

TECHNOLOGICAL EVALUATION OF CARBOXYMETHYL SESBANIA GALACTOMANNAN GUM DERIVATIVES AS THICKENERS IN REACTIVE PRINTING

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Galactomannan gum isolated from the seeds of sesbania was subjected to chemical modification via carboxymethylation in non-aqueous medium using monochloroacetic acid under the catalytic action of sodium hydroxide. The obtained derivatives were subjected to measuring the degrees of substitution (D.S.), rheological properties of their pastes, and evaluated as thickeners in printing cotton fabrics with reactive dyes. Highly substituted derivatives could be used successfully as thickening agents in printing cotton fabrics either alone or in admixture with sodium alginate. Prints acquire soft handle, colour strength (K/S), and fastness properties nearly identical to corresponding samples that were printed using sodium alginate.

Keywords: Carboxymethylation; Galactomannan gum; Rheology; Sesbania seeds; Textile Printing

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INTRODUCTION

Sesbania (family: Leguminosae), is an Egyptian fast growing spreading tree. The sesbania seeds consist of three components namely: coat (6.9-18.9%), endosperm (30-42 %), and germ (51.1 %) (Faroogi et al. 1972).

The endosperm is composed of a mannan backbone linked together by β - (1 \rightarrow 4) glycosidic linkages and having galactose side chain residues linked by α - (1 \rightarrow 6) i.e.galactomannan. It has been reported that acid hydrolysis of sesbania galactomannan gum yielded galactose and mannose in the ratio of 1.2:2.2 compared to 1 to 3.9 for Locust bean (carob) , 1 to 2 for Guar gum and 1 to 3 for Tara gum. The different degree of branching is believed to account for the differences in properties between galactomannan gums. A greater number of side units reduces the amount of molecular association and increases cold water dispersion of the gum (Evans and Roter 1987; Gutteridge 1998; Heering et al. 1992; Parvathy et al. 2005; Wang et al. 1993; Wang et al. 1995; Whistler and Be Miller 1993) .

The main aim of the present work is to isolate galactomannan gum from locally available sesbania seeds and subject it to chemical modification via carboxymethylation to improve its properties. Furthermore, the work was extended to comprise the characterization and evaluation of the prepared derivatives as thickening agents in printing cotton fabrics with reactive dyes.

EXPERIMENTAL

Materials

Substrate

Cotton poplin fabric (140g /m²) supplied by Misr/ Helwan for Spinning and Weaving Co. Cairo, Egypt was used throughout the present work.

Plant seeds

Sesbania seeds were supplied from Faculty of agriculture, Menoufia University, Egypt.

Thickening agents

Sodium alginate of high viscosity type manufactured by Ceca Kolloid Chemie, Paris, France, was kindly supplied under the commercial name Cecalginat HV/KL 600.

Reactive dye

Two different mono-functional reactive dyes were used, namely:

- Cibacron Red P-B (Reactive Red 24) Supplied by Ciba-Speciality Chemicals, Cairo. Its reactive center is monochlorotriazine.
- Remazol Brilliant Red 5B (Reactive Red 35) Supplied by Hoechst. Its reactive center is vinyl sulphone.

Chemicals

Monochloroacetic acid, sodium hydroxide, potassium iodide, copper sulphate, sodium thiosulphate, toluene, ethyl alcohol, ammonium hydroxide, acetic acid, urea, and sodium bicarbonate were of laboratory grade.

Methods

The experimental methods used for the separation of galactomannan gum from the Sesbania Sesban seeds, along with its chemical modification via carboxymethylation as well as the method used for its application as thickening agent in textile printing are briefly described in this section.

Separation of the gum from the seeds

The gum was separated from the seeds according to a previously reported method (Hossain et al. 2003).

Carboxymethylation of the gum

Carboxymethylation of the purified sesbania galactomannan gum was carried out according to the non-aqueous method described by Paddison and Sommers (El-Molla 2000; Ragheb et al. 1997).

Purification

The prepared carboxymethylated sesbania gum derivatives so obtained were purified via extraction in a Soxhlet apparatus using 80% ethyl alcohol until free from salts and finally dried in desiccators over dry calcium chloride.

