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Ceric ion initiated synthesis of polyacrylamide grafted oatmeal: Its application as flocculant for wastewater treatment

Srijita Bharti, Sumit Mishra*, Gautam Sen

Department of Applied Chemistry, Birla Institute of Technology, Mesra, Ranchi 835215, Jharkhand, India

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ABSTRACT

Polyacrylamide grafted oatmeal (OAT-g-PAM) was synthesized by *conventional method*. The grafting of the PAM chains on the biomaterial backbone was confirmed through intrinsic viscosity study, FTIR spectroscopy, elemental analysis (C, H, N, S and O), SEM morphology and TGA study. The intrinsic viscosity of oatmeal appreciably improved on grafting of PAM chains, thus resulting grafted product with potential application as superior viscosifier. Further, flocculation efficacy of the graft copolymer was studied in coal fine suspension, kaolin suspension, iron-ore suspension and then in municipal wastewater through 'jar test' procedure.

1. Introduction

One key factor that will continue to influence the development of nations in 21st century is the availability of fresh water. Fresh water is essential for man, agriculture as well as industry. The standard of living of a country is directly influenced by the fresh water availability as well as cost of its procurement. Water once used by man or industry, leads to the formation of wastewater. The treatment of wastewater not only results in more usage of water but also eliminates the possibility of depletion of water resources of the receiving water body. Treatment of wastewater is getting more and more priority in economical development owing to its limited availability. Often the industrial operation in a particular location critically depends on the ability to treat wastewater, thus ensuring minimum damage to ecology. Among the various technologies available to treat wastewater, flocculation is most suitable as it involves nominal instrumentation as compared to other processes.

Flocculation is a physical process of agglomeration of colloidal particles, accelerated by presence of chemical agents called flocculants. Grafted polysaccharides can form network between large number of colloidal particles, thus leading to better floc formation.

Polysaccharides are widely applied in the mineral processing industry as depressants in froth flotation, as flocculants and as selective flocculants. All of these applications involve adsorption of the polysaccharides onto minerals. Grafted polysaccharides are far more effective than simple polysaccharides due to their highly branched nature, which results in extensive network formation between colloidal particles, hence heavier floc formation. Also, for the removal of suspended solids from the coal washery effluent, wastewater, river water, flocculation aided by synthetic and semi synthetic flocculants (Mishra, Sen, Usha Rani, & Sinha, 2011; Mishra, Usha Rani, & Sen, 2012; Pal, Sen, Ghosh, & Singh, 2012; Pal, Sen, Karmakar, Mal, & Singh, 2008; Rani, Mishra, & Sen, 2013; Sen, Mishra, Usha Rani, Rani, & Prasad, 2012; Usha Rani, Mishra, Sen, & Jha, 2012) continues to be a very effective strategy.

Although, more efficient flocculation systems are available in these suspensions (Pal, Mal, & Singh, 2006; Rath & Singh, 1998; Tripathy, Bhagat, & Singh, 2001; Tripathy, Pandey, Karmakar, Bhagat, & Singh, 1999; Tripathy & Singh, 2000) but no study has been reported using oatmeal based material as flocculant.

Oatmeal (*Avena sativa*) is a hexaploid wild oat and a minor crop. Oats are generally considered "healthful", or a healthy food, being touted commercially as nutritious. The discovery of the healthy cholesterol-lowering properties has led to wider appreciation of oats as human food. A class of polysaccharides known as beta-D-glucans comprise the soluble fibre in whole oats. Oat beta-glucan is a soluble fibre. It is a viscous polysaccharide made up of units of the monosaccharide D-glucose. Oat beta-glucan is composed of mixed-linkage polysaccharides. This means the bonds between the D-glucose or D-glucopyranosyl units are either beta-1,3 linkages or beta-1,4 linkages. This type of beta-glucan is also referred to as a mixed-linkage (1→3), (1→4)-beta-D-glucan. The (1→3)-linkages

* Corresponding author. Tel.: +91 9431337645.
E-mail address: sumitmishra1@gmail.com (S. Mishra).

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break up the uniform structure of the beta-D-glucan molecule and make it soluble and flexible (Singh, De, & Belkheir, 2013).

Oats are the only cereal containing a globulin or legume-like protein, avenalin, as the major (80%) storage protein. The more typical cereal proteins, such as gluten and zein, are prolamines. The minor protein of oat is a prolamine and avenin. Oat protein is nearly equivalent in quality to soya protein. Till now, oat has not been modified and used as graft copolymers to the best of our knowledge. In this research work we aim at suitably modifying this natural resource towards its utilization as a flocculant for waste water treatment.

1.1. Plan of investigation

The study described in this paper involves the synthesis of graft chains of poly acrylamide (PAM) onto the backbone of oatmeal, thus resulting in formation of poly acrylamide grafted oatmeal (OAT-g-PAM). The synthesis has been carried out by *conventional method*, which involved ceric ammonium nitrate (CAN) in an inert atmosphere, to initiate the grafting reaction. The flocculation efficacy of the grafted product has been studied in coal fine suspension towards its application in coal washery effluent treatment, kaolin suspension, wastewater and iron ore suspension for mineral benefaction.

2. Materials and methods

2.1. Materials

Acrylamide was supplied by CDH, New Delhi, India. Ceric ammonium nitrate was supplied by E. Merck (India), Mumbai, India. Acetone was purchased from Rankem, New Delhi, India. All the chemicals were used as received; without further purification.

2.2. Synthesis of graft copolymer

2.2.1. Synthesis of graft copolymer by 'conventional method' (using ceric ammonium nitrate as a free radical initiator) [OAT-g-PAM]

Grafting reaction was carried out by ceric ion induced redox initiation method (Karmakar & Singh, 1998). The details of the synthesis and the reaction conditions are as follows.

