



Remediation of Pharmaceutical Effluents by Coagulation-Flocculation Process using Agro-Product

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Abstract The capability of *mucuna pruriens* seed coagulant (MSS), for the removal of total suspended and dissolved particles (TDSP) in pharmaceutical effluent (PHE) was undertaken at room temperature using specified doses at pH of 4.3 and 11.3. Alum was used under the same condition as a control. The turbidity measurement was based on nephelometric standard method. The influence of coagulant dose, settling time, and pH on TDSP removal was investigated. Coagulation – flocculation kinetic parameters such as rate constant K_m , period $\tau_{1/2}$ etc. were evaluated. The optimal parameters obtained for MSS are k_m ($4 \times 10^{-5} \text{ m}^3/\text{kg.S}$), dosage ($0.6 \times 10^{-3} \text{ kg/m}^3$), pH (11.3), $\tau_{1/2}$ (33.3S) and E(%) 82%. At the conditions of the experiment MSS performed relatively better than alum in alkaline medium.

Keywords Remediation, pharmaceutical effluent, coagulation, flocculation, agro-product

Introduction

The increased level of industrialization and urbanization over the years have resulted in the waste generation from domestic and industrial sources, which invariably make water bodies unfit and dangerous to both man and aquatic animals. Pharmaceutical industries are one of the major contributors of hazardous and toxic waste [1]. As a result of the inherent hazardous characteristics, pharmaceutical wastewater, if disposed with insufficient treatment may lead to great damage to the environmental and groundwater resources. Hence, quick and simple methods need to be developed for wastewater treatment prior to discharge. Pharmaceutical industry wastewater is known to contain chemical remnants from antibiotics, anticonvulsants, painkillers, cytostatic drugs, hormones, lipids, regulators, beta blocks, antihistamines. The frequent occurrence of these compounds in streams, some of which are used as sources of drinking water, gives rise to concern over the potential for these compounds to affect human health through chronic exposure [2]. Characteristics of this pharmaceutical wastewater differ widely. Therefore, a treatment process selected for a given wastewater may not be suitable for another. However, coagulation and flocculation option seem to be preferred due its unique nature: safe, simple, effective, cheap and versatile in improving surface and wastewater PFRA [3].

Coagulation and flocculation as a unit process operation, plays an important role in the conventional water and wastewater treatment. Coagulation traditionally entails the addition of metal salt (Aluminum sulphate, ferric chloride) during relatively intense mixing to destabilize naturally occurring particles and macromolecules or to precipitate additional particles [4]. However, studies have shown that there are some limitations attributed to the use of these conventional coagulants: production of a large sludge volume which do not readily settle, making the process slow and expensive, increase of total dissolved solid in the treated effluent [5].

Against this back drop, attention is given to the study on *mucuna pruriens* seed as a potential alternative source of coagulant of plant origin.

Mucuna pruriens seed is of a tropical plant, non-toxic, ecofriendly and easily biodegradable. Previous work on the application *mucuna sloanei* seed in the treatment of coal washery effluent was promising and effective in removal of colloidal particles [6]. Since *mucuna* seeds are found abundantly in the country and in line with quest for backward integration of the economy, this study aims at exploring the potential application of *mucuna pruriens* seed as a coagulant for the treatment of pharmaceutical effluent.



Material and Methods

Material sampling, preparation and characterization.

Sample collection

The PHE was collected at Awka, in a black polyethylene bottle, tightly covered (to avoid photochemical reaction) and characterized according to AWWA standard method on arrival to the laboratory. The characteristics result is presented in table 1.

Mucuna Pruriens Seed

Mucuna pruriens seed sample was obtained from Nsukka Nigeria and prepared according to method reported by Adebawale and Adebawale, 2007 [7].

Coagulation Experiment

Experiments were carried out using bench scale jar test method. Appropriate doses of MSS in the range of 0.1 to 0.6×10^{-3} kg were added to 250 ml of PHE and tuned to pH 4.3 and 11.3 in each case by application of 10M HCl and NaOH, respectively. The suspension was subjected to 2 min of rapid mixing (100 rpm), 18 min of slow mixing (10 rpm), followed by 40 min settling. 10 ml of the sample were withdrawn using pipette from 2 cm depth at various time intervals of 2,4,6,10,20,30 min and analyzed for turbidity using turbidimeter 212R model. The coagulation pH was kept at 4.3. The above procedure was repeated for the coagulation pH of 11.3. The procedure was also repeated using Aluminum salt in a control experiment.

Theoretical Principle and Particle Aggregation Kinetics

Relationship between turbidity and TDSP for effluents systems is generically expressed as product of turbidity (NTU) and TDSP factor (mg/l) [8].

