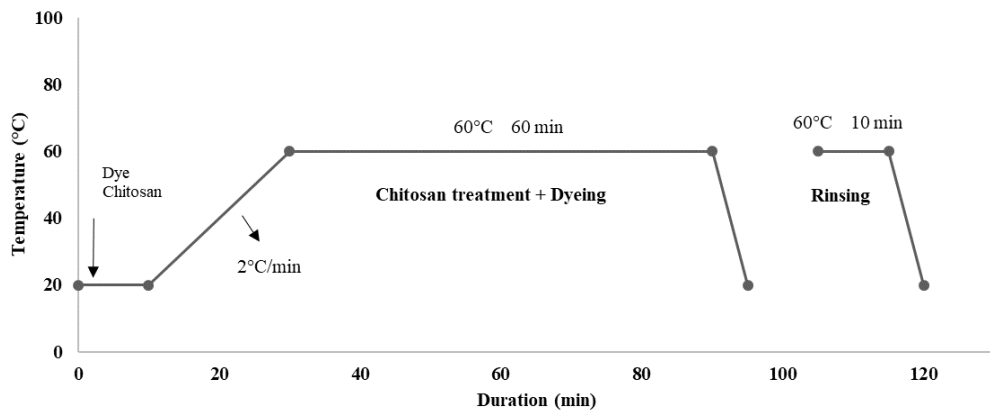


175

176 **Figure 2.** Pre-mordanting and dyeing details

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180 **Figure 3.** Meta-mordanting and dyeing details

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184 Details related with the treatments are given in the Table 1 and Table 2.

185

186

187 **Table 1.** Pre-mordanting and dyeing details

188

Yarn	Chitosan	Chitosan concentration	Treatment phase	Dyeing with
CO	LMW	0%	Dyeing by exhaust with chitosan-mordanted yarns	Cu, S
	LMW	1%		
	LMW	3%		
	LMW	5%		
	MMW	0%		
	MMW	1%		
	MMW	3%		
	MMW	5%		

189

190

191

192 **Table 2.** Meta-mordanting and dyeing details
 193

Yarn	Chitosan	Chitosan concentration	Treatment phase	Dyeing with
CO	LMW	0%	Dyeing by exhaust with chitosan	Cu, S
	LMW	1%		
	LMW	3%		
	LMW	5%		
	MMW	0%		
	MMW	1%		
	MMW	3%		
	MMW	5%		

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197 The infrared spectra of the dyed recycled cotton fabrics were recorded in a
 198 NICOLET iS50 FT-IR spectrophotometer and measurements were performed at
 199 20°C and at a relative humidity of 65%.

200

201 Color coordinates (L^* , a^* , b^* , C^* , and h°) and color strengths were measured by
 202 reflectance measurements (under D65 illuminant and 10° standard observer) the
 203 Konica Minolta CM-3600D spectrophotometer. The K/S values were calculated
 204 using the Kubelka-Munk equation. The color strength (K/S) equation (Akgun et al.
 205 2014) is presented in Equation 1.

206

$$207 \quad K/S = (1 - R)^2 / 2R \quad (1)$$

208

209

210 In the Equation 1, R is the decimal fraction of the reflectance of fabric, K is the
 211 absorption coefficient, and S is the scattering coefficient.

212

213 Fastness properties of the dyed samples were determined according to standard
 214 methods of TS EN ISO 105-C06/A1M (color fastness to domestic and commercial
 215 laundering).