Preparation of printing pastes

The printing pastes were prepared according to the following recipe:

Reactive dye	40g
Urea	100g
Thickener*	X
Sodium bicarbonate	30g
Resist salt (Lyoprint RG)^	10g
<u>Distilled water</u>	<u>Y</u>
Total	1000g

*The thickeners used were either sodium alginate (50g / kg) or carboxymethyl sesbania gum derivative (150 g / kg), according to viscosity.

^Lyoprint is a reduction inhibitor for reactive dyes; it is a sodium salt of nitrobenzene sulfonic acid.

Printing technique

All the printing pastes were applied to cotton fabric according to the conventional screen printing technique.

Fixation

All the printed goods were subjected to fixation via either:

- (1) Steaming at 102°C for 10 minutes
- (2) Thermofixation at 150°C for 5 minutes.

Washing

After fixation the printed goods were subjected to washing, which was carried out through five stages as follows:

1. Rinsing thoroughly with cold water.
2. Treatment with hot water.
3. Treatment near the boiling temperature (90-95 °C) with solution containing 2g/L Hostapal CV.ET. (non – ionic detergent)
4. Washing with hot water.
5. Rinsing with cold water.

Finally the samples were air dried and assessed for colour strength and overall fastness properties.

Analysis and Measurements

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

The D.S., expressed as carboxyl groups, was estimated iodometrically according to a method described by Finkel Stein et al. (1959).

Measurement of the rheological properties

The rheological properties of the printing pastes were measured using a Rheomat-15 at 25°C (Hebeish et al. 1989). The rheomat consists principally of two co-axial cylinders. The inner one is axially connected to a motor and can rotate with 15 speeds.

The other one is kept stationary. The apparent viscosity (η) at various rates of shear was calculated from the shearing stress (τ) and rates of shear ($\dot{\gamma}$) as follows:

$$\eta = \frac{\tau}{\dot{\gamma}} \quad (1)$$

Colour strength measurements

The colour strength of the printed samples expressed as K/S was evaluated by high reflectance technique (Judd et al. 1975). Three measurements were done for each sample, the means of which have been used for the results represented.

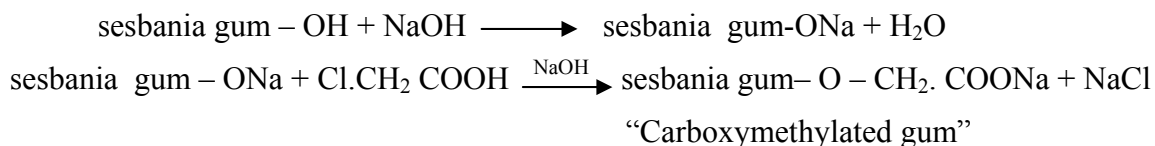
Fastness properties

The colour fastness properties to washing (AATCC Method 23, 1993) to rubbing (AATCC Method 36, 1972) and to perspiration (AATCC Method 30, 1993) were carried out according to standard methods.

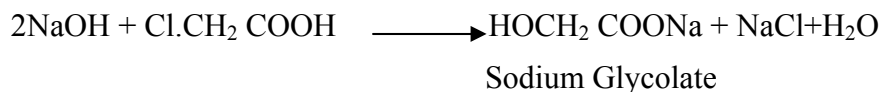
RESULTS AND DISCUSSION

Sesbania galactomannan gum (Iain 1975; Parvathy et al. 2005) can react with monochloroacetic acid, as in the case of other carbohydrates, to give a carboxymethyl derivative.

The carboxymethylation proceeds by a two-step consecutive reaction and is accompanied by an undesired side reaction. In the main reaction the sodium hydroxide reacts first with the hydroxyl groups of the gum to give alkoxide groups. The carboxymethyl groups are then formed in the reaction between the gum alkoxide and monochloroacetic acid as follows:



The side reaction takes place in both the bulk liquid and the gum phase (Green 1963), and results in the formation of sodium glycolate from monochloroacetic acid and sodium hydroxide as follows:



Galactomannan gum was isolated from Egyptian sesbania seeds according to the procedure mentioned in the Experimental section. The carboxymethylation reaction was carried out in non-aqueous 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).

