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REVIEW

A critical review on flocculants and flocculation

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ABSTRACT

The article critically reviews the applications of biopolymers in the area of water treatment. A number of biopolymers were reported for their flocculation behavior for treating the wastewater and making it suitable for various industrial as well as agricultural purposes via chemical alteration onto its backbone. Wastewater treatment was followed by flocculation process using standard 'Jar test protocol'. Minute quantities of dosage were found be ideal to diminish the organic, biological and suspended loads from wastewater sample. The process of flocculation using biopolymers was also found economically suitable compared to other various processes applied in water treatment.

1. Introduction

From last few decades graft copolymerization uses an advanced procedure to synthesize a novel polymeric material by altering the physical and chemical properties of natural and synthetic biopolymers. It is detained by chemical bonding and permits two biopolymers to be closely linked together. To meet pleasing end user necessities graft copolymerization offers an exclusive procedure for alteration of biopolymeric molecule due to their potential application as viscosifier, and flocculants in mineral ore beneficiation as well as wastewater and mining sludge treatment. Biopolymers with higher solubility in water are new category of polyelectrolyte that reveals their exclusive behaviors by distributing positive charges among the backbone of macromolecular compounds. Due to strong attraction with the negatively charged colloidal particles graft copolymers represent much awareness in wastewater and paper making fields. A number of researches linked with development and significance of graft copolymer as flocculants are reported recently. The literature review is critically based on categorization, mechanism and application of graft copolymers applied dynamically as polymeric flocculants for wastewater treatment.

1.1 Flocculation

In a solution when colloidal particle under goes brownian motion forms bridging bond as a result of their collisions. More colloidal particles get attached onto the polymeric chains due to repetition of process further, build up bigger flocs which steadily settle down in suspension. The phenomenon often referred as flocculation. Various advantages of flocculants include:

(a) An efficient quality of water is obtained for partition

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of colloids with minute quantity (ppm level) than coagulants (ppt. level).

(b) It is cheap, non-toxic and degradable and flocs formed during flocculation are of longer shelf life.

(c) Diminish the concentration of colloidal particles, counting bacteria and microorganisms, heavy metals, turbidity and color present in any water system.

(d). It is applied to remove pollutant load from wastewater when coupled with suitable adsorbent like fly-ash etc.

1.2 Natural vs. synthetic flocculants

Natural polymers are easily accessible from renewable sources and are non-toxic with lower efficiency, thus require high dosage. The chief benefit includes their biodegradability with disadvantage of short life. On the other hand, synthetic polymers are extremely proficient with biological as well as chemical stability, uniformity and product consistency. The functional groups present on their backbone can easily be tailored. Molecular weight and structure of synthetic polymer customize a number of applications with major drawback as most of them shows non-biodegradable, expensive, noxious nature[1,2].

1.3 Polymeric vs. inorganic flocculants

By adding polymeric flocculants with low dosage the flocs formed rapidly and are larger as well as stronger. Polymeric flocculants are more useful with various advantages than inorganic flocculants. On the other hand, pH adjustment often required for inorganic flocculants but not in case of polymeric flocculants. Though, inorganic flocculants are economical and frequently applied for financial motive.

2. Flocculants categorization

The flocculating agents are largely classified into two groups, e.g. organic and inorganic. The first one includes polymers i.e. natural (nonionic apart from some cases) and synthetic (nonionic, cationic or anionic). Further, a novel category of flocculants are generated by joining natural and synthetic polymers, called graft copolymers.

2.1. Inorganic flocculants

A number of chemicals[3-4] were being used for coagulation procedure. E.g. Hydrolysable salts of iron and aluminum.

(a) Iron compounds: pH coagulation acquired by iron compounds with similar features of flocs as well as lower cost often to aluminum sulfate. E.g. ferrous sulfate, ferric chloride, ferric chlorosulfate and ferric sulfate are included among iron compounds and are applied as coagulants.

(b) Aluminum compounds: Aluminum compounds are generally applied for removing suspended solids present in any wastewater sample in dry and liquid forms. Coagulation of wastewater can be enhanced using activated alum which contains 9% of sodium silicate while, black alum include activated carbon with effective pH varies from 5-8.

2.2 Polymeric flocculants

High molecular weight polymeric molecules (Figure 1) with water solubility are applied as flocculants[5-6].