216

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218

219 RESULTS

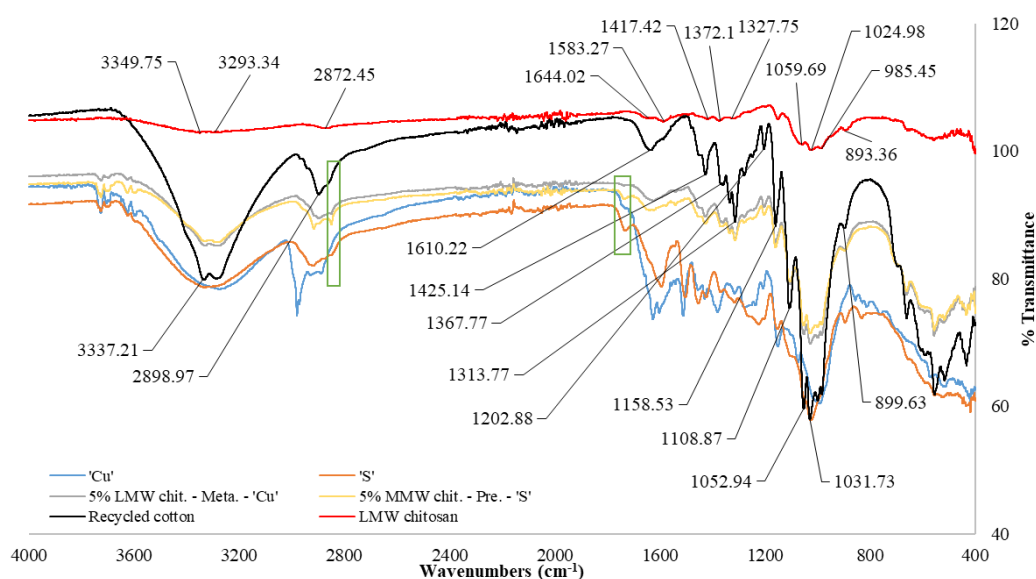
220 FTIR-ATR Analysis

221

222 Figure 4 shows the FTIR spectra of 'Cu' and 'S' dyes, cotton, and low molecular
223 weight chitosan and samples obtained with the highest color depth according to
224 different methods with 'Cu' and 'S' dyes; i.e. dyed with 'Cu' according to the meta-
225 mordanting with LMW chitosan used at 5% concentration, dyed with 'S' according
226 to the pre-mordanting with MMW chitosan used at 5% concentration.

227

228



229

230

231

232 **Figure 4.** FTIR spectra of 'Cu', 'S', dyed samples, cotton, and low molecular weight chitosan

233

234 The recycled cotton fabric showed a broad peak centered at 3337 cm^{-1} was
235 characteristic of the H-bonded hydroxyl (OH) stretching of cellulose, lignin and
236 water. The peak at 2898 cm^{-1} was observed corresponding to the C-H asymmetric
237 and symmetric stretching of cellulose and hemicellulose. The band observed at
238 1610 cm^{-1} could be related to adrobed water. Bands visible in raw cotton at 1425
239 cm^{-1} was assigned to CH wagging (in-plane bending). Peaks around 1367 cm^{-1} and
240 1313 cm^{-1} were relative to CH bending (deformation stretch) and CH wagging
241 vibrations, respectively, of the aromatic rings in cellulose. A peak at 1202 cm^{-1}
242 related to OH in-plane bending. C-O-C asymmetric bridges were observed by peaks
243 at 1158 cm^{-1} and 1108 cm^{-1} . Intense peak vibrations observed at 1052 cm^{-1} and 1031
244 cm^{-1} were related to asymmetric in-plane ring and C-O stretching vibrations of the

245 cellulose, respectively. A peak at 899 cm^{-1} was due to asymmetric out-of-phase
246 ring stretch; i.e. the presence of β -glycosidic linkages between monosaccharides
247 (Chung et al. 2004; Portella et al. 2016). Although the peaks of cellulose showed
248 themselves clearly in the fiber dyed with two different dyes, the band intensities of
249 the peaks of the dyed fibers at similar points decreased compared to cellulose. This
250 was thought to be an indication that the fibers were dyed. In addition, the peaks,
251 which became evident in 2847 cm^{-1} and appeared in 1730 cm^{-1} , in the fiber dyed
252 with 'S', and indicated by the green rectangle, could be caused by the dyeing of the
253 fibers.

254

255 In Figure 4, for the low molecular weight chitosan, the broad peak at 3349 cm^{-1} -
256 3293 cm^{-1} was caused by N-H and -OH stretching vibrations (Corazzari et al. 2015;
257 Radha and Sudha 2018). The peak observed at around 2872 cm^{-1} was due to the
258 typical C-H stretching vibrations (Zhou et al. 2013; Shajahan et al. 2014). The
259 characteristic band at 1644 cm^{-1} and 1583 cm^{-1} were assigned to the amide bands
260 and symmetrical stretching absorption of amino groups (Kong et al. 2010; Tanuma
261 et al. 2010; Yang et al. 2014). The absorption peaks at 1417 cm^{-1} and 1372 cm^{-1}
262 could be assigned to the symmetrical deformation vibrations of CH_3 (Alhosseini et
263 al. 2012; Radha and Sudha 2018). The absorption band at 1327 cm^{-1} and 1059 cm^{-1}
264 were assigned to the C-O stretching in secondary alcohol and asymmetric
265 stretching of the C-O-C, respectively (Prakash et al. 2012; Corazzari et al. 2015).
266 Peaks around 1024 cm^{-1} , 985 cm^{-1} and 893 cm^{-1} related to saccharide structure
267 (Alhosseini et al. 2012). The reason why the peaks of chitosan could not be seen
268 clearly on the dyed fabric was thought to be shadowed by others due to its cellulose-
269 like structure and the low amount of use.