Table 1. Effect of Concentration of Monochloroacetic Acid on the D.S. and Solubility of Carboxymethyl Sesbania Galactomannan Gum Derivatives

Sample	ClCH ₂ COOH g/100g gum	NaOH (50%by volume) mL/100g gum	D.S.	Solubility in	
				Water	Ethanol
1	60	120	0.35	Soluble	Insoluble
2	80	160	0.48	Soluble	Insoluble
3	100	200	0.58	Soluble	Insoluble
4	120	240	0.74	Soluble	Insoluble
5	140	280	0.95	Soluble	Insoluble

It is clear from the data (Table I) that the extent of the carboxymethylation reaction expressed as D.S. increased by increasing the monochloroacetic acid concentration. For example, when the concentration of monochloroacetic acid was 60g/100g of dry galactomannan gum, a D.S. of 0.35 was obtained. Likewise, when the amount of monochloroacetic acid was increased to 140g /100 g gums, the D.S. was also increased to 0.95.

It is worth mentioning that sesbania seed gum swells in cold water, but its pastes are not stable to storing for more than 24 hours, where fermentation takes place. The current data indicate that modification of sesbania gum via carboxymethylation converts it into a water-soluble product, and increases the stability of its pastes to microorganisms. This holds true regardless of the D.S. value.

Rheological Properties

Since sesbania seed gum and its 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 15%. The rheological properties of the pastes were monitored before and after storing of the pastes for 3 and 7 days using the Rheomat 15. The results obtained are shown in Figs. (1-3).

The rheograms of Fig. (1) indicate that all the pastes examined exhibited non-Newtonian characteristics, since the relation between shearing stress and the shear rate was not linear. Most non-Newtonian fluids fall in the category of pseudoplastics and show shear thinning, i.e. a decrease in the apparent viscosity occurs with increasing shearing rate. Furthermore, the rheograms reveal that the pastes under investigation displayed non-Newtonian pseudoplastic behaviour, since the ascending and descending rheograms are coincident. The current data reveals the homogeneity of the molecular structure of the pastes, which were amenable to rebuild themselves and retain their original state immediately after removal of the acting force, thereby exhibiting pseudoplastic behaviour.

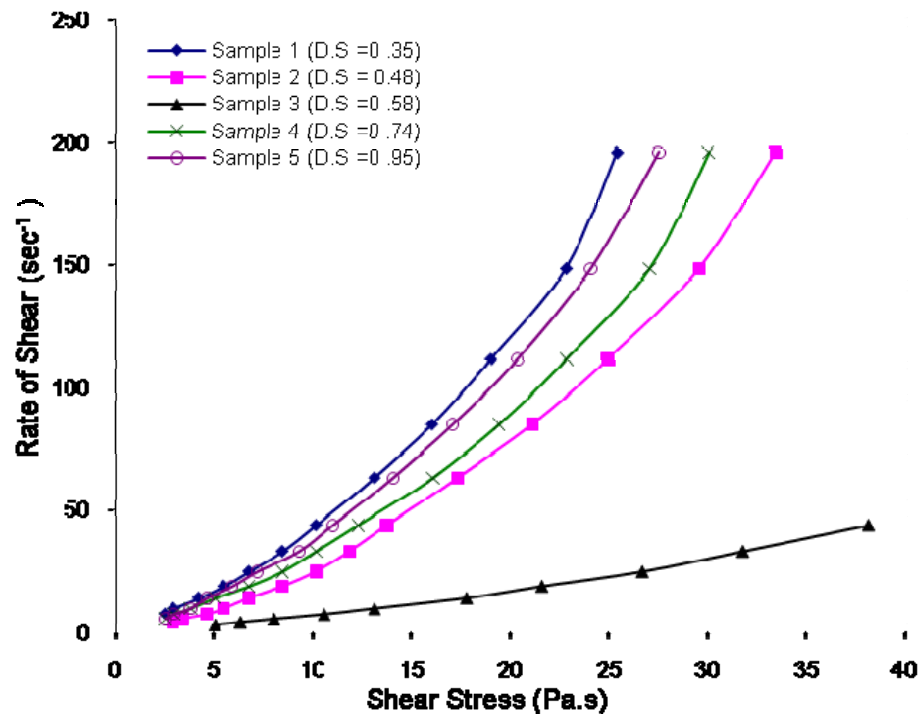


Figure 1. Rheograms of pastes of carboxymethylated sesbania galactomannan gum samples acquiring different D.S.values, freshly prepared

