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Preparation, characterization and technological evaluation of CMC derived from rice-straw as thickening agents in discharge, discharge–resist and burn-out printing

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ABSTRACT

Carboxymethyl cellulose samples of different D.S. values were prepared from rice straw via pulping followed by etherification with monochloroacetic acid under the catalytic action of sodium hydroxide. The prepared derivatives were assessed for D.S., rheological properties as well as suitability as thickening agents in different printing styles. It was found that carboxymethyl cellulose derived from rice straw is characterized by a non-Newtonian pseudoplastic behavior and its apparent viscosity at any specific rate of shear depends on the D.S. All the prepared derivatives could be used successfully as thickening agents in discharge and/or discharge/resist printing of cotton fabrics. Furthermore, they could be used also as thickening agent for burn-out printing style of wool/polyester blended fabrics using sodium hydroxide. Attractive samples could be obtained via using these techniques.

1. Introduction

Rice straw is one of the abundant lignocellulose waste materials in the world. In terms of total production, rice is the third most important grain crop in the world behind wheat and corn. As per FAO statistics, world annual rice production in 2007 was about 650 million tons. Every kg of grain harvested is accompanied by production of 1–1.5 kg of straw (Maiorella, 1985). It gives an estimation of about 650–975 million tons of rice straw produced per year globally and a large part of this is going as cattle feed and rest as waste. The options for the disposition of rice straw are limited by the low bulk density, slow degradation in the soil, harboring of rice stem diseases, and high mineral content. Nowadays, field burning is the major practice for removing rice straw, but it increases the air pollution and consequently affects public health (Parameswaran et al., 2010).

Rice straw is one of the most important problems in Egypt, since the farmers oblige to burn it. The gases and fumes of burning cause a pollution of the environment. Converting the rice straw to useful product viz. carboxymethyl cellulose (CMC) has great economical and environmental advantages. CMC is characterized by high viscosity and good film forming properties. That is why CMC finds many applications in textile industry as printing pastes, sizes, finishing and lubricants (Abd El-Rahman, Nassar, & Abd El-Thalouth, 2002).

The main methods of carboxymethylation are the aqueous and the non-aqueous (Cheng & Biswas, 2011; Leong, Chung, Noordin, & Takayama, 2011; Parvathy, Susheelamma, Tharanathan, & Gaonkar, 2005; Saghir, Hussain, & Heinze, 2011; Togrul & Arslan, 2003; Varshney, Gupta, Bhatt, & Soni, 2006). The cellulose pulp can be produced from different agricultural wastes, like rice straw, cotton stalks and wood manufacturing wastes. It has been reported that carboxymethylation of the pulp obtained from these wastes gives a degree of substitution (D.S.) and rheological properties of the CMC which rely on the plant source and degree of purity of cellulose pulp.

The present work aims to prepare pure cellulose from rice straw, and subjecting it to carboxymethylation (using different concentrations of the etherifying agent) and to investigate its chemical and rheological properties. Furthermore, the suitability of the prepared derivatives as thickening agents in discharge, discharge–resist and burn-out printing was also investigated.

2. Experimental

2.1. Materials

2.1.1. Substrate

1. Mill desized, bleached and mercerized cotton fabrics, produced by Misr El-Mehalla for Spinning and Weaving Company, El-Mehalla, Egypt, were used.

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2. Wool/polyester (70/30) blended fabric, produced by Misr El-Mehalla for Spinning and Weaving Company, El-Mehalla, Egypt, was used.

2.1.2. *Rice straw*

Native rice straw was supplied by Racta Co. for Paper Manufacture, Alexandria.

2.1.3. *Thickening agents*

1. High viscosity sodium alginate from brown algae, manufactured by Fluka Chemical Company, was used as a thickening agent.
2. British gum, supplied by Misr Company for Starch and Glucose, Cairo, Egypt.
3. Mypro gum T-8, supplied by El-Mehalla for Spinning and Weaving Company, manufactured at Germany.

2.1.4. *Dyes*

Reactive dyes: Sunzole Red F3B and Sunfix Yellow MF-D were supplied by Ohyoung Industrial Co. Korea, their reactive center is vinylsulphone.

