



Synthesis, characterization and application of novel cationic and amphoteric flocculants based on amylopectin

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ABSTRACT

The synthesis of novel cationic flocculants based on amylopectin (AP), acrylamide (AM) and (3-acrylamidopropyl) trimethylammonium chloride (ATMAC) were done by free radical polymerization using ammonium persulphate (APS) as an initiator. Three different grades of novel cationic flocculants (AP-g-C 1 to AP-g-C 3) were synthesized by varying the proportion of acrylamide and (3-acrylamidopropyl) trimethylammonium chloride monomers. Through the hydrolysis of these flocculants, in presence of NaOH, three different grades of amphoteric polymers (AP-AT-C 1 to AP-AT-C 3) were synthesized. The synthesized polymers were characterized by various methods, namely, infrared spectroscopy, NMR spectroscopy, thermal analysis, viscosity measurement, scanning electron microscopy and X-ray diffractometry. The flocculation performance of AP-g-C and AP-AT-C were studied in kaolin suspension using jar test and settling test methods at neutral pH. Dye (Methylene blue) removal tests were performed using polymer beads and analysed by UV–vis spectroscopy.

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1. Introduction

Waste water generated from household, industrial and agricultural usage is responsible for introduction of several types of chemicals in various water resources. This leads to alteration of the water cycle causing a global alarm related to the ultimate effect on wild life and human health (Sarkar, Mandre, Panda, & Pal, 2013; Deblonde, Cossu-Leguille, & Hartemann, 2011; Schwarzenbach et al., 2006). Hence proper treatment of waste water is essential to ensure safety of environment and human health. To save our environment and aquatic life from rampant disposal of wastewater, it is necessary to treat the wastewater and go for its possible recycling (Garric & Ferrari, 2005; Pham & Proulx, 1997). Untreated wastewater not only enhances dearth of potable water but also increases suspended solids, dissolved solids particles, turbidity and colouration (dye) of water. This in turn, decelerates photosynthesis, conquers the development of aquatic biota, obstructs sunlight incursion and depletes the dissolved oxygen (Verma, Dash, & Bhunia, 2012; Zahrim, Tizaoui, & Hilal, 2011). Discharges

of wastewater from respective industries carry fine particles of 0.001–10 μm size that takes prolonged time to settle down by gravity alone because of fineness of particles and surface charge (Brostow, Pal, & Singh, 2007). The removal of these colloidal particles and dyes from various wastewaters has become a challenging problem to the wastewater treatment engineers (Beltran-Heredia, Sanchez-Martin, Delgado-Regalado, & Jurado-Bustos, 2009).

Various technologies have been applied for the treatment of industrial effluents and other wastewaters, out of which flocculation is an ecofriendly and economical process. It can be considered as one of the most effective techniques, in particular for primary removal of suspended solid particles, dyes, toxic heavy metals, etc. (Crini, 2006; Renault, Sancey, Badot, & Crini, 2009; Sharma, Dhuldhoya, & Merchant, 2006). Polymeric flocculants have been observed to be efficient at lower doses, have controlled biodegradability, and possess shear resistance. Polyacrylamide and cationic polymer based flocculants used in wastewater and industrial effluent treatment are highly efficient at low doses. But they are fragile and expensive. On the other hand, polysaccharides are cheap, biodegradable, shear stable but less effective as flocculants (Singh, Nayak, Biswal, Tripathy, & Banik, 2003; Singh, Pal, Krishnamoorthy, Adhikary, & Ali, 2009; Krishnamoorthi & Singh, 2003; Krishnamoorthi, Mal, & Singh, 2007). They are required at high doses in flocculation process.

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Dye removal is one of the major challenges of wastewater treatment. Mostly reactive dyes are removed from wastewater using flocculation process by adding some adsorbent like kaolin and bentonite (Guibal & Roussy, 2007; Mishra & Bajpai, 2006; Shi, Ju, & Zhang, 2012; Sarkar, Pal, Ghorai, Mandre, & Pal, 2014; Singh, Pal, & Ali, 2014). This unnecessarily enhances the water contamination.

In the present work, cationic and amphoteric flocculants have been developed and their flocculation performances have been tested in kaolin suspension at neutral pH. The cationic flocculants have been synthesized by optimally grafting the branches of polyacrylamide and poly-(3-acrylamidopropyl) trimethyl-ammonium chloride on purified amylopectin backbones. Thereafter the graft copolymers have been hydrolysed to obtain amphoteric flocculants. The present investigation also targets development of polymer beads by using grafted cationic and amphoteric polymers based on amylopectin with sodium alginate. The polymer beads have been used to remove reactive dye by absorption mechanism.