1 g of oatmeal was dissolved in 40 ml distilled water with constant stirring and purging with nitrogen. Desired amount of acrylamide was added to above solution. They were mixed well and were transferred to the reaction vessel (250 ml borosil beaker). The oxygen free nitrogen gas was purged through the solution mixture followed by addition of catalytic amount of ceric ammonium nitrate (CAN) of desired concentration and accordingly nitrogen purging was continued. The reaction was continued for 24 h. The reaction temperature was maintained at room temperature.

Subsequently, the gel like mass left in the reaction vessel was poured into excess of acetone.

The resulting precipitate of graft copolymer was collected and was dried in hot air oven. Subsequently, it was pulverized, sieved and purified (as explained in Section 2.2.2). The reaction followed the mechanistic pathway as shown in Scheme 1(a) and (b) . In case of the polysaccharide portion of oatmeal, Scheme 1(b) is the preferred competing mechanism. Various grades were obtained by varying the reaction parameters. The percentage grafting of this conventionally synthesized OAT-g-PAM was evaluated as:

$$\%\ \text{grafting} = \frac{\text{wt. of graft copolymer} - \text{wt. of polysaccharide}}{\text{wt. of polysaccharide}} \times 100$$

The synthesis details of various grades of the graft copolymer have been shown in Table 1.

2.2.2. Purification of the graft copolymer by solvent extraction method

Any occluded polyacrylamide (PAM) formed by competing homopolymer formation reaction was removed from the graft copolymer synthesized as above, by extraction with acetone for 24 h (Kongparakul, Prasassarakich, & Rempel, 2008).

2.3. Characterization

2.3.1. Intrinsic viscosity measurement

Viscosity measurements of the polymer solutions were carried out with an Ubbelohde viscometer (capillary diameter 0.46 mm) at 25 °C. The viscosities were measured in neutral aqueous solutions. The time of flow for solutions was measured at four different concentrations (0.1%, 0.05%, 0.025% and 0.0125%). From the time of flow of polymer solutions (t) and that of the solvent (t_0, for distilled water), relative viscosity ($\eta_{rel} = t/t_0$) was obtained. Specific viscosity (η_{sp}), relative viscosity (η_{rel}) reduced viscosity (η_{red}) and inherent viscosity (η_{inh}) was mathematically calculated as:

$$\eta_{sp} = \eta_{rel} - 1$$

$$\eta_{red} = \frac{\eta_{sp}}{C}$$

$$\eta_{inh} = \ln\frac{\eta_{rel}}{C}$$

where '*C*' represents polymer concentration in g/dl.

Subsequently, the reduced viscosity (η_{red}) and the inherent viscosity (η_{inh}) were simultaneously plotted against concentration and the plots extrapolated to '*Y* axis' to get the value of intrinsic viscosity (Collins, Bares, & Billmeyer, 1973). The intrinsic viscosity thus evaluated for various grades of the graft copolymer has been reported in Table 1. The correlation of variation of intrinsic viscosity of OAT-g-PAM with percentage grafting has been graphically depicted in Supplementary Fig. 1.Supplementary data associated with this article can be found, in the online version, at http://dx.doi.org/10.1016/j.carbpol.2012.11.072.

2.3.2. Elemental analysis

The elemental analysis of oatmeal and that of OAT-g-PAM 5 (best grade of the grafted oatmeal synthesized) was undertaken with an elemental analyzer (Make – M/s Elementar, Germany; Model – Vario EL III). The estimation of five elements, i.e. carbon, hydrogen, nitrogen, oxygen and sulphur was undertaken. The results have been summarized in Table 2.

2.3.3. FTIR spectroscopy

The FTIR spectrums of oatmeal (Fig. 1(a)) and of OAT-g-PAM 5 (Fig. 1(b)) were recorded in solid state, by KBr pellet method using a FTIR spectrophotometer (Model IR-Prestige 21, Shimadzu Corporation, Japan) between 400 and 4000 cm^{-1}.

The important FTIR peaks of oatmeal and that of OAT-g-PAM 5 have been reported in Supplementary Table 1.Supplementary data associated with this article can be found, in the online version, at http://dx.doi.org/10.1016/j.carbpol.2012.11.072.

2.3.4. Scanning electron microscopy

Surface morphology of oatmeal (Fig. 2(a) and (c)) and OAT-g-PAM 5 (Fig. 2(b) and (d)) was analysed in scanning electron microscopy (SEM) in powdered form (Model: JSM-6390LV, Jeol, Japan).

Table 1
Synthesis details of various grades of OAT-g-PAM.

Polymer grade	Wt. of oatmeal (g)	Wt. of acrylamide (g)	Wt. of CAN (g)	% grafting	Intrinsic viscosity (dl/g)
OAT-g-PAM 1	1	10	0.1	917%	1.77
OAT-g-PAM 2	1	10	0.2	1157%	2.31
OAT-g-PAM 3	1	10	0.3	1130%	2.29
OAT-g-PAM 4	1	5	0.2	577%	1.14
OAT-g-PAM 5	1	15	0.2	1749%	5.78
OAT-g-PAM 6	1	20	0.2	1734%	3.58
Oatmeal (OAT)	–				0.7

$\%\ \text{grafting} = \frac{\text{wt. of graft copolymer} - \text{wt. of polysaccharide}}{\text{wt. of polysaccharide}} \times 100.$

2.3.5. TGA studies

The thermo gravimetric analysis (TGA) of oatmeal and that of the OAT-g-PAM 5 was carried out with TGA instrument (Model: DTG-60; Shimzadu, Japan). The study was performed in an inert atmosphere (nitrogen) from 25 °C to 800 °C. The heating rate was uniform in all cases at 5°/min. The concerned TGA curves have shown in Fig. 3(a) and (b).