2.1.5. *Chemicals*

Sodium hydrosulphite, titanium dioxide, glycerine, toluene, sodium hypochlorite, aluminum potassium sulphate, monochloroacetic acid, sodium hydroxide, potassium iodide, copper sulphate, sodium thiosulphate, ethyl alcohol, ammonium hydroxide, acetic acid, urea and sodium bicarbonate were of laboratory grade Chemicals. Commerical Medoxy T supplied by El-Nassr Company for Spinning, Weaving and Dyeing, Mehalla El-Kobra, Egypt.

2.2. *Methods*

2.2.1. *Preparation of cellulose pulp*

α-cellulose was prepared from native rice straw according to the following procedure:

1. Treatment with sodium hydroxide solution 4%, liquor ratio 1:5 in an autoclave at 120 °C for 2 h followed by thoroughly washing.
2. Bleaching with sodium hypochlorite (NaOCL) solution 4 g/l, liquor ratio 1:10 for 2 h at room temperature followed by thoroughly washing with running water and finally subjecting to drying at ambient conditions.

2.2.2. *Non-aqueous carboxymethylation*

The prepared cellulose pulp was subjected to carboxymethylation according to the following procedure:

100 g of dry pure cellulose powder was added gradually to a mixture of 630 ml of ethyl alcohol and 554 ml of toluene. A solution of 44.8% sodium hydroxide containing the required amount of alkali was then added gradually during mechanical stirring. The reaction mixture was mixed thoroughly and steeped at room temperature for 30 min. After that, the required amount of monochloroacetic acid was added gradually with agitation.

The reaction mixture was left overnight at room temperature. The excess alkali was neutralized with glacial acetic acid using phenolphthalein as indicator. The product was filtered off, washed with ethyl alcohol and purified before subjecting to analysis.

Using the above general procedure, three different derivatives using 75, 100 and 125 g monochloroacetic acid/100 g cellulose pulp were prepared. The amount of sodium hydroxide was 2 mol/1 mol monochloroacetic acid.

Purification: The prepared CMC derivatives were purified via extraction in Soxhlet using 75% ethylalcohol till free from salts.

2.2.3. *Dyeing of cotton fabric*

The fabric was padded in a solution containing:

Dye 3% (O.W.F.)	Wetting agent 2 g/l
Sodium carbonate 35 g/l	L.R = 1:30

Pick up 70% at room temperature.

2.2.4. *Printing techniques*

2.2.4.1. *White discharge printing.* The paste was prepared according to the following recipe:

Reducing agent (glucose + sodium hydrosulphite)[a]	250 g
Titanium dioxide	100 g
Thickening agent[b]	Y g
Water	X
Total	1000 g

[a] The ratio of glucose:sodium hydrosulphite was varied as represented in the tables.

[b] Thickening agents used were either sodium alginate (30 g) or the prepared CMC (50 g/kg).

2.2.4.2. *Discharge–resist printing.* The recipe of the uncolored printing paste was as follows:

Combination of glucose and sodium hydrosulphite	250 g
Titanium dioxide	100 g
Thickening agent	Y g
Water	X
Total	1000 g

Thickening agents used were either sodium alginate (30 g/kg) or the prepared CMC (50 g/kg).

The printed cotton samples were then over printed using printing pastes containing one of the aforementioned two reactive dyes, using the following recipes:

Reactive dye	30 g
Urea	100 g
Sodium alginate	30 g
Sodium bicarbonate	30 g
Water	X g
Total	1000 g

Fixation: The discharge and discharge–resist printed fabrics were fixed via steaming at 102 °C for 15 min.

Washing: The fixed discharge and discharge–resist printed fabrics were subjected to washing through five stages as follows:

- Rinsing thoroughly with cold water.
- Treatment with hot water.
- Treatment near the boiling temperature (90–95 °C) with a solution containing 2 g/l Aspkon 1030 (neutral detergent).
- Washing with hot water.
- Rinsing with cold water.