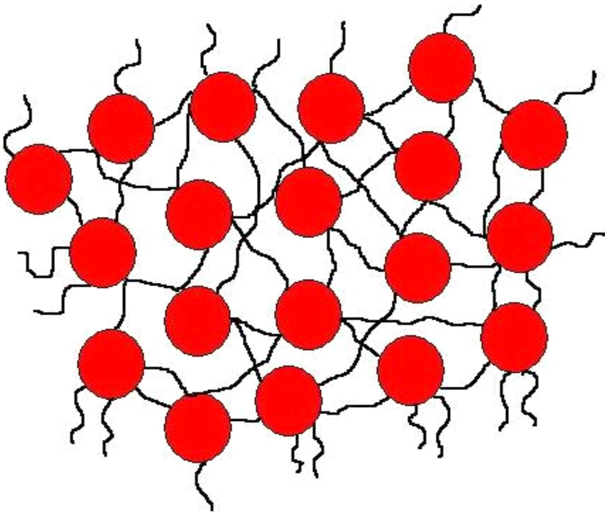

Figure 1. Schematic representation of bridge polymeric flocculants

2.2.1 Nonionic polymers

A monomeric unit in polymer with less than 1% charges is called nonionic polymers. They behave as flocculants via bridging mechanism in any water suspension with higher molecular weight for practical application. The chief member of this group comprises polyacrylamide (PAM), polymethyl methacrylate (PMMA) and polyethylene oxide (PEO) etc.

2.2.2 Natural polymers

A number of materials derived naturally which may be used as flocculants are called natural polymers. E.g. Guar gum, Psyllium, carboxy methyl cellulose, starch, gum ghatti, chitin, chitosan, cellulose, dextran, glycogen, amylopectin, casein, agar, barley, oatmeal and so on.

2.2.3 Graft copolymers

When the main chain is made entirely of one repeat unit, while the branch chains are made of another repeating unit is called graft copolymers (Figure 2). A significant

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technique to alter the property of natural and synthetic polymers is called graft copolymerization. The molecular weight, viscosity, solution rheology includes amount of pseudo plasticity and ion compatibility etc. and gelation property can be hugely altered via grafting phenomenon. Improved functionality after grafting permits them for being effectual in flocculation and more different functions like dry potency additives and preservation assist in paper etc.

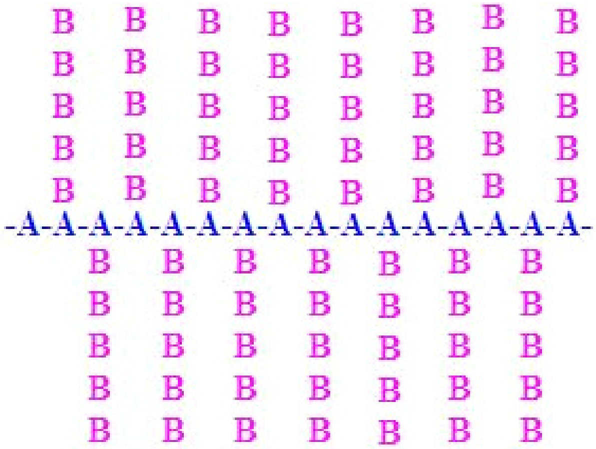

Figure 2. Schematic diagram of graft copolymers

2.2.3 (a) Anionic / Nonionic graft copolymers

Majority of graft copolymerization reaction starts using biopolymers and monomer by radiation induced or ceric ion induced technique further, the synthesized product may be efficiently functionalized as flocculating agents. Examples of this category include hydroxypropyl guar gum[7], P-psyllium[8], guar gum[9], amylopectin[10], sodium alginate[11], amylose[10,12], barley[13], oatmeal[14-18] etc.

2.2.3 (b) Cationic graft copolymers

Graft copolymerization of a variety of quaternary ammonium monomers was attempted onto biopolymer to synthesize enhanced flocculants and color preservation help in paper manufacturing[19].

3. Mechanism of flocculation

3.1 Charge neutralization mechanism

Flocculation phenomenon is more effective via charge neutralization[20] when there is proficient double layer compression simultaneously the ionic strength of solution will enhance. The expression for Debye-Huckel length is given as resultant from Gouy-Chapman model of electrical double layer.

$$[K = (4 \text{ X } 10^{-3} e^{2} N I / \varepsilon K T)^{1/2}]$$

Where,

K = Boltzman constant

N = Avogadro's number

I = Ionic Strength

T = Absolute temperature

ε = Electrical permittivity of solvent

e = Electronic charge

The neutralization phenomenon occurs onto the surface charge of colloidal particle via adsorption of polyelectrolytes with opposite charge. E.g. Al^{3+} Fe^{3+} ions.

