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Studies on guargum–*graft*–acrylamide to explore type of graft in the polymer

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Abstract
Guargum-*graft*-acrylamide (G-g-Am) is a water soluble polymer and has good industrial use. Aqueous solution of G-g-Am binds polyvalent metal ion using some polar groups when pH is raised, which may cause lack of structural regularity in polyvalent metal ion bound polymer which is understandable from TED pattern for cupric ion bound G-g-Am. Larger grafted chain of the polymer may cause entanglement in solution and may help in cross linking because of welding of polymeric chains by polyvalent metal ion which is understandable from this rheological study and TED study using G-g-Am and cupric ion bound G-g-Am.

Keywords: Graft copolymer, structural regularity, ion binding, cross linking, entanglement

1. Introduction
Guargum is a water soluble natural polymer and has good industrial use [1]. Its structure is relatively known (fig.1). Major drawback of guargum is its poor biodegradation resistance [2]. If polyacrylamide side chains are grafted on guargum, resulting graft copolymer (G-g-Am) becomes considerable biodegradation resistant and efficient flocculent for metallic ions [2]. Characterization of guargum-*graft*-acrylamide (G-g-Am) may be done by ESCA study and by TGA and DSC analysis [3-5]. In this present investigation, largeness of grafted chain in a molecule of polymeric material is considered by TED study which helps to understand chance of cross linking of molecules by polyvalent metal ion and by study using roto viscometer which helps to understand chance of entanglement of grafted chains of polymer molecules in solution. Cupric ion bound guargum-*graft*-acrylamide (G-g-Am-Cu^{2+}) is considered in this study.

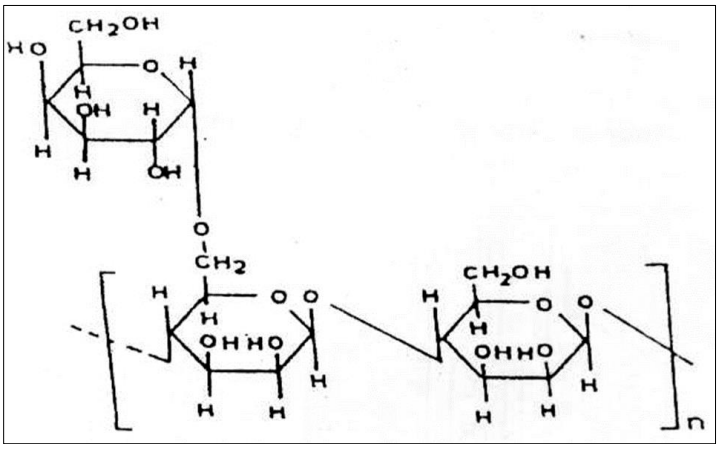

Fig 1: Structure of Guargum

2. Materials and Methods
2.1 Guargum-*graft*-acrylamide (G-g-Am)
G-g-Am has been achieved from Rheological Laboratory of Materials Science Centre of I.I.T., Kharagpur, India. From ESCA study, it was possible to understand that, it contains approximately 11 atom% nitrogen, 22 atom% oxygen and 68 atom% carbon [6]. Ceric ion initiation method is adopted for preparation of graft copolymer [2].

2.2Transmission Electron Diffraction (TED) study

TED study has been carried out using PHILIPS CM-12 transmission electron microscope and 100 KV electrons have been used [7]. Following samples are used:

Sample S1:Aqueous solution of G-g-Am.(for G-g-Am)

Sample S2:Mixture of aqueous solution of G-g-Am and $Cu(NO_3)_2$ solution(for G-g-Am-Cu^{2+}).

To prepare film for observation in TED, few drops of the sample have been placed on small thin sheet of aluminium. Another small thin sheet of aluminium has been placed over the other containing the sample. Two sheets are then pressed carefully by placing the assembly in between two glass plates. A small weight is placed over the top glass plate and the assembly is kept for 2 days for drying. Then weight and glass plates are removed and it is placed in concentrated sodium hydroxide solution taken in a watch glass. For sample S1, films are collected immediately by microscopic grids and washed immediately with dilute hydrochloric acid and then with distilled water and dried on the grid in vacuum. For sample S2, films are kept in concentrated sodium hydroxide solution for three days and collected on microscopic grids and washed with distilled water for several times and dried on the grid in vacuum. They are examined by using PHILIPS CM-12 transmission electron microscope. Images are recorded using Kodak electron image films.