2.2.4.3. *Burn-out printing of wool/polyester blend.* The printing pastes containing the following recipe:

Thickening agents	X g/kg
Sodium hydroxide	250 g/kg
Glycerin	50 g/kg
Water	Y
Total	1000 g

Thickening agents used were either British gum (400 g) or CMC (50 g/kg).

Fixation: The burn-out printed wool/polyester blended fabrics were fixed via steaming at 102 °C for 10 min.

Washing: The burn-out printed wool/polyester blended fabrics were rinsed in a bath containing 2 ml acetic acid (20%) per liter water to neutralize the alkali/cold followed by thoroughly washing.

2.3. Analysis and measurements

2.3.1. Determination of degree of substitution (D.S.)

The D.S. of the prepared CMC samples was determined by the standard method (Green, 1963; Varshney et al., 2006).

2.3.2. Measurements of the rheological properties

The rheological properties of the printing pastes were measured using Rheomat-15 Zurich, Switzerland at 25 °C and the apparent viscosity (η) at various rates of shear was calculated from the shearing stress (τ) and rates of shear (D) as follows:

$$\eta = \frac{\tau}{D}$$

2.3.3. Color measurements

The color strength expressed as K/S was assessed according to a standard method (Judd & Wyszecki, 1975).

3. Results and discussion

3.1. Carboxymethylation

In the present work, the cellulose was extracted from rice straw according to the procedure described in Section 2 and subjected to carboxymethylation with monochloroacetic acid in the presence of sodium hydroxide. Cellulose can react with monochloroacetic acid as follows (Kantouch, 1994):

$$\text{Cell.OH} + \text{NaOH} \rightarrow \text{Cell.ONa(alkalicellulose)}$$

$$\text{Cell.ONa} + \text{ClCH}_2\text{COOH} \rightarrow \text{Cell.O.CH}_2\text{COONa} + \text{NaCl}$$

The carboxymethylation reaction was carried out in nonaqueous medium using different concentrations of the etherifying agent to obtain carboxymethylated derivatives acquiring different degrees of substitution (D.S.) values.

The effect of concentration of monochloroacetic acid on the extent of carboxymethylation reaction expressed as D.S. is shown in Table 1.

It is clear from the data of Table 1 that the extent of carboxymethylation reaction expressed as D.S. increases by increasing monochloroacetic acid concentration.

For example, when the concentration of monochloroacetic acid was 75 g/100 g of pure cellulose, the D.S. was 0.66, while the amount of monochloroacetic acid was increased to 125/100 g pure cellulose, the D.S. was increased to 1.03.

It is worthy to mention that untreated pure cellulose is insoluble in water. The current data indicates that modification of pure cellulose via carboxymethylation converts it into a water-soluble product. This holds true regardless of the D.S. value in the range studied.

3.2. Rheological properties

Since carboxymethyl cellulose derivatives are generally used in the form of viscous solutions, it seemed of interest to investigate the viscometric and rheological properties of their pastes. Hence, pastes of the aforementioned derivatives were prepared at a concentration of 5%. The rheological properties of the pastes were monitored before and after storing of the pastes for three days. The results obtained are shown in Figs. 1 and 2.

Table 2
Effect of D.S. of carboxymethyl cellulose on the apparent viscosity at various rates of shear for freshly prepared pastes.

Rate of shear (s^{-1})	Apparent viscosity in poise on using carboxymethyl cellulose of		
	D.S. = 0.66	D.S. = 0.81	D.S. = 1.03
3.85	–	35.55	35.55
5.14	26.63	31.96	31.96
6.78	24.23	28.26	28.26
9.77	19.61	25.22	28.02
13.12	16.69	22.95	27.12
17.26	14.27	20.62	26.96
23.03	11.88	17.83	23.77
30.38	9.91	16.22	20.71
44.1	8.07	13.03	16.41
59.22	6.93	11.09	13.4
77.92	6.32	9.48	11.24
103.9	5.53	7.9	9.22
137.1	4.59	6.79	7.58

The rheograms 1 and 2 reveal that the pastes under investigation display non-Newtonian pseudo plastic behavior since the ascending and descending rheograms are coincident. This reveals the homogeneity of the molecular structure of the pastes which are amenable to rebuild themselves and retain their original state immediately after removal of the acting force, thereby exhibiting pseudo plastic behavior.

