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The extent of reaction traced by determination of N2 content for synthesized cationic biodegradable polymer flocculant

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Abstract:	Biodegradable "green" and safe substitutes to minimize environmental and health risks are drawing extensive attention for research interest. This research aims to create a water-soluble cationic starch-grafted biodegradable polymer that can be used as a flocculant. As polymers are strong molecules that carry charges, they are effective flocculants. Saturated polymer chains do not change the pH on adding to wastewater. The reactive groups of the starch, i.e., hydroxyls, form a complex with cations present in the reagent. According to the proposed strategy outlined in the research, the mechanism of free radical graft copolymerization of Ethyl monomers onto starch should proceed as expected. In presence of molecular Nitrogen, H+ may be replaced by reactive group R, i.e., CH2 CH2 N (Et3) in a highly alkaline medium. During the work, it was observed that starch-based flocculant exhibits a high degree of cationic charge. Developed cationic flocculant performs well in a liquid dispersion form, is fast-acting and effective, and is suitable for treating industrial wastewater. It combines low sludge formation with cost-effectiveness. The paper will discuss important practical considerations to increase the %N2 by changing the reaction parameters of the polymer produced and its effect on flocculation efficiency.

The extent of reaction traced by determination of N_2 content for synthesised cationic biodegradable polymer flocculant

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ABSTRACT

Biodegradable "green" and safe substitutes to minimize environmental and health risks are drawing extensive attention for research interest. This research aims to create a water-soluble cationic starch-grafted biodegradable polymer that can be used as a flocculant. As polymers are strong molecules that carry charges, they are effective flocculants. Saturated polymer chains do not change the pH on adding to wastewater. The reactive groups of the starch, i.e., hydroxyls, form a complex with cations present in the reagent. According to the proposed strategy outlined in the research, the mechanism of free radical graft copolymerization of Ethyl monomers onto starch should proceed as expected. In presence of molecular Nitrogen, H+ may be replaced by reactive group R, i.e., CH_2 CH_2 N (Et_3) in a highly alkaline medium. During the work, it was observed that starch-based flocculant exhibits a high degree of cationic charge. Developed cationic flocculant performs well in a liquid dispersion form, is fast-acting and effective, and is suitable for treating industrial wastewater. It combines low sludge formation with cost-effectiveness. The paper will discuss important practical considerations to increase the %N_2 by changing the reaction parameters of the polymer produced and its effect on flocculation efficiency.

Key Words: Biodegradable, Flocculant, Cationic, Starch, Polymer, %N_2

I INTRODUCTION

1.1 Outline:

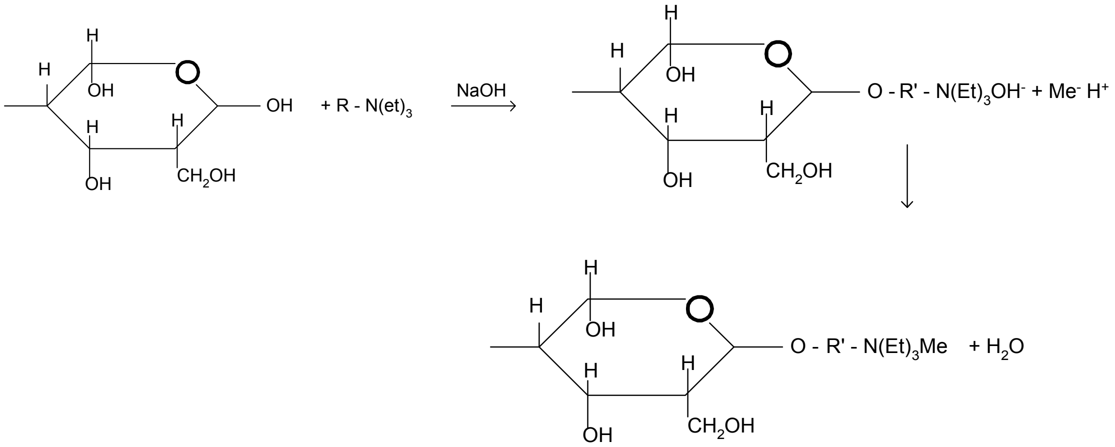
Water is the environment's lifeblood, necessary for all living things - plant, animal, and human - and anything must be done to ensure its purity today and in the future.

Biodegradable "green" and safe substitutes to minimize environmental and health risks are drawing extensive attention for research interest [1, 2, 3]. This research work is experimental to develop a biodegradable starch-grafted polymer flocculant to treat industrial wastewater and make our environment clean and healthy. A grafted starch-based biodegradable polymer is developed to gain both the advantage of natural and synthetic polymers.

Polymers are effective flocculants because they are potent molecules with the ability to carry charges. Small particles can become stuck in polymer chains because they are so big & long, accumulating enough mass to impede their retention in solution. The saturated polymer chains also do not change the pH on adding to wastewater [4, 5].

This work deals with the synthesis of water-soluble cationic biodegradable starch as a flocculant [6, 7, 8, 9]. The reactive groups of the starch, i.e., hydroxyls, form a complex with cations present in the reagent. Free radical sites may form on the polysaccharide backbone if the complex dissociates. The suggested strategy predicts that the mechanism of free radical graft copolymerization of Ethyl monomers onto starch would proceed as expected. In presence of molecular Nitrogen, H+ may be replaced by Reactive group R, i.e., CH_2 CH_2 N (Et_3) in a highly alkaline medium (Ref. to eq.1).