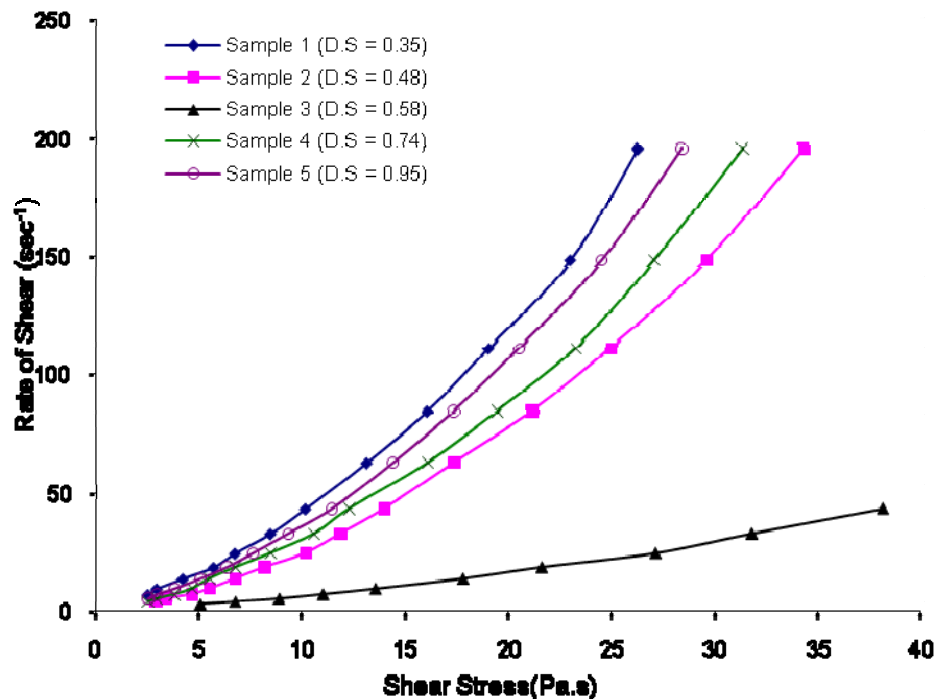


Figure 2. Rheograms of pastes of carboxymethylated sesbania galactomannan gum samples acquiring different D.S. values, after storing for 3 days.