Consequently, flocculation of colloidal particles with negative charges is done via adsorption of polymers with cationic groups.

3.2 Bridging mechanism

The fundamental approach of bridging flocculation was first projected by Ruehrwein and Ward in 1952[21]. A model was revealed in which a particular polymeric sequence was bridged among two or more units. This occurrence is realistic up to a fixed dose of polymer away from that flocculation reduced and the procedure is called as steric stabilization (Figure 3). There is no sufficient occurrence of bridging among particles with low polymeric dosage. On the other hand with high polymeric dosage, a reverse phenomenon occurs with inadequate surface area of particles for being attached further, leads destabilization (Figure 3).

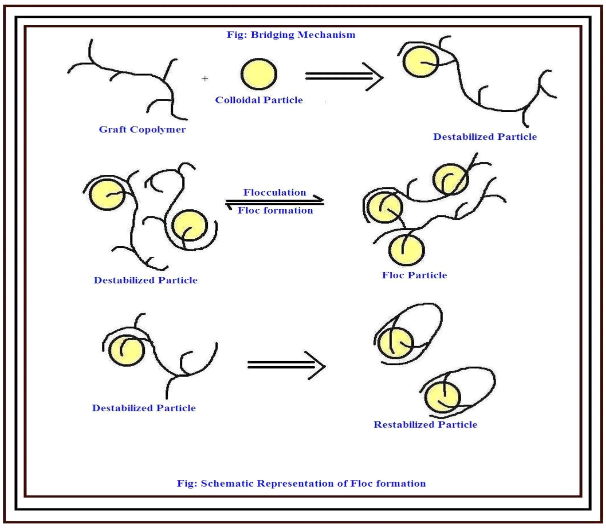

Figure 3. Schematic illustration of bridging flocculation and restabilization of adsorbed polymer

The process elucidates the idea of non-equilibrium flocculation phenomenon[22]. When a particle carrying some active polymer is encountered by other particle with free surface creates flocculation. The existence of optimal arrangement is controlled by adsorption energy of polymer segments to the surface of solid and collision frequency

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between particles, surface area and size of polymer. Higher collision frequency is caused by particles with higher concentration further possibility of polymer in an active state is enhanced. Bridging mechanism includes natural and synthetic bridged polymers. Natural polymers utilized as bridging flocculants with very low molecular weight than synthetic one creates a lower extent of flocculation. Examples include oatmeal, gums, starch, alginates, glues etc. While, synthetic polymers are organic polymers with high solubility in water as well as high molecular weight with the capability to adsorb on particles. On the other hand, the polymers with higher molecular weight get to be adsorbing on more numbers of particles at a time further forms a three-dimensional network.

3.3 Electrostatic patch mechanism

First charge on polymer appeared as ‘island’ patch, bordered by reverse charge region [23]. Though, charge neutralization mechanism and bridging mechanisms reveals non-ionic and anionic polymers effect during flocculation. In anionic colloidal suspensions with an arrangement of high cationic charge[24], the high energy of interaction permits a compressed adsorbed pattern (Figure 4,5) that diminishes loops and trains formation, which will be further able to bridge / connect suspended particles. Such processes don’t neutralize the surface charge of particles separately with oppositely charged polymeric fragments.

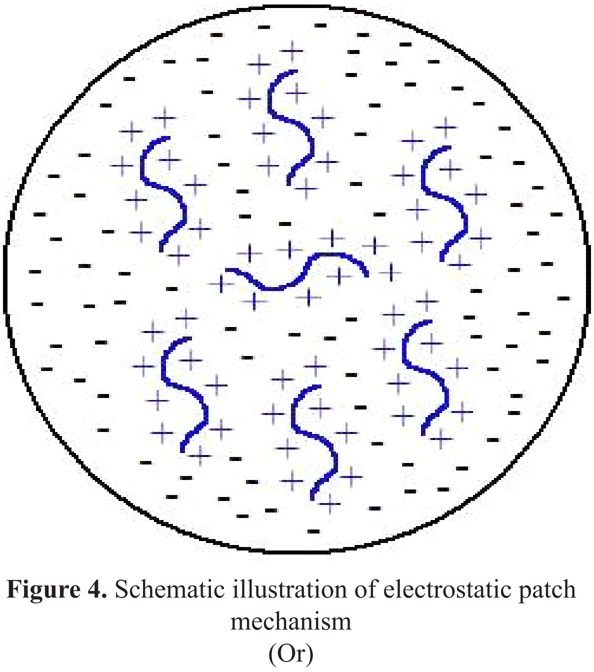

Figure 4. Schematic illustration of electrostatic patch mechanism
(Or)