Fig. 1 also shows that the location of the rheograms with respect to the rate of shear axis depends on D.S. of the carboxymethyl derivative. As the degree of carboxymethylation increases the rheogram is located far from the rate of shear axis indicating an increase in the apparent viscosity as it is clear from Table 2.

Fig. 2 represents the rheograms of the aforementioned pastes after storing for three days. It is clear from the rheograms that storing has practically no effect on the rheological characteristics of these pastes where each of these pastes remains exhibiting non-Newtonian pseudo plastic behavior after storing for three days.

3.2.1. Apparent viscosity

The apparent viscosity at various rates of shear of the aforementioned pastes was calculated from the values of shearing stress and rate of shear. The results obtained are given in Tables 2 and 3 before and after storing for three days.

It is clear from the data of Table 2 that as the D.S. of carboxymethyl cellulose increases the apparent viscosity increases. This phenomenon holds true regardless of the rate of shear. For example at a rate of shear of 13.12 s^{-1} .the apparent viscosity increases from 16.69 to 22.95 to 27.12 poise as the D.S. increases from 0.66 to 0.81 to 1.03, respectively.

The increase in the apparent viscosity by increasing the D.S. may be due to the increase in the molecular weight of the obtained CMC as the D.S. increases.

Table 1
Effect of concentration of monochloroacetic acid on D.S. and solubility of carboxymethyl cellulose derivatives.

Weight of cellulose (g)	Weight of $Cl.CH_2COOH$ (g)	Weight of NaOH (g)	D.S.	Solubility in	
				Water	Ethyl alcohol
Untreated	0.00	0.00	–	Insoluble	Insoluble
100	75	75	0.66	Soluble	Insoluble
100	100	100	0.81	Soluble	Insoluble
100	125	125	1.03	Soluble	Insoluble

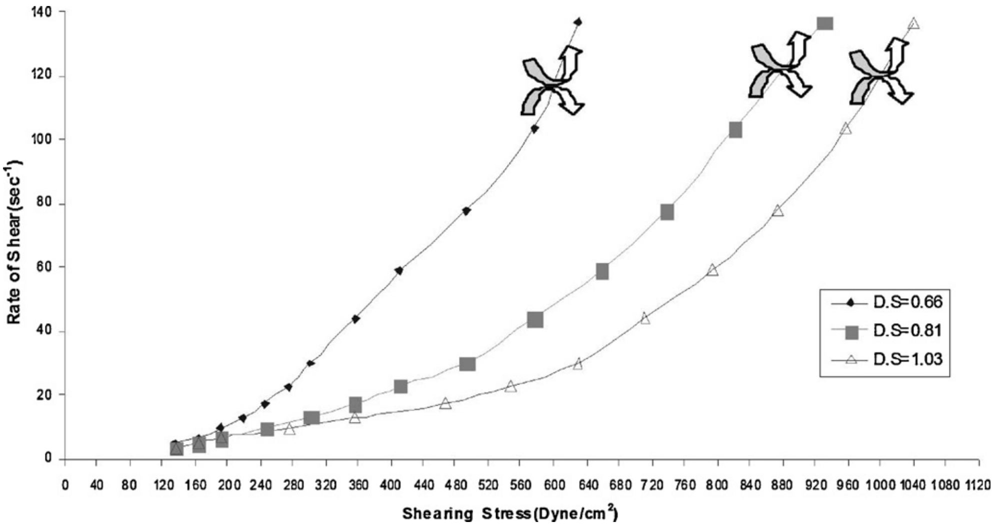

Fig. 1. Rheograms of freshly prepared carboxymethyl cellulose pastes derived from rice straw.

Table 3
Effect of D.S. of carboxymethyl cellulose on the apparent viscosity at various rates of shear for prepared pastes after storing for three days.