Developed cationic flocculant performs well in a liquid dispersion form. In addition, it is a fast-acting and effective flocculant that may be employed in treating industrial effluent. It combines the low-sludge-production properties of organic flocculants with the cost-effectiveness of inorganic flocculants. The anionic impurities removal efficiency is mostly improved with the increase of the grafting ratio [10]. Hence the amount of nitrogen content was tested to confirm the increase in the grafting ratio. It was evident that the presence of nitrogen increases flocculation activity. The positive charges of nitrogen atoms in the quaternised grafted starch influenced antioxidant activity. The substituted electronegative groups increased the charged density of the grafted starch and had significantly higher hydroxyl and hydrogen peroxide scavenging activity than starch. [11].



…eq. 1

The experiments were carried out by changing various reaction parameters to increase the % N_2 content of the produced polymer and to check its efficiency on flocculation.

1.2 Objective:

This research is to develop a biodegradable flocculant for industrial wastewater and make our environment clean. A grafted starch-based biodegradable polymer is developed to gain both the advantage of natural and synthetic polymers.

Both inorganic and organic flocculants are required for wastewater and industrial effluent treatment. However, polymeric flocculants (synthetic and natural) are chosen among organic flocculants because of their low dosage, ease of handling, inertness to pH changes, capacity to produce substantial cohesive flocs, and varied tolerability. In addition, environmental and ecological concerns justify using a biodegradable flocculant in wastewater and industrial effluent treatment [12, 13, and 14].

Amylase (a low molecular weight linear polymer) and amylopectin are two starch components (a high molecular weight, branched polymer). The grafted amylopectin has been discovered to be an effective flocculant for various industrial effluents.

1.3 Mechanism:

The quantity, size, density, surface qualities of the solid particles, and the density of the exterior phase or dispersion media determine the stability of a suspension.

The particle surface in aqueous suspensions has an electrical (typically negative) charge, and if counter ions (e.g., Ca^{2+} or Mg^{2+}) are present in the surrounding water, they collect on the suspended particles' surface, forming an ionic double layer. Zeta potential is created by excess negative charge at the double layer's sheer surface. The repulsion between the particles grows stronger as the zeta potential rises, and the suspension becomes more stable. The cationity of many commercial cationic flocculants comes from either protonation of amine groups or the creation of quaternary nitrogen groupings at charge locations along the polymer chain. While the latter is unaffected by pH, the cationic charge produced by protonation can be adversely influenced by both pH and ionic strength, to the point that a flocculant may fail to work correctly unless the surrounding solution's acid/base parameters are correct. As a result, cationic polymers are frequently used in conjunction with a pH modifier. The charge patch technique is commonly utilized to bring about flocculation using smaller molecular weight cationic polymers, such as polyethylene amine. [15, 16, 17,18]

Individual particles must move and collide to generate flocs. Flocculation is divided into orthokinetic (as in stirring mechanism) or perikinetic (as in random motion). Particle motion is caused by turbulence in the suspension in the first scenario and Brownian motion in the second. In industrial applications, orthokinetic motion is virtually usually the case.

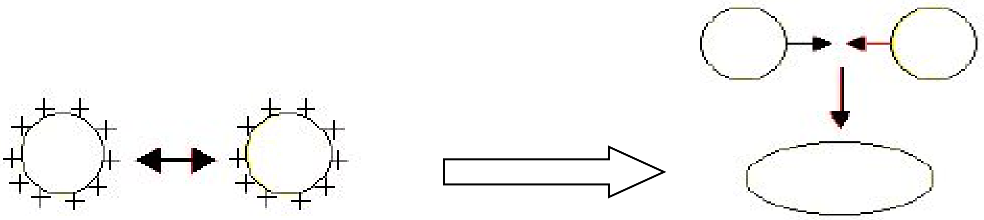
Polar materials are attracted to each other at close distances by dipole-induced dipole interactions, also known as van der Waals forces. Ionization of surface groups gives the particle in most aqueous solutions, and overall negative ionization of surface groups generates the negative charge.

The double layer is a collection of positive ions surrounding the suspended charged particles. When particles approach each other, the electrostatic repulsion of the two layers inhibits flocculation. The repulsion is reduced by increasing the ionic strength of the liquid medium until the particles begin to agglomerate at the critical flocculation concentration. The double layer gets closer to the surface as the charge of these positive ions forming the double layer increases by adding higher charged ions to the system, allowing the particles to become closer and attracted by the van der Waals forces. Double-layer compression is a mechanism that is frequently stated for inorganic flocculating agents that add trivalent ions, such as alum and ferric salts.

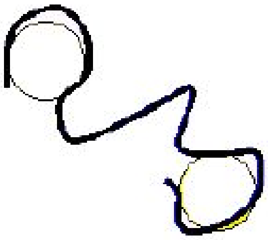
If the molecular weight is high enough, cationic polymers can also bridge between particles. The method for cationic retention aids is referenced as bridging. When the substrate has a strong negative charge, the cationic polymer adsorbs in a flatter shape than the anionic polymer, with fewer loops spreading out to bridge with other particles.

1.3.1 Flocculation Mechanism:

Charge Neutralization:



Like charged particles repel Removal of charge enables particles to coagulate.



Bridging by Flocculant

Fig. 1: Charge Neutralisation

Polymer chains can bridge particles to create a larger mass that settles down (Ref. to fig. 1) Bridging occurs when cationic polymers on the surface of the inorganic particle bind to the tiny fibers. The cationic polymer is usually injected first to neutralize the charge on the particles and generate charge patches. This can also be done with alum or ferric salts. Small flocs are generated, which are then flocculated by an anionic polymer with a larger molecular weight. This frequently results in efficiently removing all suspended particles while maintaining a high level of fine fiber retention. Organic polymeric flocculants can flocculate small hydroxide flocs formed by the hydrolysis of inorganic salts. Increasing nitrogen content can boost cationic charges, producing efficient anionic floc removal. [19, 20, 21, 22, 23]

II. EXPERIMENTAL

2.1 Materials:

All the chemicals employed are analytical grade and used as received.