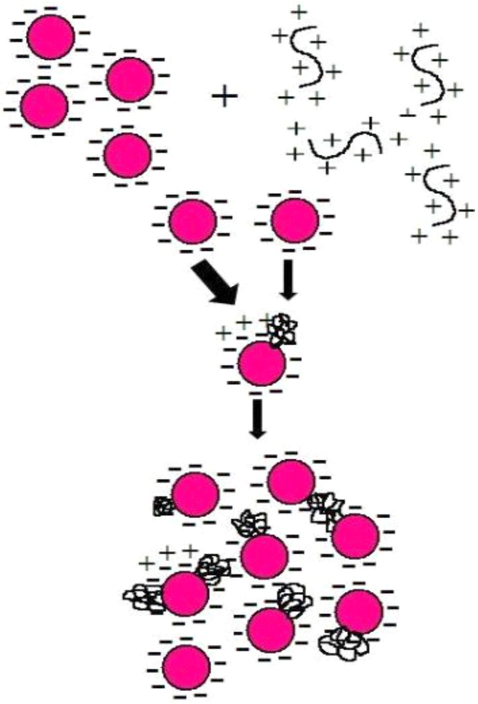

Figure 5. Schematic illustration of electrostatic patch mechanism

3.4 Singh’s easy approachability model

The model deals with the flocculation behavior of unhydrolyzed and hydrolyzed polyacrylamide grafted biopolymers[25-27]. It state that the floppy polyacrylamide (PAM) branches have effortless approachability towards contaminants when they grafted onto the rigid biopolymeric backbone (Figure 6). According to the theory, the more acrylamide chains grafted onto biopolymer chances to form aggregate of the contaminants will be enhanced.

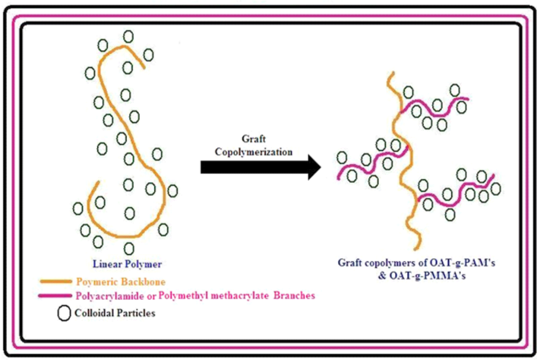

Figure 6. Schematic representation of singh’s easy approachability model

3.5 Brostow, singh and pal model of flocculation

The theory states that radius of gyration is directly proportional to flocculation efficiency & creates a better impact

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on flocculation phenomenon. I.e. radius of gyration will increase and automatically the flocculation efficiency enhanced[28].

$$[y = a.R^{b}_{G}]$$

Where,

a and b = characteristic parameters for a given suspension medium, that is both the dispersed phase and the majority liquid.

R_G = Radius of gyration

y = rate of flocculation

4. Flocculation: the test method

The main principle of flocculation is to create an optimal situation to form flocs[29] with clarification of turbid water sample. On the other hand, settling test can be choosing for solids recovery. Determination of the optimal flocculation condition is based on parameters basically continuous mixing of additives etc. Flocculation is a primarily technique to remove or diminish the concentration of dye, pollutant load in terms of organic loads (BOD_5, COD), suspended particles load (TS, TSS, TDS) and heavy metal ions present in any water system.

4.1 Standard jar test protocol

The standard 'jar test' protocol is most widespread parts of work-table experiment machinery present in water treatment laboratories to recognize possible coagulation-flocculation phenomenon in liquid suspensions. The method applied to verify the favored chemicals with their most excellent dosage. The equipment made up of six equal jars which hold the similar concentration and volume of feed, with six diverse dosages of potentially effectual polymeric flocculants (Fig. 7). The jars are subjected for stirring concurrently at identified rate and rapid mixing of treated feed samples were done and then permitted to settle. Further, the supernatant liquid collected from each jar is subjected for water analysis.