Rate of shear (s^{-1})	Apparent viscosity in poise on using carboxymethyl cellulose of		
	D.S. = 0.66	D.S. = 0.81	D.S. = 1.03
2.18	–	–	62.79
2.93	–	46.72	56.06
3.85	35.55	42.67	56.89
5.14	31.96	37.28	53.26
6.78	28.26	36.34	52.49
9.77	25.22	30.82	47.64
13.12	22.95	27.12	43.82
17.26	22.2	26.96	38.07
23.03	20.21	24.96	33.28
30.38	18.02	22.53	28.84
44.1	14.27	18	22.35
59.22	12.02	15.25	18.49
77.92	10.19	13	15.46
103.9	8.43	10.8	12.64
137.1	6.98	8.98	10.58

On comparing Tables 2 and 3 it is obvious that storing of the current pastes for three days causes a slight increase in the apparent viscosity.

For example the sample of D.S. 0.66 at a rate of shear 13.12 s^{-1} it increases from 16.69 to 22.95 for the freshly prepared and after storing for three days.

The increase in the apparent viscosity by storing reflects the stability of the pastes and may be due to the increase in the swell ability and adhesive power of the pastes on storing

3.3. *Printing*

As previously indicated, the main aim of the present work was to investigate the suitability of carboxymethyl cellulose derivatives as thickeners in discharge, discharge–resist and burn-out printing. To achieve this goal, series of printing pastes thickened with the prepared carboxymethyl cellulose derivatives of different D.S. values (0.66, 0.81 and 1.03) were prepared. For the sake of comparison, other pastes thickened with sodium alginate, British gum were also prepared. After printing and drying of the printed goods, the fixation was carried out via steaming, given below the results obtained along with the appropriate discussion.

3.3.1. *Discharge printing*

To investigate the suitability of CMC samples derived from rice straw as thickening agents in discharge printing, cotton fabric

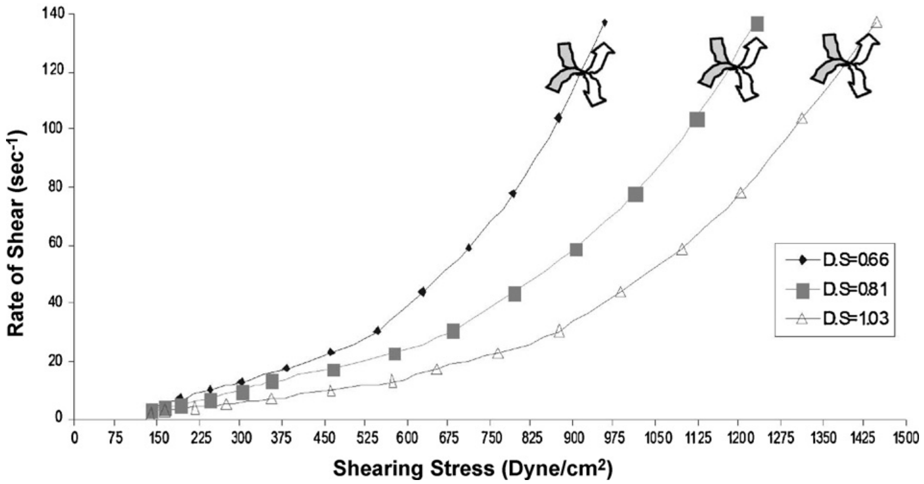

Fig. 2. Rheograms of carboxymethyl cellulose pastes derived from rice straw after storing for three days.

Table 4
Effect of sodium hydrosulphite: glucose ratio on % decrease in K/S and whiteness degrees of discharge printed samples.

Thickener used	Ratio of sodium hydrosulphite: glucose	Discharge printed samples		Whiteness degrees
		K/S	% decrease in K/S	
Sodium alginate	2:1	0.62	80.92	90.78
	1:1	1.13	59.69	58.62
	1:2	1.36	58.15	56.42
	1:4	1.41	56.61	50.21
Carboxymethyl cellulose D.S. 0.66	2:1	0.63	80.61	90.24
	1:1	1.3	60	58.64
	1:2	1.34	58.76	56.64
	1:4	1.4	56.92	50.31
Carboxymethyl cellulose D.S. 0.81	2:1	0.64	80.3	89.71
	1:1	1.29	60.3	95.24
	1:2	1.32	59.38	56.84
	1:4	1.39	57.23	50.42
Carboxymethyl cellulose D.S. 1.03	2:1	0.66	79.69	88.43
	1:1	1.31	59.69	58.33
	1:2	1.36	58.15	56.79
	1:4	1.44	55.69	50.77

Samples were dyed via padding with Sunzole Red F3B.
The K/S of the original dyed samples was 3.25.

samples were dyed using a dischargeable reactive dye namely Sunzol Red 3B according the procedure described in Section 2.