CH_2 CH_2 N (Et_3) (Reagent) was used. Merck Ltd., Mumbai, supplied methanol, NaOH, and Na_2CO_3. Iso Propyl Alcohol and Acetic Acid were provided by S. D. Fine Chem. Ltd., Mumbai. Starch supplied by Anil Starch Products Ltd., Ahmedabad. The Equipment used is a 3-necked RBF, Supplied by Durasil Glassware, Vadodara.

Three effluents are used for the experiments, procured from three different CETPs. All of them were optimized based on the type of flocculant, pre-treatment was given (i.e., coagulation treatment), various tests were carried out, and price.

2.2 Method:

Preparation of cationic starch-based flocculant

Initially, starch was dispersed in water with a 1:2 to 1:3 ratio to make a slurry. The 3-necked RBF was placed in a water bath at 40°C. NaOH was added to distilled water and used to adjust the pH. In a few batches, Na_2CO_3 was also added with NaOH for pH adjustment. The solution was kept under stirring, and NaOH and Na_2CO_3 were added drop by drop to the stirred slurry for ½ hr. pH was continuously measured, and the temperature was constantly observed. The solution was kept for more than ½ hr under stirring after getting desired pH level of the solution. Then reagent was diluted in water with a 2:3 ratio and added slowly for ½ hr to the alkylated starch. The bath temperature was raised gradually to 55°C in two hours. It was kept under stirring for one more hour at 55°C. pH was continuously checked to keep the solution neutral. Then the solution was cooled down, filtered, and held for air-drying. Optimized parameters to maximize % nitrogen, such as reaction time, reaction temperature, % reagent, and % alkalinity, by varying one parameter and keeping the rest constant.

This cationic starch was prepared by reacting with a triethylamine reagent in the presence of an alkali, as described above. The nitrogen content of starch derivatives was determined by using the Kjeldahl method. This work aims to prepare cationic starch. By changing various reaction parameters, i.e., reaction time, reaction temperature, % reagent, and % alkalinity, nitrogen content was analyzed to establish optimum synthesis. Using these cationic starch as flocculants have been studied on different factors affecting flocculation efficiencies such as optimal dose of flocculant, pH of the flocculating medium, COD, and color removal.

2.3 Polymer characterization:

2.3.1 % N2 content:

The quantitative determination of nitrogen content was carried out using the Kjeldahl method. First, a weighted sample of the mixture was dried. Then, the nitrogen content of the mixture was determined, and the proportion of each component was computed using this information and the known or assumed nitrogen amounts of the two components.

2.3.2 Flocculation

The efficiency of synthesized polymer flocculant was measured with a simple jar test conducted with three different types of Industrial wastewater collected from 3 CETPs. They were given four different treatments: lime-alum, lime–ferrous sulfate, lime–ferric chloride, and without any treatment (raw wastewater). And after flocculation, it was allowed to settle down for 24 Hrs, and then different parameters were tested, like COD, TDS, TSS, Colour, and pH, for three various effluents by giving four different types of treatments to each and using eight different types of flocculants.

III RESULTS AND DISCUSSION

Ten batches were carried out to prepare starch-grafted bio-based cationic polymer flocculant. Different parameters were optimized to get the maximum amount of % Nitrogen for the best flocculation efficiency. All parameters are discussed in detail.

The experiments started at 80°C, 5% of NaOH, 60 min. Reaction time, Na2CO3 was added to adjust the pH and reagent 6% of overall mass. To study one parameter, all other parameters are kept as per the initial condition and changed the parameter to be studied.

Table 1: Effect of reaction Temperature on Nitrogen content

Temp.°C	55	55	60	60	62	62	65	65	70	80
%N2	0.07	0.08	0.57	0.6	0.70	0.8	0.70	0.57	0.6	0.56

Table 2: Effect of amount of NaOH on Nitrogen content

NaOH in gms	0	1	1.5	2	2	5	7	8	9	10
%N_2	0.07	0.08	0.07	0.07	0.1	0.57	0.6	0.56	0.57	0.6

Table 3: Effect of reaction time on Nitrogen content

Reaction Time	45	45	45	60	60	120	120	120	120	120
%N_2	0.57	0.59	0.6	0.6	0.57	0.07	0.1	0.07	0.07	0.08

Table 4: Effect of amount of Na_2CO_3 on Nitrogen content

Na_2CO_3 in gms	0	1	1	1.5	1.5	2	2	2
$\%N_2$	0.07	0.07	0.08	0.07	0.07	0.1	0.07	0.07

Table 5: Effect of quantity of reagent on Nitrogen content

Reagent in ml	16.6	16.6	16.6	20	22	24	25	25	28	30
$\%N_2$	0.07	0.08	0.1	0.57	0.6	0.56	0.57	0.6	0.6	0.59