2.4. Flocculation study

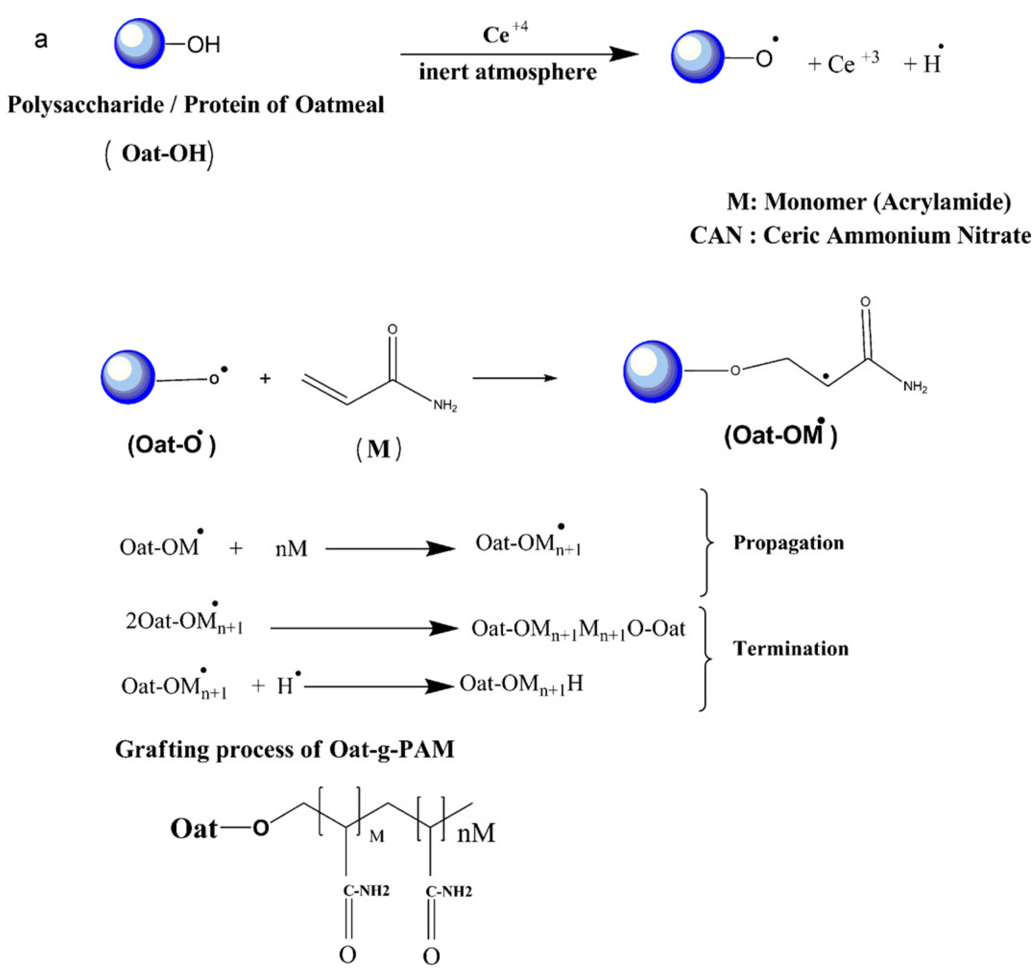
2.4.1. Flocculation study in coal fine suspension

Flocculation studies were carried out in 1% coal fine suspension, by standard 'jar test' procedure.