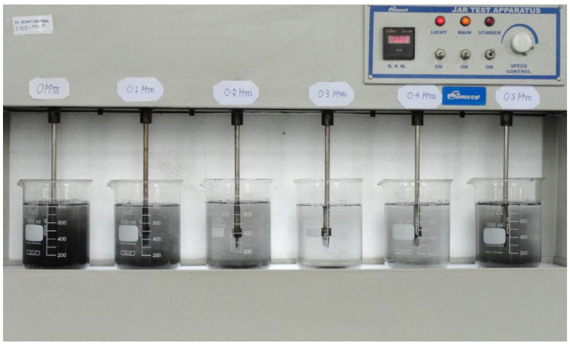

Figure 7. Schematic representation of standard 'jar test' protocol

4.2 The settling test

A traditional settling test performed according to the International standard organization (ISO)[30]. In a cylinder a high solids content suspension was kept and well mixed via continuous shaking. Required dose of flocculants is added. Five times the cylinder is upturned down and upside and permitted for settling which form interface among supernatant and flocs. A curve shows fall in settling time vs. interface height are drowned[31,32]. Few times, for settling study narrow glass tubes (20 cm long and 0.48 cm diameter) are being applied[33]. The experimental set up revealing the settling test phenomenon is depicted in (Figure 8).

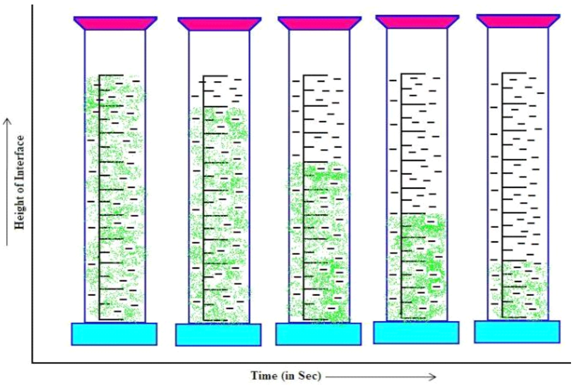

Figure 4. Schematic representation of settling test experiment

5. Coagulation and flocculation

Both dissolved and suspended particles are present in the surface water. Variation of suspended particles occurs in terms of particle size, shape, density, sources and composition charge. A technique employed largely to separate the suspended solids from water is called as coagulation and flocculation[34,35]. Charge neutralization is done by adding inorganic chemicals causes destabilization in coagulation. The little suspended particles are able of join together after neutralizing the charges and forms somewhat superior particles called microflocs, which were not visible by naked eye before coagulation. Examples includes inorganic coagulants like aluminum and iron salts. On the other hand, flocculation includes the flocs formation via adsorption of high molecular weight polymeric materials on colloidal particles surface. The flocs formed are of larger size, bounded strongly and of porous nature. These applications of flocculants over coagulant enhanced the flocculation phenomenon using bridged polymers than using inorganic coagulants.

5.1 Stability of colloidal suspension

In case of colloidal suspension, the striking forces amongst particles occur called as Van der Wall force. Virtual constancy is the standard uniqueness of particles present in water suspension. A well-defined interface between the water and solid phases occurs for hydrophobic particulates with low affinity towards water[36].

5.2 Electrical double layer

Electrostatic adsorption of ions onto the surface contaminates the colloidal particles which give extent to an extensive charge on the surface. These charges on to the surface do two significant things:

(a) Two precipitated particles survive at the time when they approach each other creates repulsion i.e. responsible for particles sticking together fundamentally.

(b) Counter ions get attracted into vicinity of the particles[36].

Electrostatic attraction and ionic diffusion are the two opposite forces which create a cloud of ions diffuse around the particles. This co-existence of neutralizing excess of counter ions and original charged surface are called together as electrical double layer[37]. The thickness of double layer is a function of pH and ionic strength of the solution. The schematic diagram is revealed in Figure 9.

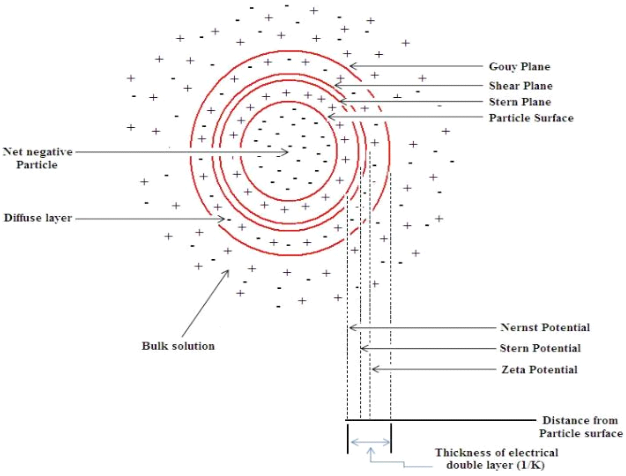

Figure 9. Schematic representation of nature of electrical forces around a colloidal particle in bulk solution

The theory states that the approach of two particles with each other for adhesion is prevented by an energy barrier resulted from the repulsive forces. On the other hand, if collision of particles occurs with adequate energy to overcome the barrier / blockade, then the attractive force will drag them into contact where they adhere strongly and irreversibly together. It is fine revealed in Figure 10.