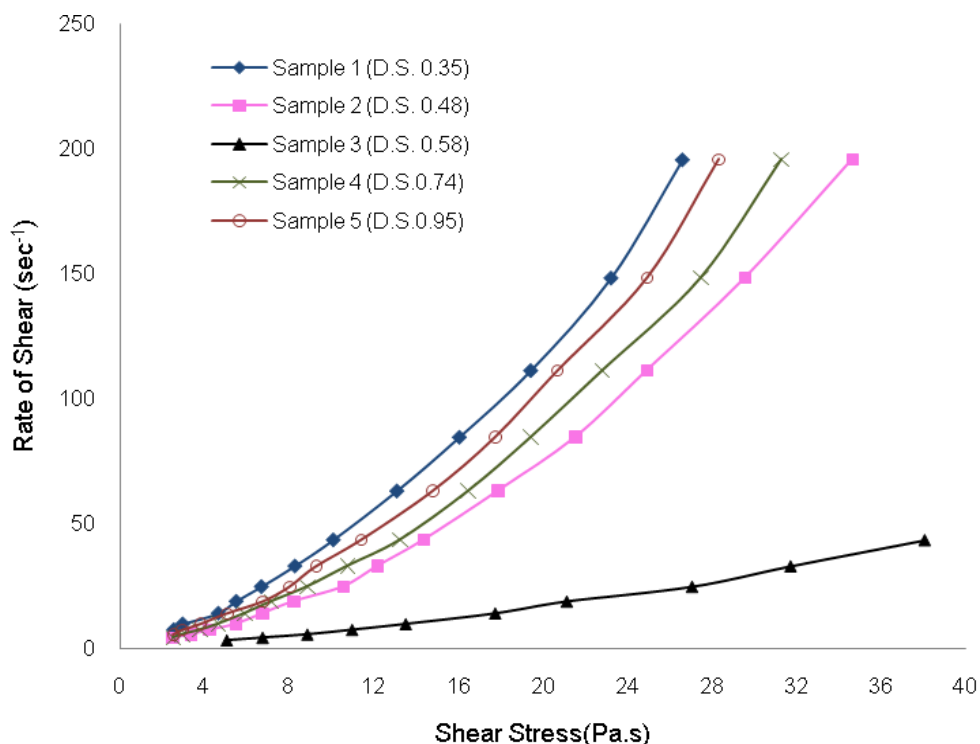


Figure 3. Rheograms of pastes of carboxymethylated sesbania galactomannan gum samples acquiring different d.s. values, after storing for 7 days

Figure 1 also shows that the location of the rheograms with respect to the shear rate axis depended on the nature of the thickening agent used, i.e. the degree of carboxymethylation expressed as D.S. As the value of D.S. increased from 0.35 to 0.58, the rheogram was located far from the shear rate axis, indicating an increase in the apparent viscosity. Further increase in the D.S. values (from 0.58 to 0.95) was accompanied by a decrease in the apparent viscosity.

Figures 2 and 3 represent the rheograms of the aforementioned pastes after storing for 3 and 7 days respectively. It is clear from the rheograms that storing had practically no effect on the rheological characteristics of these pastes, where each of these pastes continued to exhibit non-Newtonian pseudoplastic behaviour after storing.

It is clear from the data that the apparent viscosity values of these pastes nearly remained unchanged after storing for 24 hours or more. This reflects the influence of introducing carboxymethyl group on the stability of sesbania galactomannan gum polymer.

A clearer picture of the effect of the D.S. of carboxymethyl sesbania gum on the apparent viscosity of their pastes at various rates of shear is given by Fig. 4. The flow curves in the figure show that all of the pastes of carboxymethyl Sesbania galactomannan gum studied were characterized by a non-Newtonian pseudoplastic behaviour. The sample of lowest D.S. (0.35) showed the lowest degree of pseudoplasticity.