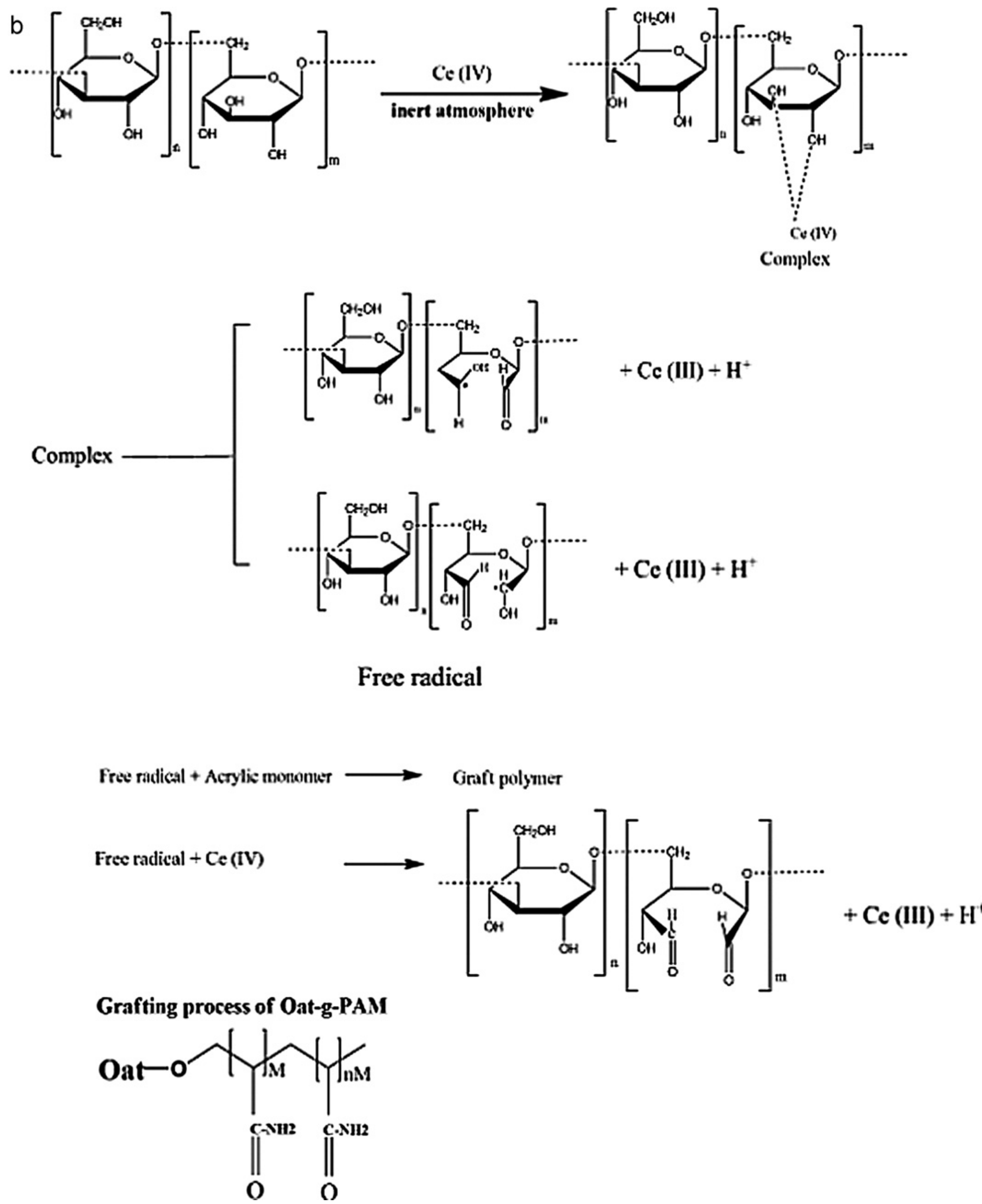
All flocculation experiments were carried out in jar test apparatus (Make: Simeco, Kolkata, India). The test protocol involved taking a measured quantity (800 ml) of the 1% coal fine suspension in 1 l borosil® beakers. Calculated amount of the flocculant (oatmeal or various grades of OAT-g-PAM) was added in concentrated solution form (except in case of blank, where no flocculant was added) to achieve the desired dosage (ranging from 0 ppm to 1.5 ppm). The solutions were identically stirred (in jar test apparatus), at 150 r.p.m. for 30 s, 60 r.p.m. for 5 min, followed by 5 min of settling time. Afterwards, supernatant liquid was collected from each jar and optical density was measured in a calibrated spectrophotometer (DR/2400, Hach®) at λ_{max} 520 nm. The flocculation efficacy thus studied for oatmeal, and various grades of OAT-g-PAM have been graphically compared in Fig. 4(a).

2.4.2. Flocculation study in kaolin suspension and iron ore suspension

Flocculation tests in 0.25% kaolin suspensions and 1% iron ore suspensions were carried out through a similar procedure as above,



Scheme 1. (a and b) Schematic representation of mechanism for 'conventional' synthesis of OAT-g-PAM.



Scheme 1. (*continued*).

Table 2
Elemental analysis of oatmeal and OAT-g-PAM 5 (best grade).

Polymer grade	%C	%H	%N	%O	%S
PAM	50.69	7.09	19.71	22.51	0.00
OAT-g-PAM 5	44.83	7.705	16.19	31.01	0.278
Oatmeal	43.58	8.47	2.192	45.47	0.297

however in these cases, turbidity of supernatant collected was measured in nephelo-turbidity meter (Digital Nephelo-Turbidity Meter 132, Systronics, India). The flocculation efficacy thus studied for oatmeal, and various grades of OAT-g-PAM have been graphically compared in Fig. 4(b) (for kaolin suspension) and Fig. 4(c) (for iron ore suspension).

2.4.3. Flocculation study in wastewater

Flocculation efficacy of the best grade of the grafted oatmeal (OAT-g-PAM 5) in municipal wastewater, at the optimized dosage as determined by earlier flocculation experiments, was studied and compared to the efficacy in case of the raw material (oatmeal). The flocculation efficacy was evaluated by assessment of pollutant load in the collected supernatant. The experiment was done in three sets.

SET 1: Wastewater without flocculant
SET 2: Wastewater with 1.25 ppm of oatmeal
SET 3: Wastewater with 1.25 ppm of OAT-g-PAM 5

The water quality of these supernatants was analysed by standard procedures (Greenberg, 1999), as reported in Table 3.

3. Results and discussion

3.1. Synthesis of OAT-g-PAM by conventional method

OAT-g-PAM has been synthesized by conventional method. The term *conventional method* has been coined by us in our earlier study.