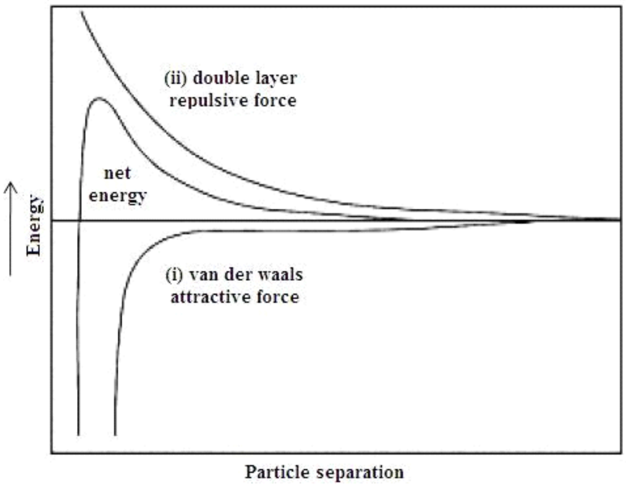

Figure 10. Schematic diagram showing the energy barrier of colloidal particle

6. Zeta potential

A significant indicator of electronic charge on the particle surface, which is used to forecast and manage the constancy of colloidal suspension, is called zeta potential. Discrete charged particles in an ionic media with propensity to have a concentration of oppositely charged ions fascinated for it. I.e. negatively charged particle gathers various positive counter-ions. As we shift a part from the particle, the concentration of counter-ions reduced because of diffusion till it achieved ionic equilibrium. A scheme reveals an exponential decay in charge contribution by these ions vs. distance from the surface of particles (Figure 9). A particle withdraws its counter-ions towards itself during its movement which sets the potential difference and a plane of shear called, as zeta potential (ζ). The suspension is more stable due to greatest zeta potential as a result of repulsion of charged particles thus get to be aggregate. Particles with low values of zeta potential couldn't create any force to avoid the particles coming jointly and causes the dispersion instability. A line dividing among unstable and stable dispersion of water is basically taken at either -30 mV or +30 mV (Figure 11). Particles having zeta potentials more positive than +30 mV as well as more negative than -30 mV are generally measured as stable particle.

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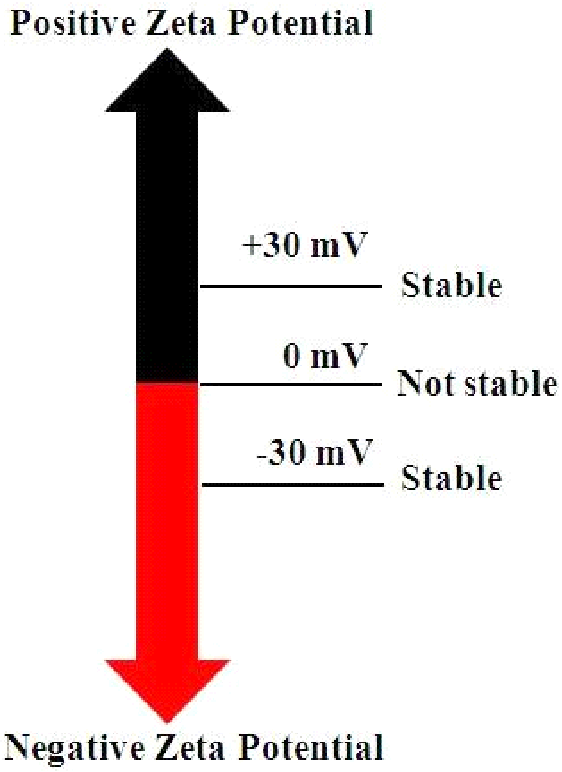

Figure 11. Schematic representation of positive and negative zeta potential

pH is very important thing that influences zeta potential of any suspension. The value of zeta potential without ionic strength, pH and concentration of any additives is invaluable. Generally, it can be states that, pH vs. zeta potential curve will be positive at low pH and it will be negative or lower at high pH (Figure 12). 'Point to zero charge' (PZC) or iso-electric point' is a point where zeta potential is zero at which the system is slightest steady / stable.