Printing pastes thickened with either CMC or sodium alginate containing different ratios of sodium hydrosulphite: glucose (viz. 2:1, 1:1, 1:2 or 1:4) were prepared and used in printing the colored cotton fabrics. After printing the goods were subjected to steaming at 102 °C for 15 min followed by washing as previously mentioned in Section 2. After washing and drying the fabrics were assessed for K/S and degree of whiteness. The results obtained were represented in Table 4.

Generally speaking it is clear from the data of Table 5 that as the ratios of glucose increase, the K/S increases and the % decrease in K/S decreases. This phenomenon holds true regardless of the thickening agent used, i.e. in case of using sodium alginate or any sample of the prepared CMC.

The current data are expected since as the glucose ratio increases the sodium hydrosulphite which is more powerful reducing agent decreases and hence the % decrease in K/S decreases.

For example on using CMC of D.S. 0.81 the % decrease in K/S decreases from 80.3 to 60.3 to 59.38 to 57.23 as the sodium hydrosulphite:glucose ratios varied from 2:1 to 1:1 to 1:2 to 1:4, respectively.

The current data indicates that the nature of the thickening agent used has no remarkable effect.

Table 4 also shows the values of the degree of whiteness. It is clear that as the % decrease in K/S decreases the degree of whiteness also decreases. Where it decreases from 90.24 to 50.31 as the % decrease in K/S decreases from 80.61 to 56.92 in case of using CMC of D.S. 0.66.

It can be concluded that water soluble CMC derived from rice straw could be used successfully as thickening agent in discharge printing of cotton fabrics, regardless of the value of its D.S. in the range studied.

3.3.2. *Discharge/resist printing*

To investigate the suitability of the prepared different CMC samples as thickening agent in discharge/resist printing, samples of cotton fabrics were printed with uncolored printing paste containing different ratios of sodium hydrosulphite/glucose (2:1, 1:1, 1:2 and 1:4) and thickened with either sodium alginate or CMC derivative. After printing and drying, the printed fabrics were over printed using an open screen by a paste containing a reactive dye namely either Sunzole Red F3B or Sunfix Yellow MF-D with sodium alginate as thickening agent. After overprinting the fabrics were subjected to steaming for 15 min at 102 °C followed by washing as mentioned in Section 2. Finally the fabrics were dried at ambient conditions and assessed for K/S. The results obtained are given in Table 5.

It is clear from the data that, irrespective of the nature of the thickening agent used or the value of the D.S. of the used CMC or the reactive dye used, as the percent of sodium hydrosulphite increases the K/S decreases, hence the % decrease in K/S increases. The highest % decrease in K/S was obtained at a ratio of 2:1 sodium hydrosulphite:glucose.

The current data reveal that there is no remarkable difference between the prepared CMC samples and sodium alginate, which reflect the success of these derivatives in discharge/resist printing under the aforementioned experimental technique.

3.3.3. *Burn-out printing*

Finally, a trial was made to investigate the suitability of the prepared CMC derivatives as a thickening agent in burn-out printing. To achieve this, sample of wool/polyester blend (70/30) was chosen, and a burn-out printing paste containing 250 g/kg sodium hydroxide (thickened with either British gum or CMC sample derived from rice straw) were prepared. The blended fabric samples were printed with the prepared pastes, followed by drying at ambient conditions. The printed goods were steamed at 102 °C for 30 min followed by