270

271 **Color Coordinates**

272

273 Color coordinates of the yarns which mordanted with different molecular weight
274 chitosans as LMW and MMW and dyed with natural dyes ('Cu' and 'S') are given
275 in Table 3 and Table 4.

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Table 3. Color coordinates of yarns dyed with ‘Cu’ and ‘S’ treated by using LMW chitosan

Dye	Treatment	Chitosan conc. (%)	L*	a*	b*	C*	h°	
Cu		0	76.701 ± 0.13	0.223 ± 0.07	33.237 ± 0.11	33.242 ± 0.13	89.583 ± 0.13	
	Pre	1	75.078 ± 0.09	5.539 ± 0.11	59.642 ± 0.11	59.964 ± 0.15	85.134 ± 0.10	
		3	73.756 ± 0.13	5.763 ± 0.08	62.284 ± 0.09	62.551 ± 0.18	84.718 ± 0.06	
		5	72.911 ± 0.19	6.102 ± 0.08	65.051 ± 0.10	65.287 ± 0.13	84.315 ± 0.06	
	Meta	1	74.946 ± 0.10	6.190 ± 0.19	62.482 ± 0.07	63.785 ± 0.19	84.066 ± 0.18	
		3	71.163 ± 0.11	7.776 ± 0.17	65.965 ± 0.12	66.452 ± 0.18	83.438 ± 0.08	
		5	70.990 ± 0.20	8.016 ± 0.16	67.611 ± 0.09	68.056 ± 0.09	82.065 ± 0.17	
	S		0	69.999 ± 0.14	10.456 ± 0.11	12.612 ± 0.14	16.399 ± 0.20	40.194 ± 0.20
		Pre	1	63.456 ± 0.16	15.879 ± 0.11	9.828 ± 0.05	20.447 ± 0.12	43.289 ± 0.14
3			62.776 ± 0.08	17.356 ± 0.08	11.485 ± 0.18	21.382 ± 0.11	42.071 ± 0.09	
5			60.398 ± 0.20	17.929 ± 0.14	14.834 ± 0.14	23.735 ± 0.08	40.370 ± 0.07	
Meta		1	66.637 ± 0.19	13.910 ± 0.05	5.493 ± 0.09	15.275 ± 0.10	45.675 ± 0.06	
		3	65.649 ± 0.09	14.870 ± 0.11	6.681 ± 0.12	15.835 ± 0.06	44.586 ± 0.06	
		5	65.566 ± 0.05	14.975 ± 0.14	7.287 ± 0.14	16.118 ± 0.11	44.203 ± 0.20	

282
283
284

285 The color hues of the dyeings showed that dyeings with ‘Cu’ obtained colors in the
286 yellow region, and dyes conducted with ‘S’ obtained colors in the red-orange
287 region. In the literature, it was stated that the colors in the yellow region with ‘Cu’
288 were obtained without mordant (Samanta et al. 2014; Yusuf et al. 2017) or tin
289 (stannous chloride) mordant (The Society of Dyers and Colourist 1971). Obtaining
290 yellow color without using mordant showed that chitosan could be used instead of
291 tin. It was also mentioned that the red-orange color was obtained by dyeing the
292 chrome (potassium dichromate) mordanted cotton with ‘S’ (The Society of Dyers
293 and Colourist 1971). However, this color was obtained without using chrome
294 mordant showed that chitosan could be used instead of chrome mordant in addition
295 to tin.