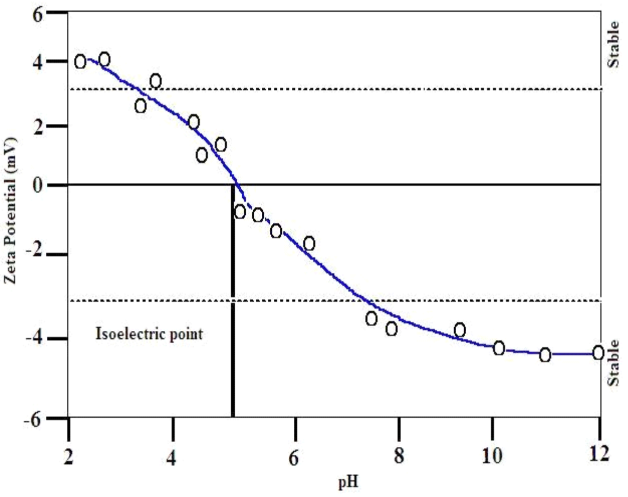

Figure 12. Graphical representation of zeta potential revealing iso-electric point

6.1 Principle of measuring zeta potential

In early 1970s, zeta potential was deliberate via micro-electrophoresis technique [38]. A suitable liquid phase was applied to immerse the sample which was to be measured and being subjected to a beam of laser. The sample was dipped by a pair of electrodes. The charged particles within the sample will shift towards the electrodes and the direction of movement shows the charge carrying by particle, i.e. the positively charges particle will move towards the negative electrode and vice-versa under the influence of an applied electrical field.

The electrophoretic mobility (u) i.e. the velocity of particles per unit electric field is measured using the equation as follow:

$$[u = V / E]$$

Where,

V = velocity of particle

E = strength of an applied field

The equation applied for changing observed movement into efficient electro kinetic potential based on value of dimensional quantity 'ka' [39].

Where,

a = particle radius

k = quantity

$[k = 4 \pi e^2 \Sigma n z^2 / \varepsilon k T]$

From above appearance, $k = 1\times10^6$ cm at 25°C in water ($\varepsilon/\varepsilon_o = 78$) contains 1 mM of 1:1 electrolyte. For other concentration 'k' value follow easy proportion. If 'ka'>200 it will be adequately precise to apply smouluchowski formula in unique unrationalised form is $u = \varepsilon\zeta/4\pi\eta$.

Where,

ε = permittivity of the suspending medium

η = viscosity

Less than one volt per cm, for micron per second, in case of water at 25°C, zeta potential in mV is as:

$[\zeta = 12.83 u]$

6.2 Implication of the measured zeta potential

From 'electro kinetic unit' surfaces moving through the solution the best zeta potential can be pragmatic. Ions basically adsorbed from the solutions are included by the 'electro kinetic' entity and reflected in the zeta potential value. Diffused doubled layer in the solution of zeta potential is due to the dependents of its relevant potential on diffuse layer. Particle charge can also be calculated from zeta potential[39].

6.3 Zeta potential and suspension stability

Suspension is more stable with increasing value of zeta

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potential. If the surface charge of particle is strong, it results in major mutual repulsion between particles keeping out the particles to come close. These repulsion forces are eradicated during zero charge of these particles[40].

7. Factors affecting the flocculation phenomenon

7.1 Effects of polymer molecular weight and charge density

There is a tendency for each polymer molecule to adsorb on to a single particle with their low molecular weight as a sequence of adding these polymers; the degree of flocculation is diminished. An increased quantity of adsorption to form flocs can be obtained using higher molecular weight polymeric materials. Both the settling rate and optimum dose increased along with polymers of high molecular weight. An extremely elevated molecular weight anionic flocculants for treating coal washery effluents are increasingly significant for achieving quicker rates of sedimentation. Though, the polymers with lower molecular weight are sufficient for filtration purpose.