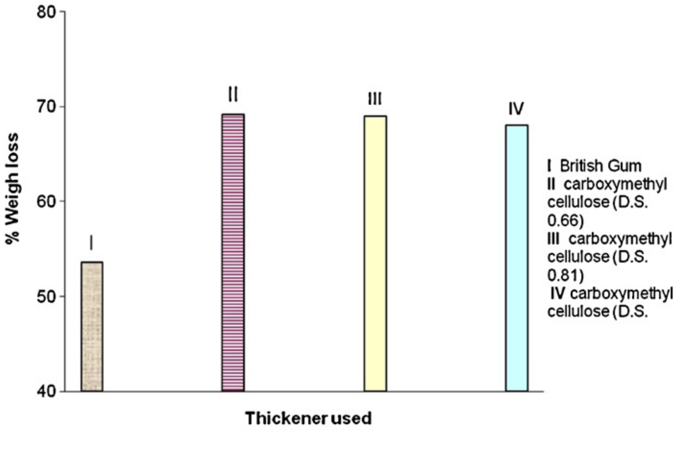

Fig. 3. Weight loss of wool/polyster (70:30) blended fabrics printed with burn-out using carboxymethyl cellulose derivatives.

Table 5
Effect of sodium hydrosulphite: glucose ratio on % decrease in K/S of discharge–resist printed samples.

Thickener used	Ratio of sodium hydrosulphite: glucose	Discharge printed samples on using Sunzole Red F3B		Discharge printed samples on using Sunfix Yellow F-D	
		K/S	% decrease in K/S	K/S	% decrease in K/S
Sodium alginate	2:1	0.07	97.66	0.57	95.25
	1:1	0.14	95.33	0.71	94.08
	1:2	0.16	94.66	0.76	93.66
	1:4	0.26	91.33	0.78	93.5
Carboxymethyl cellulose D.S. 0.66	2:1	0.09	97	0.56	95.33
	1:1	0.15	95	0.72	94
	1:2	0.16	94.66	0.76	93.66
	1:4	0.29	90.33	0.79	93.41
Carboxymethyl cellulose D.S. 0.81	2:1	0.07	97.66	0.56	95.33
	1:1	0.14	95.33	0.72	94
	1:2	0.17	94.33	0.76	93.66
	1:4	0.27	91	0.81	92.25
Carboxymethyl cellulose D.S. 1.03	2:1	0.07	97.66	0.56	95.33
	1:1	0.15	95	0.71	94.08
	1:2	0.16	94.66	0.76	93.66
	1:4	0.27	91	0.78	93.5

Samples were fixed via steaming.

washing. The loss in weight was measured and the results obtained are represented in Fig. 3.

It is clear from the data that the % loss in weight depends on the nature of the thickening agent used. Where the loss in weight of the blended fabric on using CMC derivatives is higher than their corresponding sample printed using British gum. This phenomenon holds true regardless of the D.S. of the used CMC derivatives.

However, increasing the D.S. of the CMC is accompanied by a slight decrease in % loss, whereas the D.S. increases from 0.66 to 0.81 to 1.03 the % loss in weight decreases from 69.2 to 69 to 68, respectively. The decrease in the loss in weight as the D.S. of CMC increases may be due to the increase in the apparent viscosity as previously mentioned, as the viscosity increases, the penetration of the paste inside the printed fabric decreases and hence its effect decreases too.

4. Conclusion

Water soluble carboxymethyl cellulose samples of different D.S. values were prepared from rice-straw, the rheological properties of their pastes, their solubility in cold water as well as suitability as thickening agent in discharge, discharge–resist and burn-out printing styles were investigated.

The results obtained from this study can be summarized as follows:

1. The extent of the reaction expressed as D.S. increases by increasing the etherifying reagents, i.e. monochloroacetic acid and sodium hydroxide.
2. Modification of cellulose via carboxymethylation converts it into a water-soluble product, and increases the stability of its pastes to microorganism's fermentation.
3. Regardless of the D.S. of these products, their aqueous solutions are characterized by pseudo plastic behavior.
4. The D.S. of the products has a remarkable effect on apparent viscosity of their pastes at a constant rate of shear. It increases by increasing the D.S.
5. Carboxymethyl cellulose derivatives can be used successfully as thickening agents in discharge, discharge–resist and burn-out printing of wool/polyester blend.

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