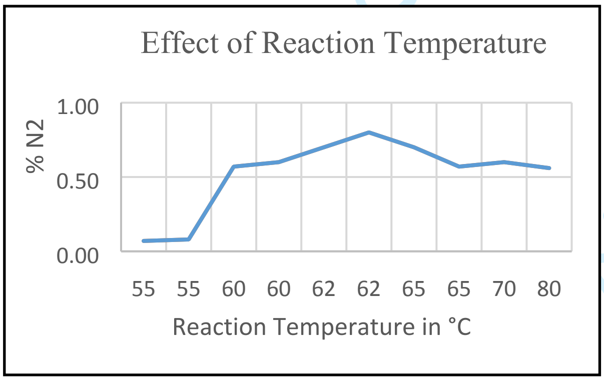


Fig. 2: Effect of reaction Temperature

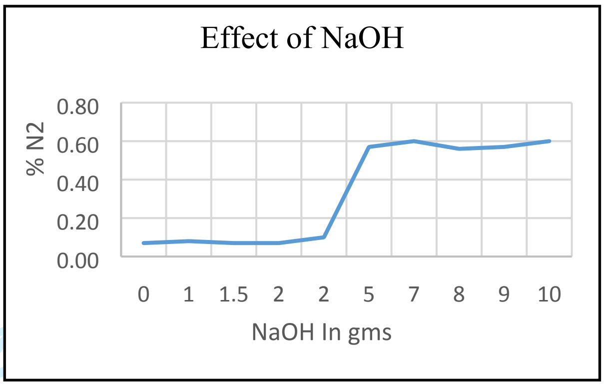


Fig. 3: Effect of amount of NaOH

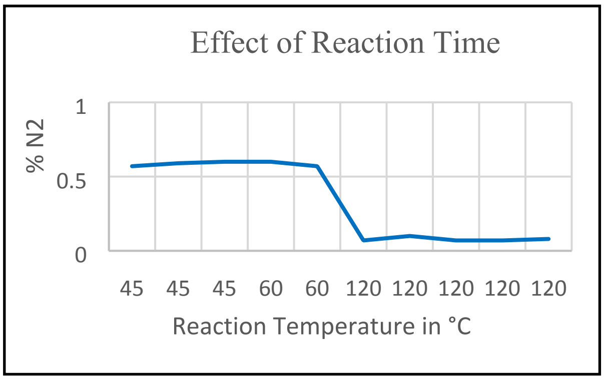


Fig. 4: Effect of amount of Reaction Time

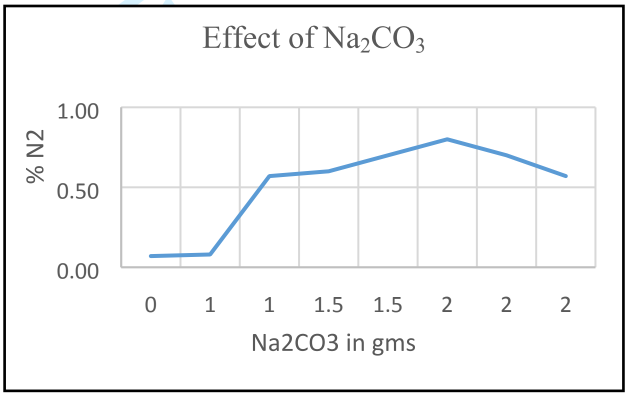


Fig. 5: Effect of amount of Na_2CO_3

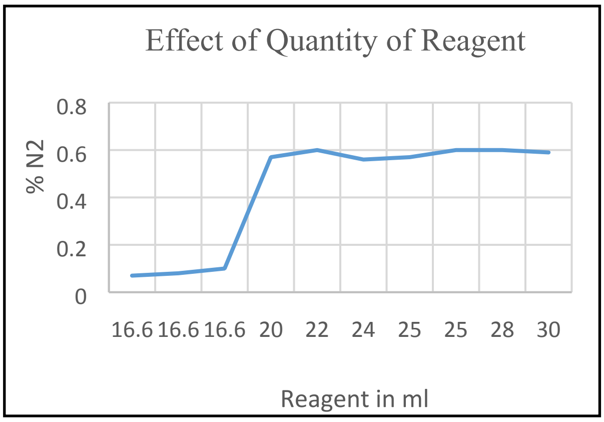


Fig 6: Effect of quantity of reagent

The water to starch ratio was varied from 1:2 to 1:3. It shows that the ideal batch size was 1:3. It was found that the amount of water taken in reaction mass doesn't play any significant role in the final nitrogen content; it only takes part as a mixing medium. (Refer to Table 1, Fig 1). It was tried to optimize temperature for maximum nitrogen content in synthesized flocculant and found that at 62 °C, it achieved 0.8%. It can be observed from fig 1 that when the temperature rises above 55 °C, nitrogen content shoots up and keep rising till 62 °C. Above 62 °C, it shows further reduction in %N2. It was tried to track the fall in %N_2 up to 80°C and found to be reduced up to 0.56%. (Refer to Table 2, Fig 2).

Alkalinity plays a significant role in establishing %N_2 on the flocculant. It has been found that with an increasing quantity of NaOH, the %N_2 found increasing reached up to 0.8%. The ratio of starch to NaOH varied from 1% to 10 % of starch. The maximum N_2 content was found at 2.5%. (Refer to Table 3, Fig 3). Reaction time optimization shows that more than 45 min is needed to complete the reaction. It has been observed that with continuing reaction beyond 45 min, the %N_2 increases. When the reaction was carried out for 120 min, it showed max %N_2. Further increases in time do not show much change in %N_2. Hence 120 min is the optimum time for a reaction. (Refer to Table 4, Fig 4).

Na_2CO_3 was tried in place of NaOH for the reaction as an alkaline medium. A few batches were carried out to explore the possibility of improvement in %N_2. It shows that 1% of Na_2CO_3 with respect to starch is the most optimum qty. (Refer to Table 5, Fig 5).

A reagent used in this synthesis plays a significant role in %N_2. It can be observed from data that %N_2 raising with reagent and reaches a maximum of 12% with respect to starch. On the other hand, the N_2 content increases drastically by increasing Alkalinity. The effect of Time, Temperature, and Reagent is negligible compared to Alkalinity, and after a specific limit, there will be no further effect of Alkalinity. Hence, the most optimized condition for maximizing the %N_2 is a starch/ NaOH ratio of 2.5% at a temperature of 62° C and a starch/ reagent ratio:12%. The effect of the developed flocculant was then analyzed on various parameters.