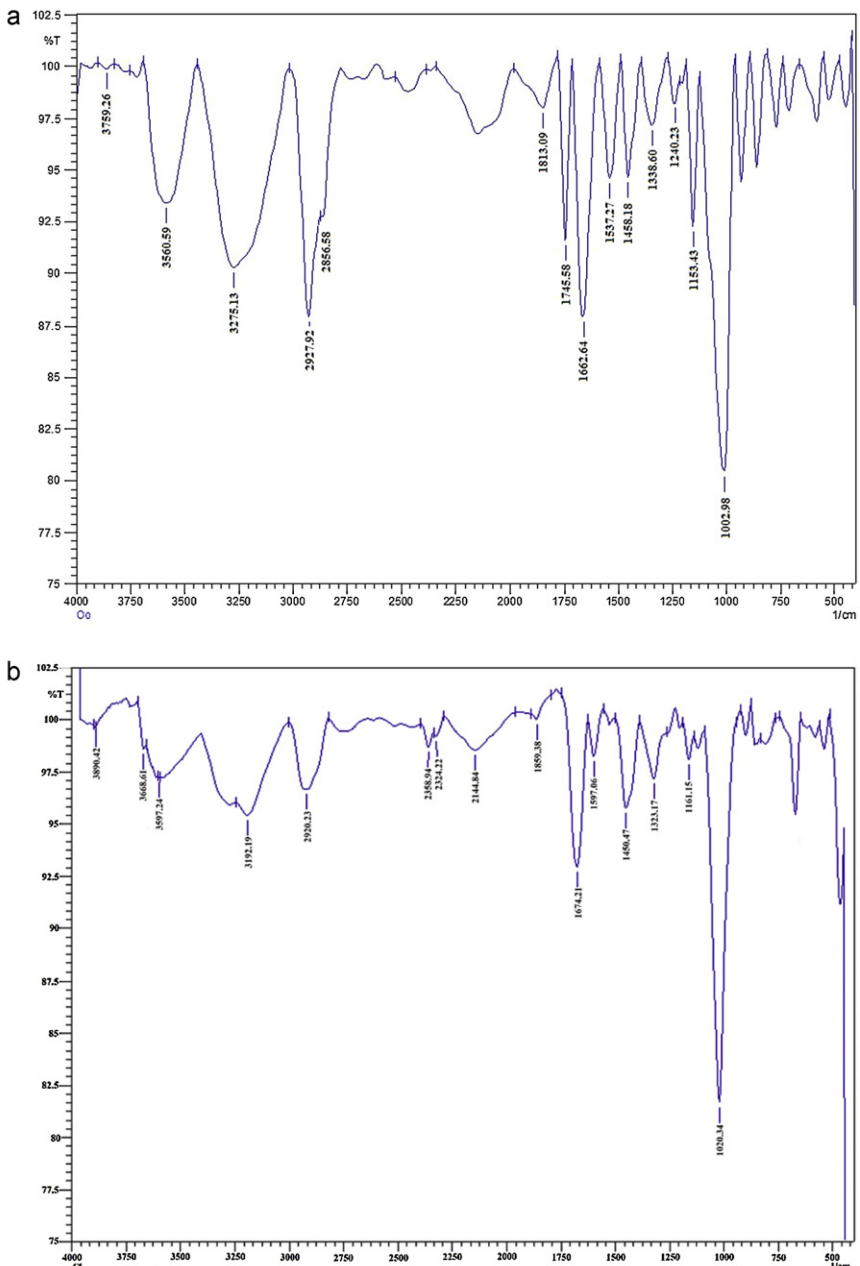


Fig. 1. FTIR spectrum of (a) oatmeal and (b) OAT-g-PAM 5.

Table 3
Comparative study performance of best grade of OAT-g-PAM (i.e. grade 5) for the treatment of municipal sewage wastewater.

Parameter	Supernatant liquid SET 1 [i.e. wastewater without flocculant]	Supernatant liquid SET 2 [i.e. wastewater with 1.25 ppm of oatmeal]	Supernatant liquid SET 3 [i.e. wastewater with 1.25 ppm of OAT-g-PAM 5]
Turbidity (NTU)	63.13	57.51	28.02
Optical density (at λ_{max} = 520 nm)	0.206	0.168	0.104
Na^+ (ppm)	459	217	108.8
Ca^{2+} (ppm)	289	156	69.9
TS (ppm)	500	300	100
TDS (ppm)	350	200	50
TSS (ppm)	150	100	50
BOD (ppm)	2.5	2	1.5
COD (ppm)	6.15	3	0.94
Chromium VI (ppm)	4.08	3.16	1.8
Fe^{3+} (ppm)	8.24	5.44	1.92