296

297 Application of LMW chitosan as pre- and meta-mordants increased the darkness,
298 redness, yellowness and saturation of colors compared to dyeing without LMW
299 chitosan. It was observed that these changes became more pronounced with
300 increasing chitosan concentration, that is, the usage of LMW chitosan at 5%
301 concentration provided the darkest, reddest, the most yellow and saturated colors.
302 Comparisons of the use of LMW chitosan in meta- and pre-mordantings according
303 to dyes showed that while meta- was the application giving the darkest, reddest,
304 most yellow and saturated colors for ‘Cu’, similar changes were taken from the pre-

305 mordanting for ‘S’. These results showed that both dyestuffs could bind to LMW
 306 chitosan. In addition, the changes in the color coordinates caused by the increase in
 307 LMW chitosan concentration were interpreted as increasing the number of
 308 functional end groups to which the dyestuff could be attached on the fiber.
 309

310 **Table 4.** Color coordinates of yarns dyed with ‘Cu’ and ‘S’ treated by using MMW chitosan
 311

Dye	Treatment	Chitosan conc. (%)	L*	a*	b*	C*	h°	
		<i>0</i>	76.701 ± 0.13	0.223 ± 0.07	33.237 ± 0.11	33.242 ± 0.13	89.583 ± 0.13	
Cu	<i>Pre</i>	<i>1</i>	76.074 ± 0.11	6.047 ± 0.19	48.814 ± 0.12	52.109 ± 0.07	85.415 ± 0.17	
		<i>3</i>	75.485 ± 0.06	6.195 ± 0.07	53.528 ± 0.18	54.773 ± 0.16	85.063 ± 0.06	
		<i>5</i>	75.051 ± 0.06	6.254 ± 0.11	55.172 ± 0.07	55.512 ± 0.18	84.858 ± 0.15	
	<i>Meta</i>	<i>1</i>	74.179 ± 0.19	6.092 ± 0.06	62.376 ± 0.19	62.837 ± 0.12	84.545 ± 0.14	
		<i>3</i>	73.025 ± 0.11	7.535 ± 0.05	63.801 ± 0.08	64.092 ± 0.18	83.422 ± 0.06	
		<i>5</i>	71.456 ± 0.07	7.856 ± 0.06	66.848 ± 0.07	68.303 ± 0.12	83.070 ± 0.16	
		<i>Meta-2hours</i>	<i>1</i>	72.027 ± 0.18	7.685 ± 0.11	64.369 ± 0.08	65.803 ± 0.18	83.431 ± 0.12
			<i>0</i>	69.999 ± 0.14	10.456 ± 0.11	12.612 ± 0.14	16.399 ± 0.20	40.194 ± 0.20
	S	<i>Pre</i>	<i>1</i>	63.087 ± 0.16	17.818 ± 0.16	13.329 ± 0.18	21.961 ± 0.17	28.953 ± 0.16
<i>3</i>			61.608 ± 0.06	17.453 ± 0.10	14.104 ± 0.08	24.142 ± 0.11	25.663 ± 0.19	
<i>5</i>			59.915 ± 0.12	18.087 ± 0.06	14.645 ± 0.07	25.269 ± 0.15	22.211 ± 0.19	
<i>Meta</i>		<i>1</i>	65.528 ± 0.14	16.190 ± 0.19	8.472 ± 0.16	18.287 ± 0.12	32.061 ± 0.07	
		<i>3</i>	64.890 ± 0.07	16.302 ± 0.10	8.627 ± 0.10	19.076 ± 0.12	31.711 ± 0.19	
		<i>5</i>	64.441 ± 0.09	17.125 ± 0.15	9.280 ± 0.15	19.912 ± 0.12	30.726 ± 0.18	
		<i>Meta-2hours</i>	<i>1</i>	63.902 ± 0.19	15.710 ± 0.15	9.970 ± 0.06	20.393 ± 0.16	29.415 ± 0.12

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316 Similar colors were obtained from the ‘Cu’ dyeings in which LMW and MMW
 317 chitosans were used as mordants. In the dyeings conducted using ‘S’ as dye and
 318 MMW chitosan as mordant, it was observed that the color hues decreased, that is,
 319 colors shifted from red-orange region to red region compared to the ‘S’ dyeings
 320 using LMW chitosan as mordant (Table 3). These situations were interpreted as
 321 showing tin and chrome mordant effects of MMW in studies conducted with ‘Cu’
 322 and ‘S’, respectively. Thus, it showed that chitosan could eliminate any harsh
 323 chemical use in improving the color properties of cotton.