Table 6.: Effect of Developed flocculant on parameters of Industrial Effluent

Effluent		Developed Flocculant							
	W/O Floc	Lime Alum	Lime Ferrous Sulphate	Lime Ferric Chloride	T1F	T2F	T3F	T4F
pH	7.77	8.58	7.56	7.79	7.32	7.15	7.04	7.08
Colour	187.00	7.48	52.94	90.37	54.55	93.05	51.87	66.31
COD(mg/L)	1560.00	411.00	251.00	1016.00	262.82	210.26	120.51	203.53
TSS(mg/L)	196.00	39.80	105.61	70.41	72.45	65.31	109.18	54.08
TDS(mg/L)	11322.00	1757.23	2434.61	2711.71	2371.54	1852.57	2294.75	2386.38

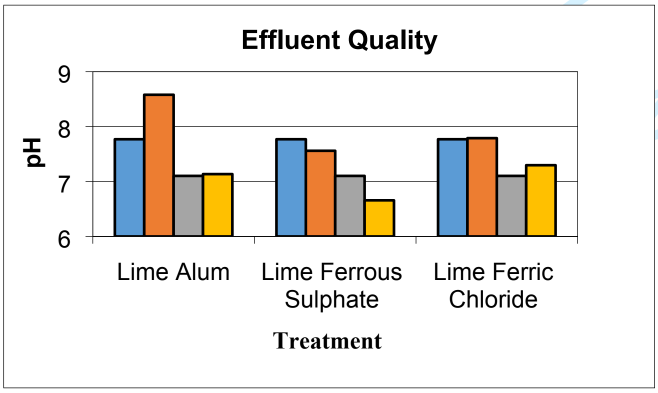


Fig. 7: Effect of Treatment and Flocculant on pH

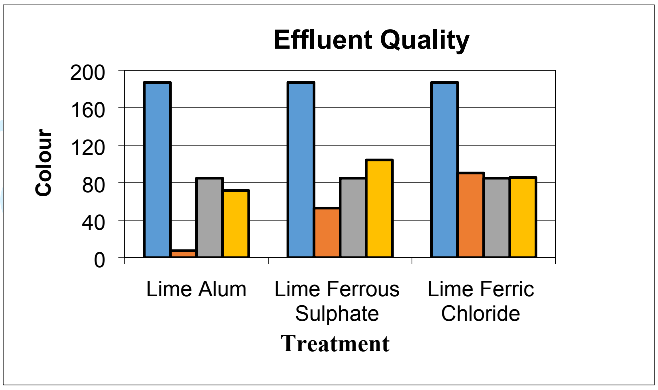
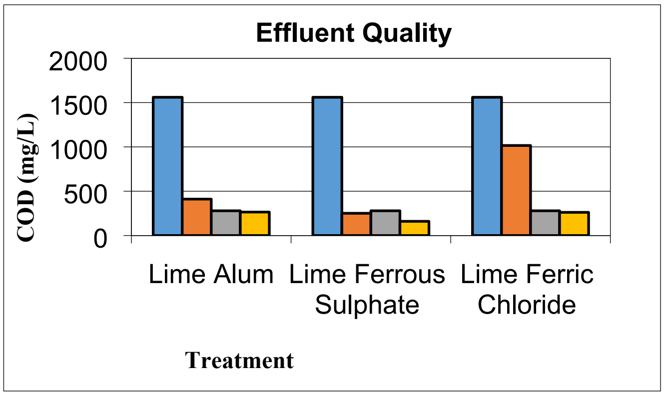


Fig. 8: Effect of Treatment and Flocculant on Colour



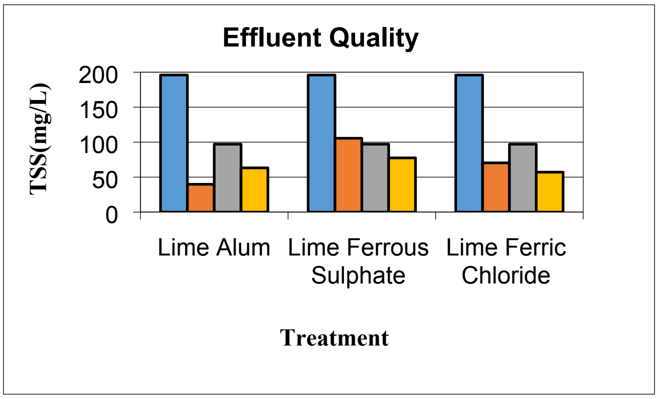


Fig. 9: Effect of Treatment and Flocculant on COD

Fig. 10: Effect of Treatment and Flocculant on TSS

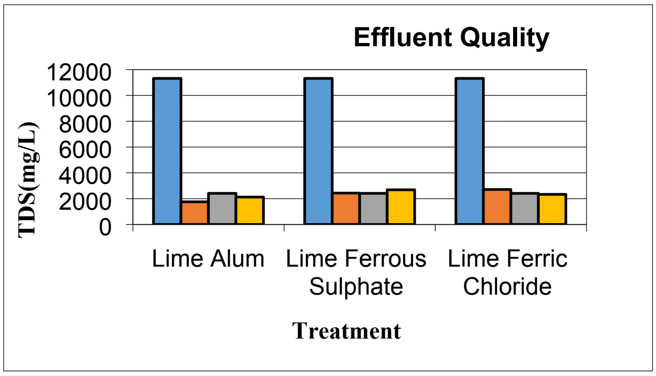


Fig. 11: Effect of Treatment and Flocculant on TDS

Remark:

pH	Appreciable
Colour	Excellent for Lime – Alum
COD	Excellent
TSS	Excellent
TDS	Outstanding