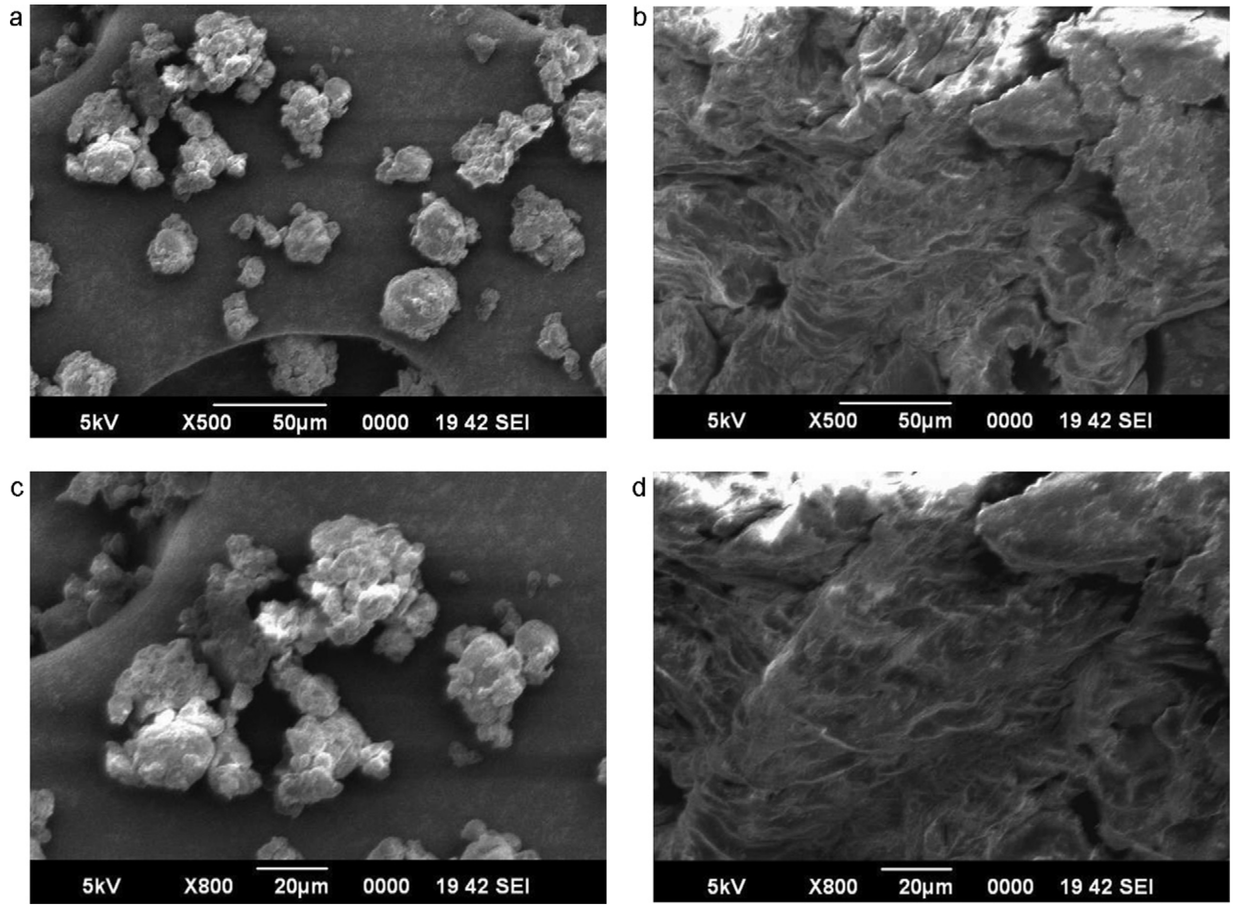


Fig. 2. SEM morphology of (a) oatmeal (500× magnification), (b) OAT-g-PAM 5 (500× magnification), (c) oatmeal (800× magnification) and (d) OAT-g-PAM 5 (800× magnification).

It refers to a process of graft copolymer synthesis, which is based on free radical mechanism using chemical free radical initiator (ceric ammonium nitrate) to generate free radical sites on the oatmeal backbone.

Various grades of the graft copolymer were synthesized by varying the ceric ammonium nitrate (CAN) and acrylamide (monomer) concentration. The synthesis details have been tabulated in Table 1. The optimized grade has been determined through its higher percentage grafting and intrinsic viscosity (which is proportional to molecular weight). The approach of synthesis involved optimization with respect to CAN, keeping the acrylamide concentration constant (i.e. OAT-g-PAM 1, OAT-g-PAM 2, and OAT-g-PAM 3); followed by optimization with respect to acrylamide, keeping the CAN concentration as optimized before (i.e. OAT-g-PAM 2, OAT-g-PAM 4, OAT-g-PAM 5, and OAT-g-PAM 6). From Table 1, it is obvious that the grafting is optimized at acrylamide concentration of 10 g and CAN concentration of 0.2 g in the reaction mixture (~40 ml).

CAN is an electron deficient molecule. It draws electrons from alcoholic oxygen in oatmeal to form a new bond i.e. Ce—O. This bond being more polar (than O—H bond), breaks easily to form free radical site on the backbone of oatmeal, from where the graft chains grow by the addition of acrylic monomer (i.e. acrylamide). Mechanism of conventional synthesis of graft copolymer has been described in details in earlier studies (Mino & Kaizerman, 1958; Schwab, Stannett, Rakowitz, & Magrane, 1962).

The proposed mechanism of conventional grafting has been depicted in Scheme 1(a) and (b).

3.2. Characterization

3.2.1. Estimation and interpretation of intrinsic viscosity

The intrinsic viscosity was evaluated for oatmeal and for all the synthesized grades of OAT-g-PAM, as shown in Table 1.

Intrinsic viscosity is the hydrodynamic volume of the macromolecule in the solvent (water in this case) solution. As evident from Table 1, intrinsic viscosities of all the grades of OAT-g-PAM are greater than that of oatmeal and intrinsic viscosity of the best grade of OAT-g-PAM (i.e. OAT-g-PAM 5) is greatest than that of all the grades of OAT-g-PAM. This higher hydrodynamic volume of OAT-g-PAM than oatmeal is due to the increase in hydrodynamic volume resulting from grafting of the PAM chains on the main polymer backbone (oatmeal). The grafted PAM chains increase hydrodynamic volume by two ways:

(1) By uncoiling of the polymeric chains of oatmeal proteins and polysaccharides through steric hindrance to intra molecular bonding.
(2) By contributing their own hydrodynamic volume.

Further, the increase in intrinsic viscosity due to grafting is in good agreement with Mark–Houwink–Sakurada relationship (intrinsic viscosity $\eta = KM^{\alpha}$, where K and α are constants, both related to stiffness of the polymer chains), which explains the increase in intrinsic viscosity as a result of increase in molecular weight (M) due to the grafted PAM chains.