324

325 In pre- and meta-mordantings, similar changes in color coordinates caused by
 326 LMW chitosan were also obtained using MMW chitosan, meaning that in
 327 applications using MMW chitosan, colors were darker, redder, more yellow and

328 saturated. As seen in the comparison of the pre- and meta-mordanting with MMW
 329 chitosan, the meta- results in the dyeings conducted with ‘Cu’ were darker, redder,
 330 more yellow and more saturated than the pre-, while these results were obtained in
 331 the pre-mordanting using the ‘S’. Moreover, it was revealed that these changes
 332 could also be seen in applications where ‘Cu’ was used, in processes performed
 333 with lower concentration of chitosan for longer periods (meta-2 hours). For
 334 example, 2 hours meta-mordanting for 1% MMW chitosan concentration gave
 335 higher results compared to 1 hour meta-mordanting for 3% MMW chitosan. It was
 336 observed that these changes in color coordinates were also proportional to the
 337 increase in chitosan concentration. In the treatments carried out using ‘Cu’, it was
 338 observed that the required process to get the darkest, reddest, the most yellow and
 339 saturated color could be meta-mordanting with LMW chitosan. Similar changes in
 340 color coordinates were obtained after MMW chitosan application before dyeing
 341 with ‘S’, that is, in pre-mordanting.

342

343 Color Strengths

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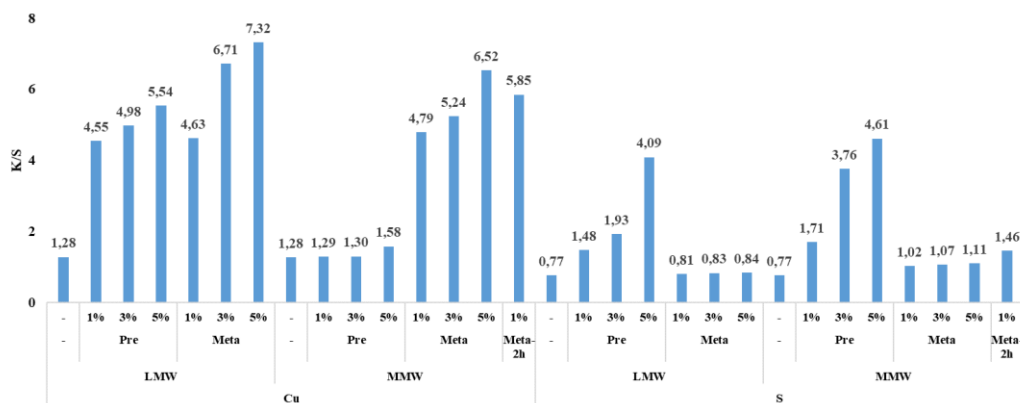
345 In Figure 5, the color strengths obtained after the pre-/meta-chitosan mordanting
 346 and dyeings are given.

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353 **Figure 5.** Color strengths of chitosan-mordanted and natural-dyed yarns

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355

356 In parallel with the L* values (Table 3 and Table 4), LMW chitosan was used
357 together with the 'Cu' (meta-), allowing higher color depths to be obtained
358 compared to other mordanting method. This could be explained by the inability of
359 chitosan to bind the dye sufficiently and the chitosan molecules binding to the
360 cellulose during pre-mordanting, leaving less space for the 'Cu' to bind to the
361 cellulose. In addition, higher color depths in meta-mordanting, where chitosan and
362 dye were applied together, than pre-mordanting might have showed that the dye
363 molecules adhered to the fiber at a higher amount than chitosan. This situation
364 suggested that 'Cu' (long, planar, and conjugated bonds) (Chakraborty 2014) could
365 bound to hydroxyl groups in cellulose in higher amounts rather than amino groups
366 formed by the acetylation of chitosan (Chawla et al. 2015). Despite these, increasing
367 color depths by chitosan mordanting showed that direct dye class member 'Cu'
368 molecules (Yusuf et al. 2017) could have been bound to the positive dyeing sites
369 formed by the cross-linked (Bashar and Khan 2013) of partially protonated chitosan
370 (Jocic et al. 2005) on the fiber surface. It was revealed that in the pre-mordanting
371 carried out with 'Cu' and two different molecular weight chitosan, LMW chitosan
372 provided color depths approximately 4 times higher than MMW chitosan. Although
373 the difference was not as high as in the pre-mordanting, higher color depths were
374 obtained with LMW chitosan than with MMW chitosan in the meta-mordanting
375 with 'Cu'. It turned out that the differences between the crystal structures and
376 solubility of LMW and MMW chitosans (Zheng et al. 2015) affect the amount of
377 dye binding to the surface of the cotton fibers. In addition, the lower color depths
378 obtained from MMW compared to LMW, in meta-mordanting could be explained
379 by a higher amount of MMW binding to fiber compared to LMW (Ang et al. 2013),
380 so could have been leaved less site for 'Cu' bind to fiber. With the increase of
381 chitosan concentration, the color depths increased as stated in the literature (Kavitha
382 et al. 2007), except pre-mordanting with MMW. Although the colors of 'Cu' and
383 'S' were different, the comparison of color strengths was interpreted as the higher
384 color depths obtained with 'Cu' could be resulted from its higher tinctorial strength.
385