2. Experimental

2.1. Materials

Acrylamide, calcium chloride and sodium hydroxide (Merck, Mumbai, India), amylopectin and methylene blue (Himedia, Mumbai, India), ammonium persulphate, acetone, kaolin and sodium alginate (S.D. Fine Chemicals, Mumbai, India) were used as

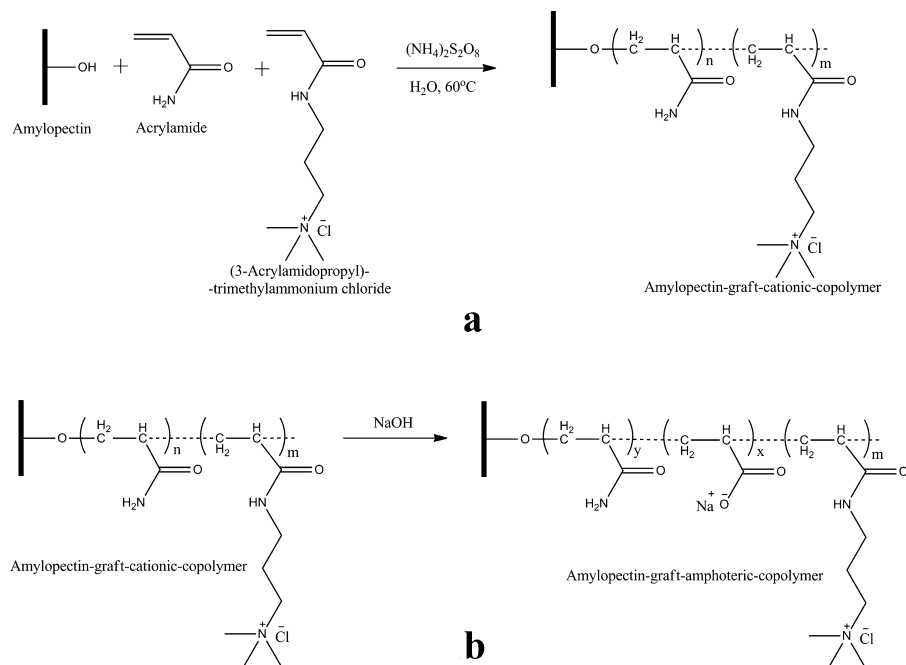
procured. (3-acrylamidopropyl) trimethyl-ammonium chloride (75%) (Sigma Aldrich, India) was purified by basic alumina column prior to use.

2.2. Synthesis of cationic polymers

Cationic polymers were synthesized by free radical solution polymerization technique, based on amylopectin, polyacrylamide (PAM), (3-acrylamidopropyl) trimethyl-ammonium chloride (PAT-MAC) in presence of ammonium persulphate as an initiator. The polymerization reaction was carried out at 60 °C in inert (N₂) atmosphere and terminated by adding 2 ml saturated hydroquinone solution. Synthesis procedure and purification of product were followed as in literature (Adhikary, Tiwari, & Singh, 2007). The reaction steps are shown in Scheme 1(a). Three different grades of cationic polymers (AP-g-C 1 to AP-g-C 3) were synthesized by varying the equivalents of monomers keeping the other reaction feeds constant. The synthesis details are summarized in Table 1.

2.3. Synthesis of amphoteric flocculants

One gram cationic polymer was dissolved in 40 ml of water and subsequently 5 ml of 0.1 N NaOH solution was added and stirred for 3 h at 50 °C. The reaction mixture was precipitated and washed twice by acetone. It was then dried for 24 h in vacuum at 15 mm of Hg. Afterwards the hydrolysed polymer was pulverized and used



Scheme 1. The polymerization and hydrolysis reaction scheme are shown in (a) and (b), respectively.

Table 1
Synthesis details of AP-g-C and AP-AT-C.

Serial no.	Factors	Cationic polymers			Amphoteric polymers		
		AP-g-C 1	AP-g-C 2	AP-g-C 3	AP-AT-C 1	AP-AT-C 2	AP-AT-C 3
1	AP (mmol)	0.5	0.5	0.5	–	–	–
2	AM (mmol)	50	50	50	–	–	–
3	ATMAC (mmol)	10	15	20	–	–	–
4	APS (mmol)	0.5	0.5	0.5	–	–	–
5	% Yield	96.4	92.2	90.1	93.8	94.7	95.2
6	NaOH (1 N)	–	–	–	5 ml	5 ml	5 ml
7	Intrinsic Viscosity (dl/g)	3.393	8.506	15.97	5.033	22.298	29.40
8	% Grafting Efficiency	96.11	91.62	89.37	–	–	–

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