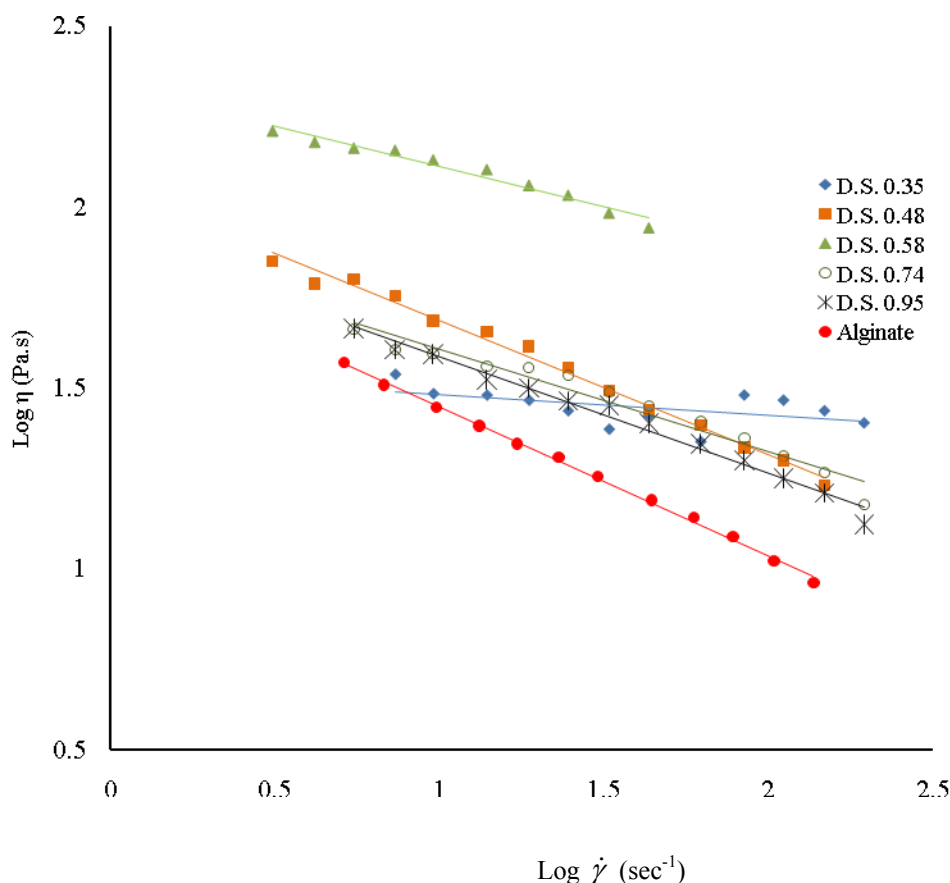


Figure 4. Log-log plots of the viscosity η , vs. shear rate $\dot{\gamma}$ of freshly prepared Carboxymethyl Sesbania pastes of different D.S. values (concentration 15%) compared to those of sodium alginate (concentration 3%)

It is clear from Fig. 4 that the apparent viscosity of pastes of sesbania derivatives increased as the D.S. increased from 0.35 to 0.58. Further increase in the D.S. for 0.74 or 0.95 was accompanied by a decrease in the apparent viscosity.

The increase in the apparent viscosity by increasing the D.S. from 0.35 to 0.58 may be due to an increase in solubility accompanying the introduction of carboxymethyl groups. However, the derivatives of relatively higher D.S. value acquire relatively lower viscosity due to a decrease in the intermolecular interaction by introducing too many carboxymethyl groups or a decrease in molecular weight due to chain degradation, which results from the influence of alkali needed for the carboxymethylation reaction, as mentioned in the experimental section.

Printing

To evaluate the suitability of carboxymethyl sesbania gum derivatives as thickeners for printing cotton fabrics with reactive dyes, it is necessary to compare prints prepared with carboxymethyl sesbania gum versus those made with sodium alginate. To achieve this goal, a series of printing pastes thickened with carboxymethyl sesbania gum derivatives of different D.S values (0.35, 0.48, 0.58, 0.74 or 0.95) containing different

classes of reactive dyes, namely Reactive Red 24 or Reactive Red 35, were prepared. For the sake of comparison, another series of printing pastes thickened with sodium alginate and containing the same dyestuffs was also prepared.

Samples of cotton fabrics were printed with these pastes via screen printing; this was done immediately after the preparation of the pastes. After drying, the printed goods were fixed either by steaming or thermofixation according to the conditions mentioned in the experimental section, washed, and air dried.

The printed goods were then assessed for colour strength, expressed as K/S, and overall fastness properties. The results are given in Table 3, relative to the use of Reactive Red 24, and in Table 4 for the use of Reactive Red 35. The data in Tables 3 and 4 indicate that the colour strength expressed as K/S depended upon: (a) the nature of thickener used, (b) the time allowed before printing, and (c) the nature of the reactive dye used.

Generally speaking, it is clear from Tables 3 and 4 that, irrespective of the nature of the thickening agent used and/or the time elapsed before printing, the K/S value was higher for samples printed using Reactive Red 24 and fixed by steaming than their corresponding samples fixed by thermofixation.

The higher K/S for the samples printed and fixed by steaming may be due to the higher amount of moisture during steaming. The moisture facilitates the mobility of the dye molecules and increases the rate of its transfer from the printed film into the fabric, thus facilitating the reaction between the reactive dye and the hydroxyl groups of cotton fabrics.

It is also clear from the data of Tables 2 and 3 that the K/S for nearly the majority of the goods printed on using carboxymethyl sesbania derivatives of relatively lower D.S values, i.e. 0.35, 0.48, or 0.58 were slightly higher than their corresponding samples printed using sodium alginate. This is probably due to the adherence of the reaction product of carboxymethyl sesbania and reactive dye on the surface of the printed cotton fabrics (the dyes used were mono-functional reactive dyes with monochlorotriazine and vinyl sulphone reactive centers, as mentioned in the experimental section). The latter cannot be removed by washing. This is substantiated by the harsh handle observed in this case. The same phenomenon was also observed on using commercial CMC or CMS samples as well as carboxymethyl guar derivative.