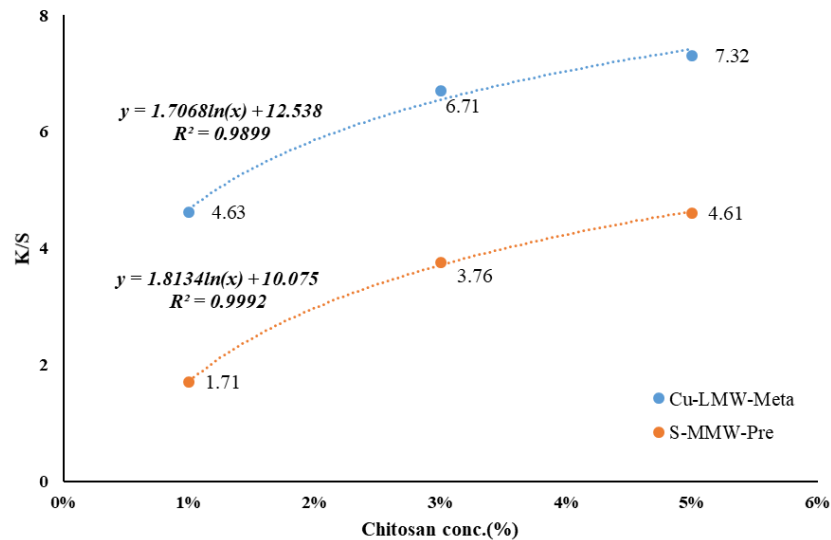
386 The pre-mordanting with LMW and MMW chitosan before dyeing with 'S'
387 provided higher values compared to meta-mordanting and dyeing. This might have
388 showed that 'S' was from the mordant dye class according to the application
389 classification (Yusuf et al. 2017). The concentration increase of LMW and MMW

390 chitosan used before dyeing with ‘S’ led to an increase in color depths. In the meta-
 391 mordanting carried out with ‘S’ dyes, the increase in chitosan concentration did not
 392 create significant differences. Higher color depths were obtained in dyeings using
 393 MMW chitosan in pre-mordanting compared to those using LMW. This could be
 394 explained by the grafting of higher amount of flavonoid-structured ‘S’ dyes onto
 395 the acetyl (C_2H_3O), which MMW had in higher amounts due to its lower degree of
 396 deacetylation compared to LMW (Sivashankari and Prabakaran 2017; Yusuf et al.
 397 2017; Liu et al. 2018).

398

399 The relationship between chitosan concentration and color strength is given in
 400 Figure 6.

401



402

403 **Figure 6.** Color strengths of chitosan-mordanted and dyed yarns

404

405

406 As stated in the comments of color strengths, there was a very strong correlation
 407 between the chitosan concentration and color strength in the meta-mordanting in
 408 which ‘Cu’ and LMW chitosan were used together ($R^2=0.98$). In other words, it
 409 was observed that the color strength increased with the increase in chitosan
 410 concentration, which the concentrations used in this paper. A similar situation was
 411 observed in the treatment performed with MMW chitosan before dyeing with ‘S’
 412 dyes ($R^2=0.99$).

413

414 The image of chitosan treated and ‘Cu’ and ‘S’ dyed fibers, which have the highest
 415 color strength values, is given in Figure 7.