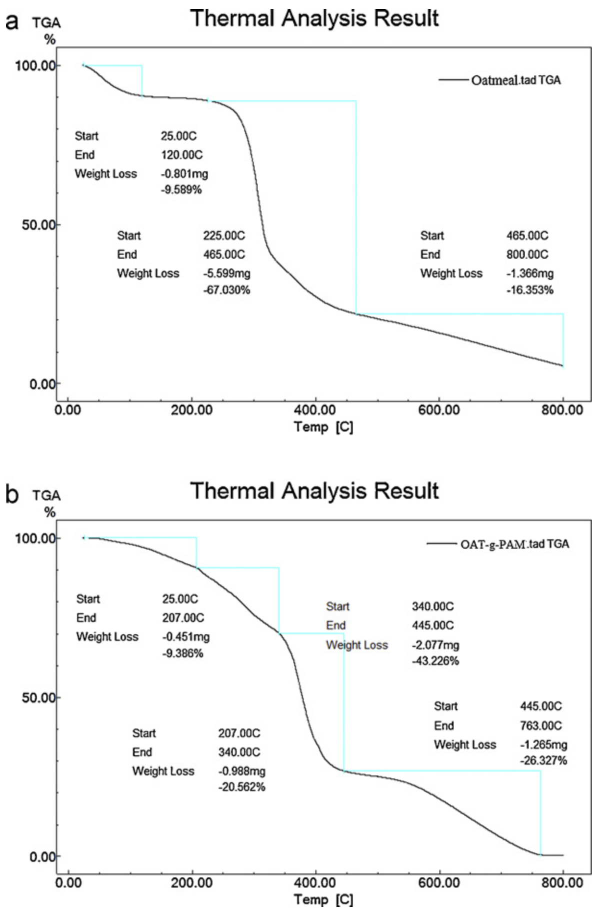


Fig. 3. TGA of (a) oatmeal and (b) OAT-g-PAM 5.

The correlation between percentage grafting (%G) and intrinsic viscosity (η) of various grades of OAT-g-PAM has been plotted in Supplementary Fig. 1.

3.2.2. Elemental analysis

The results of elemental analysis for oatmeal (OATMEAL), poly acrylamide (PAM) and that of the best grade of poly acrylamide grafted oatmeal (OAT-g-PAM 5) are given in Table 2. As evident from the table, the grafted product has an elemental composition that is intermediate of its constituents (OATMEAL and PAM).

3.2.3. FTIR spectroscopy

As evident from Fig. 1(a), oatmeal has two O—H stretching peaks are at 3560.59 cm^{-1} and 3275.13 cm^{-1} respectively, which is sharper, C=O stretching peak at 1662.64 cm^{-1} and 1745.58 cm^{-1} respectively, C—N stretching peaks are evident at 1338.60 cm^{-1} and 1240.23 cm^{-1} respectively, C—H stretching peak at 2927.92 cm^{-1}, and N—H stretching peak is evident at 3759.26 cm^{-1} due to the presence of protein chain in oatmeal.

From Fig. 1(b) it is clear that all the above peaks present in oatmeal are also present in OAT-g-PAM 5. The peak showing O—H stretching at 3560.59 cm^{-1} and 3275.13 cm^{-1} which was sharper in case of oatmeal has become subdued in OAT-g-PAM 5; proving primary O—H bond as the attack site of grafting.

The important FTIR peaks of oatmeal and that of OAT-g-PAM 5 have been reported in Supplementary Table 1.

3.2.4. Scanning electron microscopy (SEM) analysis

It is evident from the SEM micrographs of oatmeal (Fig. 2(a) and (c)) and that of the best grade of OAT-g-PAM 5 (Fig. 2(b) and (d))