🔵 - Raw Wastewater ⚪ - Only Flocculant

🟠 - Treatment 🟡 - Flocculant + Treatment

T_1 : Without Treatment, T2 : Lime - Alum, T3: – Lime - Ferrous Sulphate, T4: Lime - Ferric Chloride, F – Developed flocculant

The industrial effluent was not from a particular industry but a mixture of wastewater collected at a common plant (CETP). Therefore, the initial quantity of raw water for different parameters is given in Table 6. As it was a mixture of wastewater, various treatments were tried with the developed flocculant, and the results are presented in table 6 (Refer to Table.6). By applying different treatments like; Lime-Alum, Lime-Ferrous Sulphate, and Lime-Ferrous Sulphate with developed flocculant, all the parameters are excellent (Refer fig. 7 to 11). Moreover, the flocculant used is with the highest %N_2 content. Hence, the flocculation efficiency increases with N_2 content.

CONCLUSION:

A developed cationic flocculant is a starch-based flocculant that exhibits a high degree of cationic charge. Once inverted and hydrated in water, react readily to prove floc formation performance in solids/liquids separation processes. Developed Cationic flocculant is suitable in a liquid dispersion form.

The flocculant used is with the highest %N_2 content. Hence, the flocculation efficiency increases with N_2 content.

With all the tree treatment, the pH doesn't change much, as expected. Overall quality improves by using Lime–Alum treatment with flocculation. However, Lime ferric Chloride worked well on COD reduction. TSS was drastically reduced in the combination of Lime-Alum, and all three treatments worked exceptionally well for TDS reduction.

The developed cationic flocculant is found to be a fast-acting and effective flocculant that is employed in the treatment of industrial wastewater. It combines the low-sludge-production properties of organic flocculants with the cost-effectiveness of inorganic flocculants.

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List of Tables

Table 1: Effect of reaction Temperature on Nitrogen content

Temp.°C	55	55	60	60	62	62	65	65	70	80
%N2	0.07	0.08	0.57	0.6	0.70	0.8	0.70	0.57	0.6	0.56

Table 2: Effect of amount of NaOH on Nitrogen content

NaOH in gms	0	1	1.5	2	2	5	7	8	9	10
%N_2	0.07	0.08	0.07	0.07	0.1	0.57	0.6	0.56	0.57	0.6

Table 3: Effect of reaction time on Nitrogen content

Reaction Time	45	45	45	60	60	120	120	120	120	120
%N_2	0.57	0.59	0.6	0.6	0.57	0.07	0.1	0.07	0.07	0.08

Table 4: Effect of amount of Na_2CO_3 on Nitrogen content

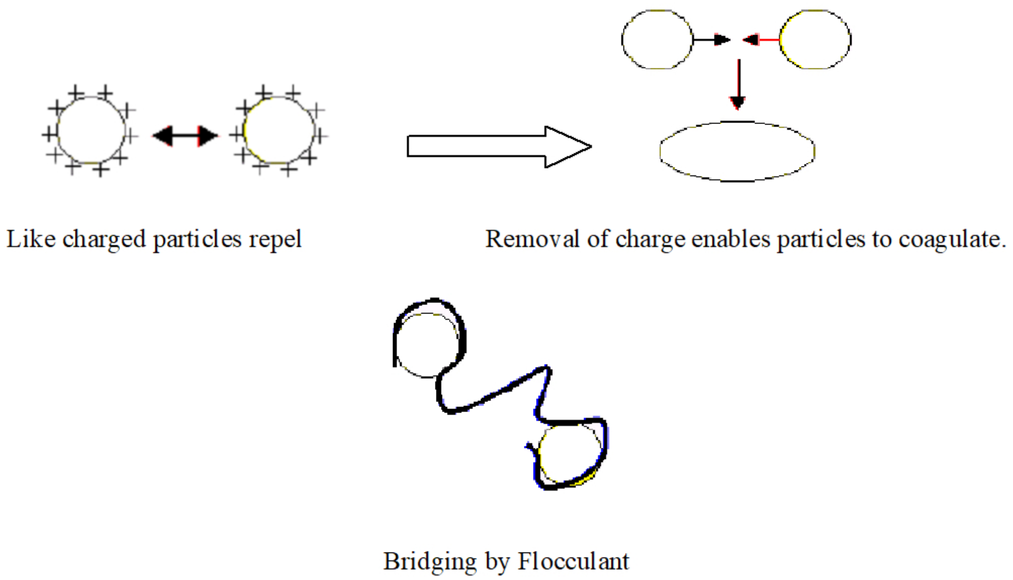
Na_2CO_3 in gms	0	1	1	1.5	1.5	2	2	2
%N_2	0.07	0.07	0.08	0.07	0.07	0.1	0.07	0.07

Table 5: Effect of quantity of reagent on Nitrogen content

Reagent in ml	16.6	16.6	16.6	20	22	24	25	25	28	30
%N_2	0.07	0.08	0.1	0.57	0.6	0.56	0.57	0.6	0.6	0.59

Table 6.: Effect of Developed flocculant on parameters of Industrial Effluent

Effluent		Developed Flocculant						
	W/O Floc	Lime Alum	Lime Ferrous Sulphate	Lime Ferric Chloride	T1F	T2F	T3F	T4F
pH	7.77	8.58	7.56	7.79	7.32	7.15	7.04	7.08
Colour	187.00	7.48	52.94	90.37	54.55	93.05	51.87	66.31
COD(mg/L)	1560.00	411.00	251.00	1016.00	262.82	210.26	120.51	203.53
TSS(mg/L)	196.00	39.80	105.61	70.41	72.45	65.31	109.18	54.08
TDS(mg/L)	11322.00	1757.23	2434.61	2711.71	2371.54	1852.57	2294.75	2386.38