416



417
418

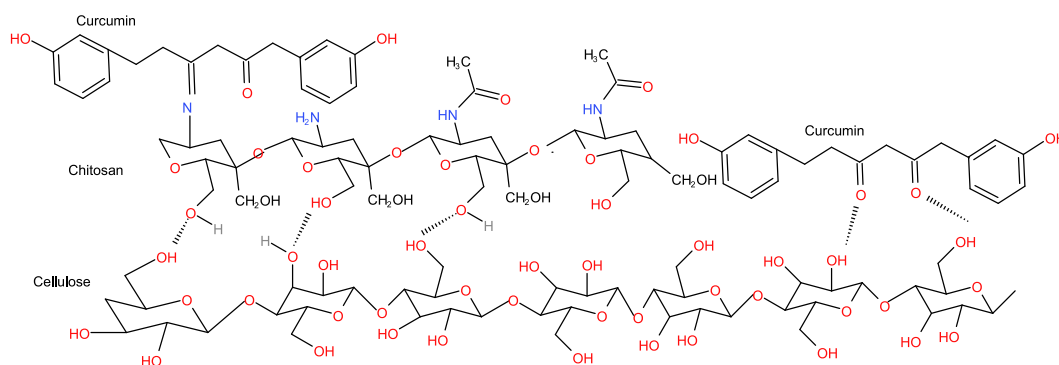
419 **Figure 7.** Samples mordanted with chitosan and dyed by exhaustion

420

421

422 The reaction mechanisms predicted to occur between natural dyes, chitosan and
423 cellulose are given in Figure 8 and Figure 9.

424

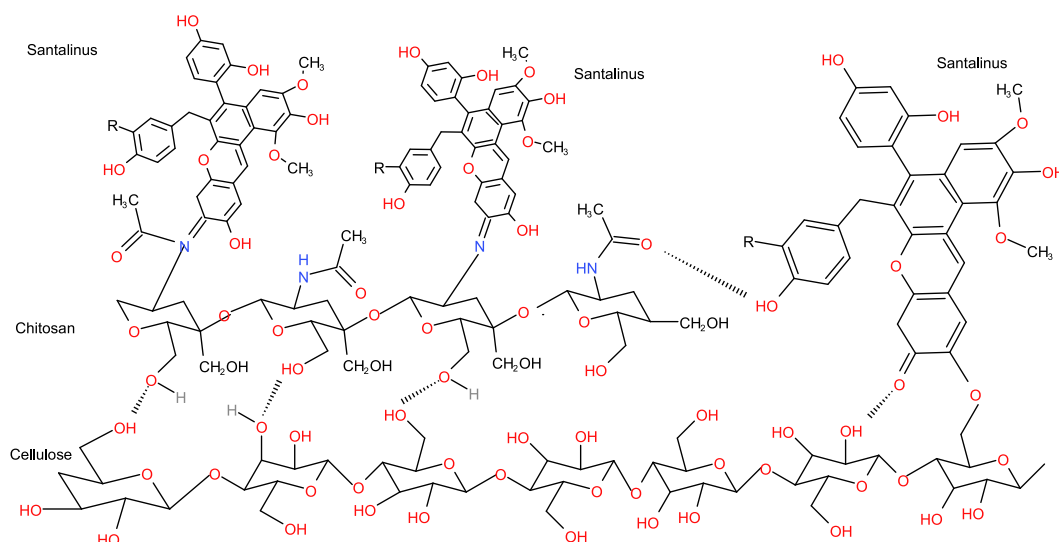


425
426

427 **Figure 8.** Samples mordanted with chitosan and dyed with *Curcuma longa*

428

429



430
431

432 **Figure 9.** Samples mordanted with chitosan and dyed with *Pterocarpus santalinus*

433

434

435 As could be seen from Figure 8, it was estimated that the bonds between linear-
436 chain structured *Curcuma longa* and cellulose-chitosan were mostly composed of
437 ionic and secondary bonds. Although strong covalent bonds were formed, it was

438 thought that these bonds could not be formed up to the *Pterocarpus santalinus* due
 439 to the straight chain structure. It could be that covalent, ionic and secondary bonds
 440 were formed between *Pterocarpus santalinus*, which has a bulky and branched
 441 structure, and cellulose-chitosan, that is, stronger and more bonds were formed
 442 compared to *Curcuma longa*. These cases were also supported by the washing
 443 fastness values.