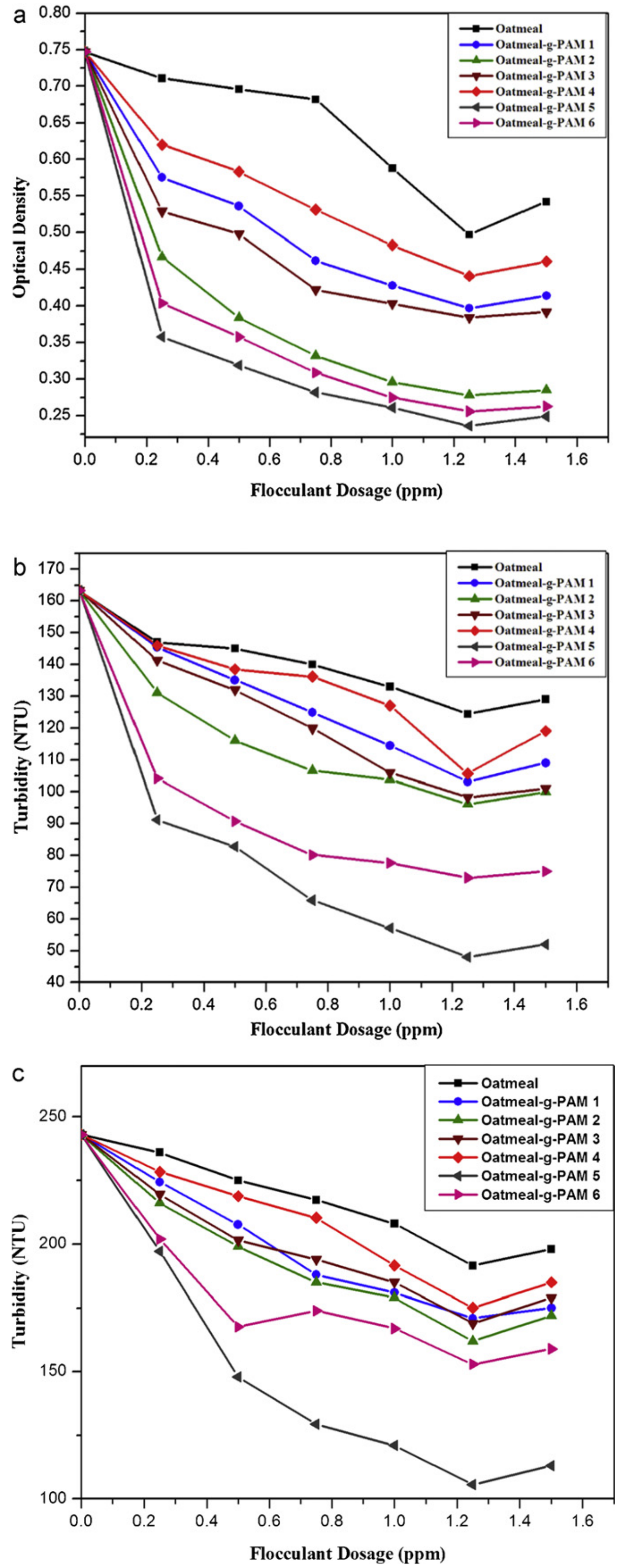


Fig. 4. (a) Flocculation profile in coal fine suspension, (b) flocculation profile in kaolin suspension and (c) flocculation profile in iron-ore suspension (turbidity).

that profound morphological change, in the form of transition from granular structure to fibrillar structure, have taken place because of grafting of PAM chains on the oatmeal backbone.

3.2.5. Thermal gravimetric analysis

The TGA curves of oatmeal (Fig. 3(a)) essentially involved three distinct zones of weight loss. The initial weight loss is at 25–120 °C. This is due to the traces of moisture present. The second zone (225–465 °C) and the third zone of weight loss (465–800 °C) were due to degradation of the oatmeal backbone.

In case of OAT-g-PAM 5 (Fig. 3(b)), a fourth zone of weight loss (445–763 °C) overlaps the third zone of weight loss of oatmeal backbone. This fourth zone of weight loss is due to PAM chains grafted on oatmeal moiety.

3.3. Flocculation study in coal fine, kaolin, iron-ore suspension and waste water

The flocculation study in 1% coal fine suspension, 0.25 kaolin suspension and 1% iron-ore suspension in jar test apparatus, for dosage varying between 0 ppm (control) and 1.5 ppm has been shown in Fig. 4(a)–(c).

All the grades of grafted oatmeal have shown better flocculation efficacy than the starting material (oatmeal). This is due to their higher hydrodynamic volume (i.e. intrinsic viscosity) as evidenced in Table 1. The higher hydrodynamic volume of the macromolecule leads to higher flocculation efficacy (Brostow, Pal, & Singh, 2007). As evident, higher the percentage grafting, higher is the intrinsic viscosity. Higher the intrinsic viscosity, higher is the flocculation efficacy.

Evidently, among the various grades of OAT-g-PAM, the optimized grade (OAT-g-PAM 5) showed maximum flocculation efficacy due to its highest hydrodynamic volume (intrinsic viscosity).

The much higher flocculation efficacy of the grafted product than the original polymers conforms to Singh's easy approachability model (Singh, 1995; Singh et al., 2000) and Brostow, Singh and Pal's model of flocculation.

For all the polymers studied, there is an optimal dosage at which the flocculation efficacy is maximum, beyond which the flocculation decreases. This behaviour of the flocculation curve finely conforms with the *bridging mechanism* (Ruehrwein & Ward, 1952).

The optimal dosage of OAT-g-PAM 5 as flocculant, in 1% coal fine suspension, 0.25% kaolin suspension, and 1% iron-ore suspension is at 1.25 ppm. This small dosage of OAT-g-PAM 5 indicates the small amount of the chemical sufficient to effect flocculation. In more practical terms, the dosage translates to the fact that one kilogram of OAT-g-PAM 5 is sufficient for the treatment of 800,000 l of coal washery effluent or wastewater.

OAT-g-PAM 5 was found to be able to considerably reduce the pollutant load of wastewater, compared to oatmeal, as evidenced by analysis of supernatants drawn from the 'jar test' procedure at optimized flocculant dosage (1.25 ppm). A comparative study (Table 3) of water quality of supernatants drawn from 'jar test' procedure in case of wastewater alone (SET 1), wastewater with 1.25 ppm of oatmeal as flocculant (SET 2) and wastewater with 1.25 ppm of OAT-g-PAM 5 as flocculant (SET 3) have shown much better water quality in case of SET 3. Drastic reduction in metal content (chromium VI and iron III) and appreciable reduction in organic load (in terms of BOD and COD) were observed. Thus, OAT-g-PAM is much better flocculant than the starting material (oatmeal). Drastic reduction in cations like Na^+ and Ca^{2+} content was also observed.

4. Conclusion

Polyacrylamide grafted oatmeal (OAT-g-PAM) has been synthesized by *conventional* technique. The synthesized grades of this graft copolymer were characterized through various physicochemical techniques. The increase in intrinsic viscosity due to grafting of PAM chains opens the prospect of application of the grafted product as a superior viscosifier. Further, the flocculation efficacy of the graft copolymer was studied through standard 'jar test' procedure in coal fine suspension, kaolin suspension, iron ore suspension and finally applied for wastewater treatment. OAT-g-PAM 5 (grade 5 i.e. best grade) with highest hydrodynamic volume (i.e. intrinsic viscosity) showed the maximum flocculation efficacy, as predicted by '*Brostow, Pal and Singh model of flocculation*'. The higher flocculation efficacy of the grafted product than the original polymer is also in accordance with '*Singh's easy approachability model*'. The high flocculation efficacy of OAT-g-PAM 5 in coal fine suspension, kaolin suspension, iron ore suspension and wastewater treatment makes it a superior flocculant than the starting material (oatmeal) for the treatment of coal washery effluents, kaolin suspension, iron-ore beneficiation and wastewater treatment. The optimized dosage of flocculation for the best grade of OAT-g-PAM (i.e. grade 5) has been found to be 1.25 ppm.

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