Bridging by flocculant

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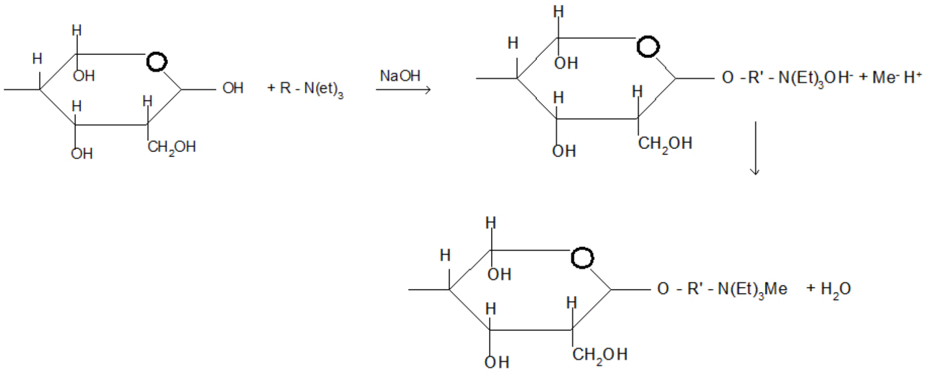

Chemistry of Reaction

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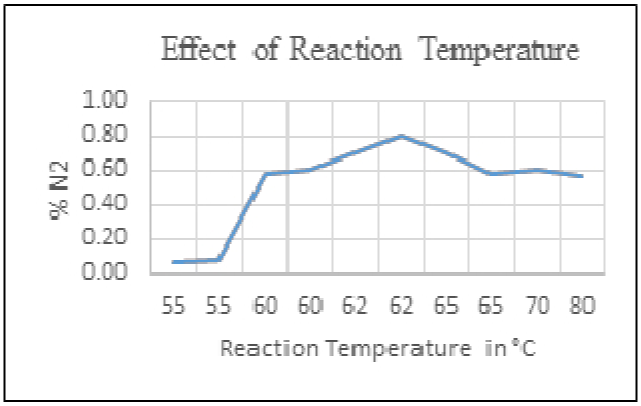

Effect of Reaction Temperature

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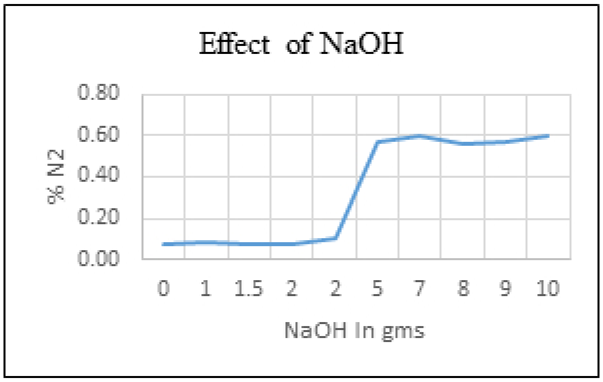

Effect of NaOH

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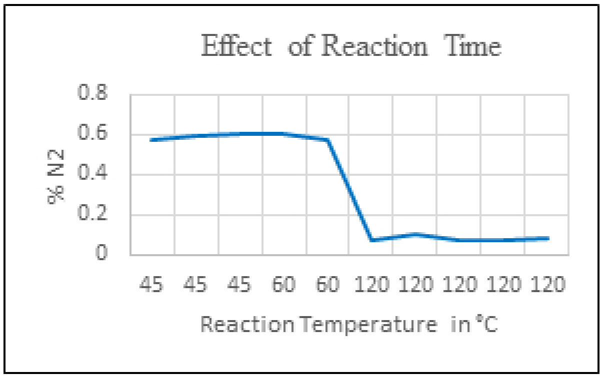

Effect of Reaction Time

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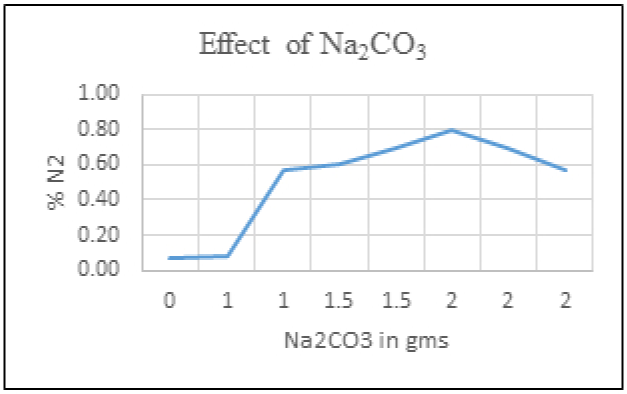

Effect of Na2CO3

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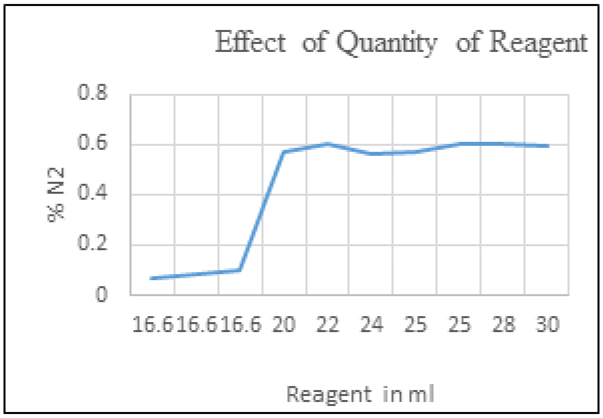