2.3 Rheological study

Rheology is the science of deformation and flow under the application of shear stress. To study rheological behavior, the rotary viscometer RHEOTEST 2 has been used. There are two co-axial cylinders. Outer cylinder is stationary and holds the substance under investigation. Outer cylinder is enclosed by a temperature control vessel which permits connection to a liquid circulation thermostat to provide temperature control of the substance under investigation. Inner cylinder rotates at a constant angular speed. Substance under investigation is at the annular space of the co-axial cylinder system [7].

Shearing stress, $\tau = \alpha . z$

Where,

τ =>Shearing stress (10^{-1}Pa.)

Z =>Constant of cylinder (10^{-1} Pa. ^{1}/Skt)

α =>Graduation mark reading of instrument (Skt)

Skt =>Graduation mark

D_r gives speed gradient in the annular gap and is often termed as speed of deformation. Corrected shearing gradient is D_{rk}

$$D_{rk} => \frac{D_r f}{50}$$

Where,

ƒ=> mains frequency (cycles / sec.) and apparent viscosity,

$$\eta_{app} => \frac{\tau \times 100}{D_{rk}}$$

Rheological measurements have been carried out at approximately 30ºC and for G-g-Am-Cu^{2+}, by filling 5 milliliters of aqueous G-g-Am, 3 milliliters of cupric nitrate solution and 5 milliliters of approximately 60% sodium hydroxide solution. Mixture has been kept for 15 minutes before start of experiment. Rheological measurements for G-g-Am-Na^{+} have been carried out by filling 5 milliliters of aqueous G-g-Am and 6 milliliters of approximately60% sodium hydroxide solution. It has been found that α changes with time. So α has been measured after approximately 2 minutes of application of shearing.

3. Results and Discussion

TED pattern (fig.2) for G-g-Am indicates presence of a structural regularity in the material due to presence of regular spots. This structural regularity may come from polar-polar interaction among chains containing polar groups. This structural regularity is absent in the TED pattern for G-g-Am-Cu^{2+} (fig.3). This is due to involvement of some of the polar groups in binding Cu^{2+}[8]. Welding of polymer molecules by Cu^{2+} may cause cross linking which is in favour of largeness of grafted chain of the molecule of the polymer. Larger grafted chain of the polymer may create entanglement in solution of the polymer and may give high τ value.

Rheological study indicates (Table -1) decrease in τ value with increase in shearing gradient which is probably due to breaking of entanglement in solution which is in favour of largeness of grafted chain of the molecule of the polymer. Rheological study for G-g-Am-Cu^{2+} (Table-2) indicates higher τ value at lower shearing gradient which is also in favour of cross linking of polymeric chains by polyvalent metal ion.