7.2 Effects of polymer dosage and mixing conditions

Dosing and the mixing sequences affect the extent of flocculation[41]. Beyond a certain optimum dosage of polymers further there are no increases in flocculation of any system and diminish competence by further adding flocculants. A relatively lower dosage of polymers work for higher concentrations of solid particulates with rapid flocculation phenomenon, but the formed flocs breaks easily at reasonable rates of stirring due to their instability. Size / settling rate of flocs can easily be detained at reduced stirring rates at raised levels after polymer addition, without successive decline. The extent of flocculation diminished at high concentration and particles are entirely enclosed by absorbed layers of polymers. In flocculation phenomenon, hydrodynamic factors raised from mechanical agitation play vital role[42].

7.3 Effect of shear on the flocs

The flocs, either formed via bridging flocculation or natural coagulation are focused for break with extreme shake-up. During agitation of excess polymers, formed flocs degrade quicker than to the flocs formed with polymers in optimum ratio. Due to repulsion of excess adsorbed polymer the efficient formation of flocs are not possible so, optimal polymeric dosage only grip for an exacting agitation degree.

7.4 Solution properties

7.4.1 Ionic strength

Ionic strength of any solution is significantly affected the configuration of polyelectrolyte which further effects the flocculation phenomenon. The ionic strength decreases as a result of increases in viscosity of solution containing polyelectrolyte as a result of repulsion in mutual charges. The charged sites are shielded with increased ionic strength which permits the folding of polymers and presume a slighter hydrodynamic volume shows via diminution in the viscosity of solution. A system containing high solids for flocculation with polymer having higher molecular weight shows decreases in ionic strength in solution and by increasing the effective particle radius bridging is improved.

7.4.2 pH effects

The efficient class may be solvated metal ion influence the flocculation with inorganic flocculants via Schulze-Hardy effects and double-layer compression. These species become charged with raise in pH value and mechanism of action varies. Presence of ionizable basic or acidic group affects the protonation by changes in pH which further affects surface charge of colloidal particles.

7.4.3 Effect of particle size

A brawny relation among molecular weight of polymers and aggregation of particle size exists via bridging mechanism to form the flocs on the other hand forces occur for breaking the flocs, i.e. instability. The enhanced charge neutralization capability of the surface of particle is done via augmentation in surface area using polymeric flocculants[43].

7.4.4 Effect of temperature

Flocculation is improved by increases in temperature, but not applicable in each case. Diffusion rate and the rate of particle collision enhanced with increased temperature. On the other hand, adsorption which is an exothermic process having unfavorable affect with higher temperature. Temperature changes with linear extension in polymeric chains. Thus, the predicting the temperature effect in any system is extremely complex.

8. Applications of flocculation

8.1. Recovery of industrial effluents and municipal wastewater

A typical water treatment system (Figure 13) holds the

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promise to produce an excellence of drinking water from raw water quality. For the clarification purpose of potable water and water discharged from industries flocculants are being applied increasingly by removing suspended particulates with their fundamental applications. Other application includes flotation processes and gravity separation to treat municipal and industrial wastewater. Graft copolymers are mainly applied for the treatment of wastewater containing organic compounds which principally removes heavy metal contents, organic loads and suspended as well as dissolved particles produced from chemicals, pulp & paper, metals, petroleum refining, textile and food processing industries.

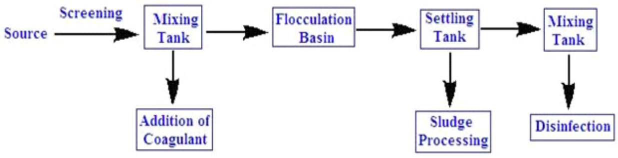

Figure 13. Schematic representation of typical water treatment plant

8.2 Selective flocculation

The procedure engrosses the flocs separation either by froth flotation or sedimentation in a discrete mixture or ore suspension[44,45]. The processes are of three different steps:

(a) Dispersion of fine particles.

(b) Formation of flocs via selective adsorption of polymeric backbone onto the flocculating particles of interest.

(c) Separation of flocs in mineral processing with their major application.

The process may also apply in different kinds of colloidal and biological systems[46]. The applications include ceramic powders clarification, hazardous solid partition from chemical waste and poisonous component exclusion from paper pulp[47-49].

8.3 Paper manufacturing

Fillers like carbonate, titanium dioxide and clays etc. are being applied to supply whiteness as well dullness to paper. During drainage the overcome of expensive fillers and fines is considerably diminished by adding flocculants both alum and bridge polymers are functionalized for negative paper fibers neutralization.

8.4 Mineral processing

Solid and liquid must be separated from each other during all mining operations being influenced by flocculants for thickening in froth flotation as well as clarification processes.

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