The variation in the K/S for the samples printed using Reactive Red 24 and their corresponding samples printed using Reactive Red 35 may be due to the difference existing between them in regards to the chemical structure, the functional groups, molecular size, configuration, substitution, etc. Such differences would certainly be reflected on the affinity of the dye for both the cotton fabrics and thickener and hence of the obtained K/S value.

Besides that, the data showed that storing of the pastes thickened with carboxymethyl sesbania gum for three or seven days had practically no effect on the K/S ratio of the printed goods.

Table 2. Properties of Cotton Fabrics Printed using Carboxymethyl Sesbania Gum of Different Degrees of Substitution as well as Sodium Alginate on Using Cibacron Red 6B for Freshly Prepared Pastes

Thickener used	Fixation Condition	K/S	Washing fastness		Rubbing fastness		Perspiration fastness				Handle
			St.	Alt.	Dry	Wet	Acidic		Alkali		
							St.	Alt.	St.	Alt.	
Sodium alginate	Steaming	6.39	4	4	4	3-4	4	3-4	4	4-5	S
	Thermo-fixation	6.08	4-5	4-5	4	3-4	4	4-5	4	4	S
Carboxymethyl gum (D.S)=0.35	Steaming	6.18	3	4	4	2-3	3-4	3	4	4	H
	Thermo-fixation	5.29	3-4	4	4	2-3	3-4	3-4	3	3	H
Carboxymethyl gum (D.S)=0.48	Steaming	6.18	3-4	4-5	4	2	3-4	3-4	4	3-4	H
	Thermo-fixation	5.70	3-4	4-5	4	3-4	4	4	4-5	4	H
Carboxymethyl gum (D.S)=0.58	Steaming	6.50	4	4-5	4	2-3	4-5	3-4	4	3-4	H
	Thermo-fixation	5.98	3-4	4	3	2-3	4-5	3-4	4	4	H
Carboxymethyl gum (D.S)=0.74	Steaming	6.08	4	4	3	3	3-4	3-4	4	4	S
	Thermo-fixation	4.93	3-4	4-5	3-4	3	4	4	3-4	3-4	S
Carboxymethyl gum (D.S)=0.95	Steaming	6.61	4	4-5	4	3-4	4-5	4	4	4	S
	Thermo-fixation	5.79	4	4-5	4	3-4	4	3-4	4	4	S

Fixation of prints was carried out via steaming, thermofixation.

St: staining; Alt: alteration; S: soft; H: harsh

On the other hand, the handle of the printed goods is one of the most important properties in the field of textile printing. The last column in Tables 3 and 4 presents the handle of the printed goods. It is clear from the data that the handle of the printed goods did not depend on the type of the reactive dye or the method of colour fixation i.e. steaming or thermofixation. Rather, it depended only on the nature of the thickening agent used. Samples of cotton fabrics that were printed using pure sodium alginate acquired a soft handle. On the other hand, samples printed using carboxymethyl sesbania acquired either a soft or harsh handle, depending on the degree of modification, i.e. the D.S. value. Samples printed using carboxymethyl sesbania of D.S. 0.74 or lower acquired a harsh handle, while the sample printed using carboxymethyl sesbania of D.S.0.95 acquired a soft handle like that printed using sodium alginate. This phenomenon held true regardless of the method of fixation and or the nature of the reactive dye used, i.e. on using either Reactive Red 24 or Reactive Red 35.

Table 3. Properties of Cotton Fabrics Printed using Carboxymethyl Sesbania Gum of Different Degrees of Substitution as well as Sodium Alginate on Using Remazol Brilliant Red 5B 15 for Freshly Prepared Pastes

Thickener used	K/S	Washing fastness		Rubbing fastness		Perspiration fastness				Handle
		St.	Alt.	Dry	Wet	Acidic		Alkali		
						St.	Alt.	St.	Alt.	
Sodium alginate	3.41	4	4	4	3-4	4	4	4	4	S
Carboxymethyl gum (D.S)=0.35	3.44	3	4	3	2-3	4	4-5	4-5	4	H
Carboxymethyl gum (D.S)=0.48	3.60	3-4	4	3	2-3	3-4	4	4	4-5	H
Carboxymethyl gum (D.S) =0.58	3.06	4	3-4	3-4	3	3-4	3-4	3-4	4	H
Carboxymethyl gum (D.S)=0.74	2.88	3-4	4	4	2-3	4	4	3-4	4	S
Carboxymethyl gum (D.S)=0.95	2.52	4	4	4	3-4	4	4	3-4	4	S

Fixation of prints was carried out via steaming.