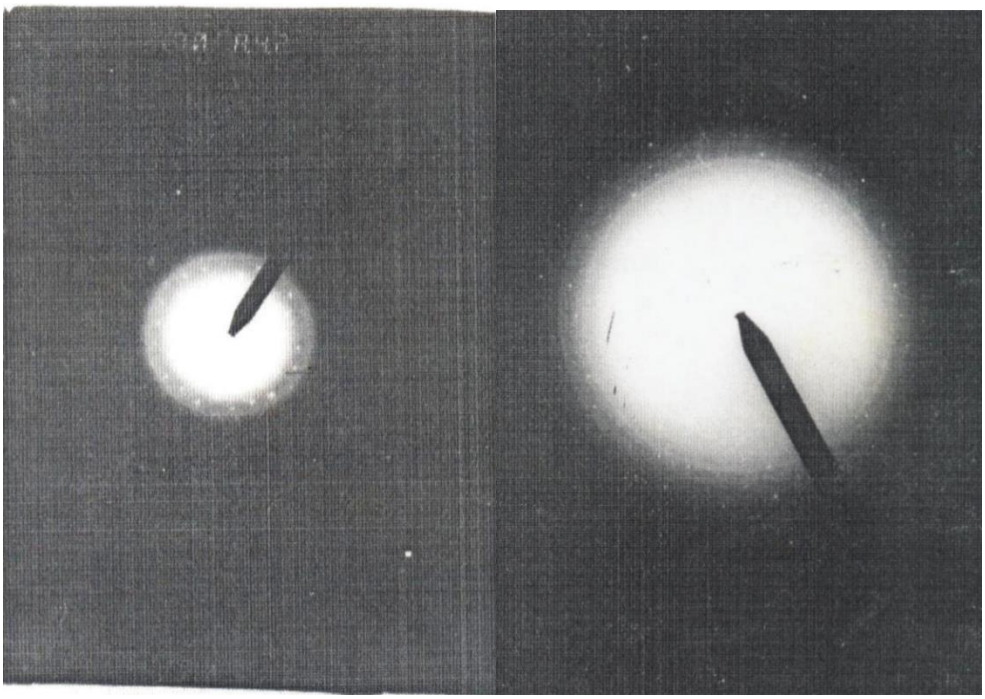

Fig 2: TED pattern for G-g-Am **Fig 3:** TED pattern for G-g-Am- Cu^{2+}

Table 1: Rheological Values for G-g-Am-Na^{+}

Rheological values for G-g-Am-Na^{+}					
Channel - I, z = 3.21					
Position	**Dr**	**f**	**Drk**	**α**	**τ**
l b	1.5	47	1.41	45	144.45
2b	2.7	47.5	2.565	12	38.52
la	3	48.5	2.91	9	28.89
3b	4.5	48.5	4.365	11	35.31
2a	5.4	413,5	5.238	11.5	36.915
4b	8,1	48	7.776	10	32.1
3a	9	48	8.64	9.5	30.495
5b	13.5	47.5	12.825	10	32.1
4a	16.2	48	15.552	10	32.1
6b	24.3	48.5	23.571	11.5	36.915
5a	27	49.5	26.462	10.5	33.705
7b	40.5	49.5	40.095	11.2	35.925
8b	72.9	49.5	72.171	20	64.2
7a	81	49	79.38	12	38.52
9b	121.5	49.5•	120.285	16	51.36
8a	145.8	49.5	144.342	17.5	56.175
10b	218.7	49.5	216.513	21	67.41
9a	243	49.5	240.57	22.5	72.225
11 b	364.5	49.5	360.855	27.5	88.275
10a	437.4	49.5	433.026	30.5	97.905
12b	656	49.5	649.44	40	128.4
1 la	729	49.5	721.71	43.2	138.672
12a	1312	49	1285.76	66.9	214.749

Table 2: Rheological Values for G-g-Am-Cu^{2+}

Channel – I, Z = 3.21					
Position	**Dr**	**f**	**Drk**	**α**	**τ**
la	3	47.5	2.85	24.5	78.645
2a	5.4	48.5	5.238	23	73.83
3a	9	48.5	8.730	23	73.83
4a	16.2	48.5	15.714	23	73.83
5a	27	48	25.920	23	73.83
6a	48.6	47	45.684	22.5	72.225
7a	81	47.5	76.950	25.5	81.855
8a	145.8	47.5	138.51	30.5	97.905
9a	243	47.5	230.85	36.7	117.807
10a	437.4	47.5	415.53	50	160.5
lla	729	47.5 •	692.550	57.5	184.575
12a	1312	47.5	1246.40	75	240.75

4. Conclusion

Guargum-*graft*- acrylamide (G-g-Am) is a water soluble graft copolymer. Its characterization based on ESCA and thermal analysis using guargum, G-g-Am and partially hydrolysed polyacrylamide has been done and already reported to understand presence of grafted sites [4, 8]. IR spectral study also helped in characterization [5].Idea of % graft has also been reported [8]. This present investigation gives idea about largeness of grafted chain in a molecule of the polymeric material. This is important from the point of view of approachability of the polymer in solution for ion binding and in flocculation.

5. Acknowledgement

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6. References

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