444

445 Washing Fastness

446

447 Fastness values of samples with the highest color depths are given in Table 5.

448

449

450 **Table 5.** Washing fastness of samples

451

Dye	Chitosan	Mordanting	CA	CO	PA	PET	PAN	WO	Fade	
Cu	-	-	-	4	4	4	5	5	5	1/2
	LMW	1%	Pre-	5	4/5	5	5	5	5	2/3
		5%		4	4	4	5	5	5	2/3
		1%	Meta-	4/5	4	4/5	5	5	5	3
		5%		4	4	4	5	5	5	3
	MMW	1%		5	4/5	4/5	5	5	5	3
		5%		5	4/5	4/5	5	5	5	3
	S	-	-	-	4/5	4/5	4/5	5	5	5
LMW		1%	Pre-	5	5	5	5	5	5	4
		5%		5	5	5	5	5	5	4
MMW		1%		5	5	5	5	5	5	4
		5%		5	5	5	5	5	5	4

452

453

454

455 The washing fastness of the samples, which were mordanted with low molecular
 456 weight chitosan before dyeing with ‘Cu’, decreased by ½ to 1 with the increase in
 457 chitosan concentration. The reason for this situation was thought to be an increase
 458 in color depth. The fading values of ‘Cu’ dyed fibers, which were pre-mordanted
 459 with LMW, were around 2/3. In general, the values of meta-mordanting with LMW
 460 were as low as ½ to 1 compared to pre-mordanting. It was assumed that this
 461 increased staining was due to increased color depth. In meta-mordanting, the
 462 staining values of MMW chitosan were slightly higher, i.e. better, compared to
 463 LMW. Staining was generally seen in CA, CO and PA fibers. The fading values of
 464 the samples dyed with ‘Cu’ in the presence of chitosan were ½ point higher than
 465 the mordanting performed before dyeing. As a result, the need to improve the low
 466 fading values obtained due to weak hydrogen bonds between chitosan and
 467 carotenoid-structured ‘Cu’(Thamaket and Raviyan 2015) emerged.

468 It was observed that samples mordanted with chitosan before dyeing with ‘S’ did
469 not stain the multifiber. Their fading values were higher than those of the pre- and
470 meta-mordanted ‘Cu’ dyed samples. This could be explained by the covalent
471 binding of the bulky phenolic ‘S’ to the amino group of chitosan (Hu and Luo 2016).
472 It was also predicted that many secondary bonds were formed between the branched
473 ‘S’-cellulose-chitosan. Chitosan improved staining and fading values in both
474 dyeings.

475

476

477 **DISCUSSION**

478

479 Climate changes, one of the biggest crises that the world is struggling with, are
480 caused by human beings’ polluting the environment by wasting resources,
481 overconsumption etc. High amounts of water and pesticides are consumed in the
482 production of cotton, which is the most preferred natural fiber in textile as the
483 second most polluting sector in the world. Most dyestuffs used to color textile
484 products pose a great threat to the environment. Chitosan is a versatile biopolymer
485 with a variety of commercial applications. For this reason, in this study, the effects
486 of coloring the recycled cotton with natural dyes and the use of chitosan as a
487 mordant were investigated. In dyeing studies carried out with natural dyes of two
488 different structures, it was observed that the molecular weights of chitosans and the
489 effects of using it before and during dyeing were different. Dyeing studies showed
490 that chitosan was used as a biomordant to give cotton the ability to be dyed with
491 natural dyes. Low fastness values, which is the general problem of natural dyes,
492 showed themselves in this study as well. It is also known that the samples obtained
493 from this study carried out with chitosan, turmeric and polyphenol have many
494 superior biological and physicochemical properties. For this reason, in future
495 studies, it is planned to improve the fastness values, investigate the other effects of
496 these components, and further develop the functional properties and biological
497 activities of chitosan with appropriate modification. This research as a first step of
498 further experiments, provide us the optimized values and applications for the future
499 researches.

500

501

502 **Declarations Conflict of interest**
503 The authors declare that they have no conflicts of interest.
504

505 **Ethical approval**
506 No animal or human trials were undertaken or conducted for this study.
507

508 **Consent to participate**
509 All the three authors are aware of the submission and have given their consent to participate. All
510 the authors have read and approved the manuscript before submission.
511

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514
515
516

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