St: staining

Alt: alteration

S: soft.

H: harsh

The soft handle, for the printed goods, obtained on using sodium alginate as a thickener, is indicative of the absence of any chemical reactions between sodium alginate and the reactive dyes used. The same holds true for carboxymethyl sesbania derivatives of D.S. 0.95. The harsh handle obtained on using carboxymethyl sesbania derivatives of D.S. 0.35, 0.58, or 0.74 may be due to the formation of carboxymethyl sesbania - reactive dye (monochlorotriazine and vinyl sulphone reactive centers) reaction product, which is difficult to remove during washing. Adherence and/or inclusion of such reaction products with fabric results in harsh handle. Furthermore, Tables 3 and 4 contain the overall fastness properties of the aforementioned printed goods, i.e. colour fastness properties to rubbing, to washing, and to perspiration.

It is clear from the data that the overall fastness properties of cotton fabrics printed using either sodium alginate or carboxymethyl sesbania derivatives, especially those printed using the derivative having a D.S. of 0.95, were nearly identical.

It can be concluded that carboxymethyl sesbania derivative of D.S. 0.95 could be used safely as a thickening agent in printing cotton fabrics with reactive dyes, where it allowed printed goods to acquire a soft handle and overall fastness properties nearly identical to the goods printed using sodium alginate; and K/S either slightly higher or lower than those obtained on using sodium alginate.

To investigate the compatibility of carboxymethylated sesbania galactomannan gum (CMG) with sodium alginate and the effect of mixing this carboxymethylated derivatives with sodium alginate on the properties of the printed cotton fabrics, the data obtained reveal that mixing carboxymethyl sesbania derivatives samples with sodium alginate improved the K/S of the printed goods without impairing the overall colour fastness properties. However carboxymethyl sesbania derivatives sodium alginate 1:1

mixture (w/w) seemed to be the best. From this investigation, it can be concluded that carboxymethylated sesbania galactomannan gum derivative of relatively higher D.S. value (0.95) could be used safely, either alone or in a mixture with sodium alginate in printing cotton fabrics, with reactive dyes without impairing the properties of the printed goods.

CONCLUSIONS

Galactomannan gum was isolated from the seeds of sesbania and subjected to chemical modification via carboxymethylation with monochloroacetic acid under the catalytic action of sodium hydroxide. The carboxymethylation was carried out in non-aqueous medium using different concentrations of the etherifying agent to obtain carboxymethylated sesbania galactomannan gum (CMG) derivatives with different degrees of substitution (D.S.).

It was found that the extent of carboxymethylation expressed as D.S. increased by increasing the concentration of the etherifying agent. The D.S. increases from 0.35 to 0.95 by increasing the amount of monochloroacetic acid from 60 to 140 g / 100g dry gum.

Native sesbania seeds gum swells in cold water, but its pastes are not stable to storing, since fermentation takes place readily. Modification of sesbania galactomannan gum via carboxymethylation converts it into a water soluble product and increases the stability of its pastes to storing

Carboxymethylated CMG gum pastes are characterized by a non-Newtonian pseudoplastic behaviour. Storing has practically no effect on the rheological characteristics of these pastes.

The results also signify that CMG samples could be used successfully as a thickening agent in printing cotton fabrics using reactive dyes. The colour strength of the printed goods depended on the nature of the reactive dye used, the type and D.S. of the thickening agents, methods of colour fixation, as well as the time elapsed before printing .

However, the overall fastness properties of the goods printed using carboxymethylated sesbania galactomannan gum derivatives were nearly comparable with their mates printed using sodium alginate, irrespective of the nature of the reactive dye used, and /or of the storing time given to the pastes before printing .

Mixing CMG samples with sodium alginate improved the K/S of the printed goods without impairing the overall colour fastness properties. However, CMG sodium alginate 1:1 mixture (w/w) seemed to be the best. Carboxymethylated sesbania galactomannan gum derivatives could be used safely, either alone or in a mixture with sodium alginate in printing cotton fabrics, with reactive dyes without impairing the properties of the printing goods.

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