Effect of qty of reagent

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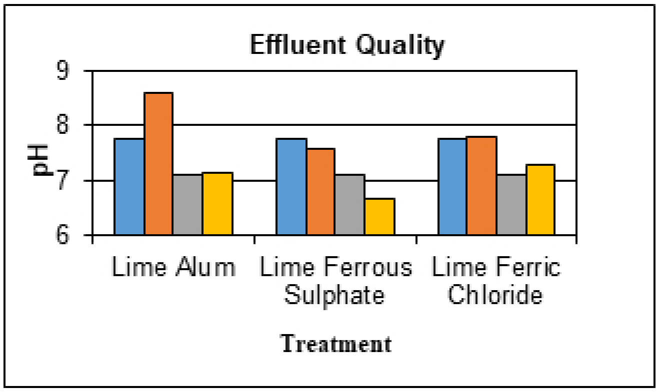

Effect on pH

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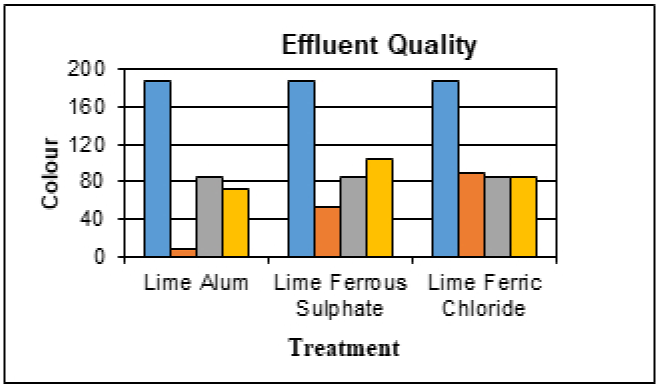

Effect on color

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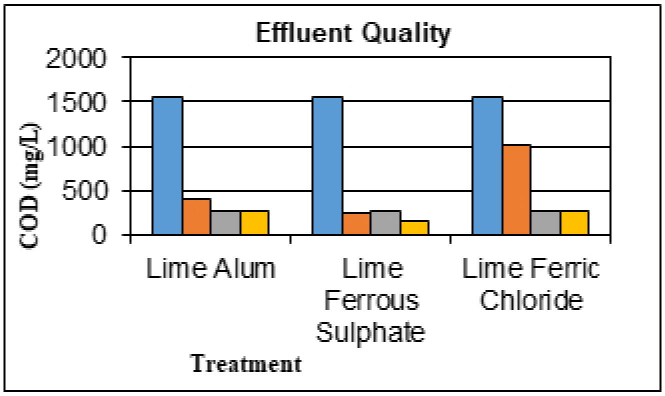

Effect on COD

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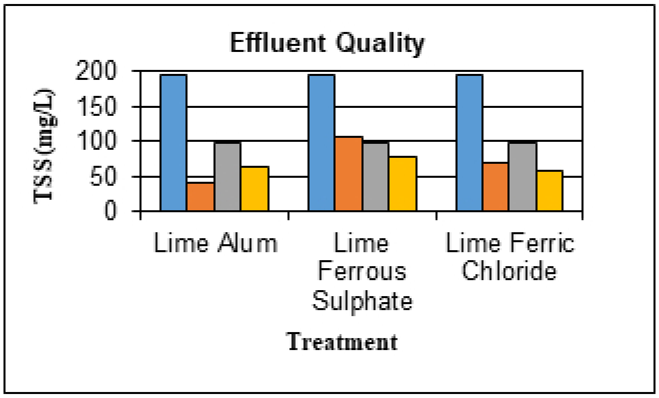

Effect on TSS

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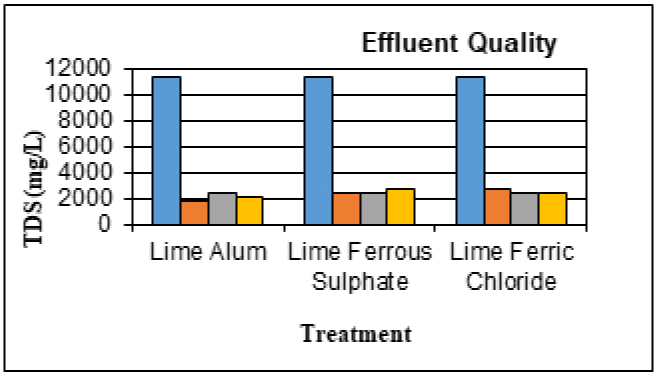

Effect on TDS

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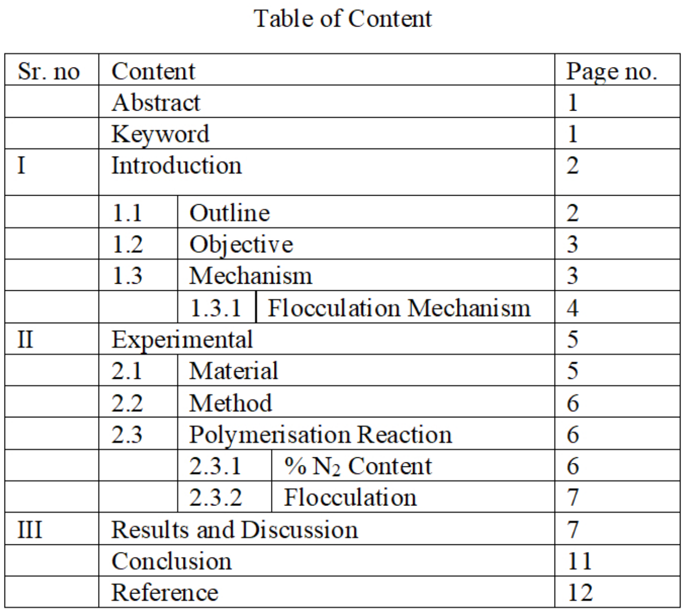

Table of Content

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