Coag-flocculation studies by Von Smoluchowski (1917) [9], show that coagulation rate is determined from the principles of aggregation of colloidal particles which is controlled by Brownian movement. For such process, time evolution of the cluster size distribution for monodispersed colloidal particles of size i and j to form particle of size K is described by Jin(2005) [10] as:

$$\frac{dN_k}{dt} = \frac{1}{2} \sum_{i+j=k}^{\infty} \beta_{Br}(i,j)n_i n_j - \sum_{i=1}^{\infty} \beta_{Br}(i,k)n_i n_k \quad (1)$$

Where $\beta_{Br}(i,j)$ is Brownian aggregation factor for flocculation transport mechanism, n_i, n_j is particle aggregation concentration for particle of size i and j , respectively.

It has been established that [10-11]:

$$\beta_{Br} = \frac{8}{3} \varepsilon_p \frac{K_B T}{\eta} \quad (2)$$

Where K_B , T , η , ε_p are Boltzman constant, temperature, viscosity and collision efficiency factor, respectively. The general equation representing particles aggregation (singlet, doublet and triplet) as a function of time is obtained by solving the combination of equations 1 and 2 analytically to yield

$$- \frac{dN_t}{dt} = K_m N_t^\alpha \quad (3)$$

Where N_t is total particle concentration (TDSP) at time t , $N_t = \sum n_k$

$$\text{And } K_m = \frac{1}{2} \beta_{Br} \quad (4)$$

K_m is defined as Menkonu coag-flocculation constant accounting for Brownian Coag-flocculation transport of destabilized particles at α^{th} order. N_t is the concentration of TDSP at time t [12-14]. Hence equation 3 transposes to:

$$- \frac{dN_t}{dt} = K N_t^\alpha \quad (5)$$

$$\text{Also } \beta_{Br} = 2 \varepsilon_p K_R = K_m \quad (6)$$

Combining equation 3,4 and 5 yields:

$$- \frac{dN_t}{dt} = \varepsilon_p K_R N_t^\alpha \quad (7)$$

Where K_R is von smoluchowski rate constant for rapid coagulation [12, 15] given as:

$$K_R = 8 \Pi a D \quad (8)$$

Where 'a' is particle radius [15-16]

$$D = K_B T / B \quad (9)$$

Where D is diffusion coefficient, B is the friction factor.

$$\text{However } B = 6 \Pi \eta a \quad (10)$$

Where η is the viscosity of the medium of coagulating and flocculating effluent).

$$\text{Substituting equations 8 and 9 into equation 7, show } K_R = \frac{4}{3} \frac{K_B T}{\eta} \quad (11)$$

Graphical representation of linear form of equation (5) at $\alpha = 2$ yields:



$$\frac{1}{N_t} = K_m t + \frac{1}{N_o} \quad (12)$$

Where N_t is the final particle concentration at time t , N_o is the initial N_t at $t = 0$

Equation 12 can be solved to obtain Coag – flocculation period, $\tau_{1/2}$

$$\tau_{1/2} = (0.5 N_o K_m)^{-1} \quad (13)$$

Equation (1) solved results in generic equation (14) for microscopic aggregation

$$\frac{N_m(t)}{N_o} = \frac{(t/\tau_{1/2})^{m-1}}{(1+t/\tau_{1/2})^{m+1}} \quad (14)$$

Equation (14) gives generalized expression for particle of any m^{th} order. Hence for primary particles $m = 1$

$$N_{m1,t} = N_o \left(\frac{1}{(1+t/\tau_{1/2})^2} \right) \quad (14a)$$

For doublet $m = 2$

$$N_{m2}(t) = N_o \left(\frac{(t/\tau_{1/2})}{(1+t/\tau_{1/2})^3} \right) \quad (14b)$$

For triplet $m = 3$

$$N_{m3(t)} = N_o \left(\frac{(t/\tau_{1/2})^2}{(1+t/\tau_{1/2})^4} \right)$$

(14c) Evaluation of coag-flocculation efficiency is given as:

$$E\% = \left(\frac{N_o - N_t}{N_o} \right) 100 \quad (15)$$

Results and Discussion

The effect of settling time on the coagulant efficiency

The general observable coagulation and flocculation behaviors in figures 1 to 4 show that coagulation efficiency increases with time. The E% indicated the effectiveness of MSS to remove TDSP from the effluent. The maximum efficiency recorded after 30 minutes of coagulation flocculation were 48% for MSS (pH 4.3), 82% for MSS (pH 11.3), 69% for Alum (pH4.3) and 44% for Alum (pH 11.3). This is expected because on the addition of predominantly positively charged coagulants to a negatively charged particle dispersion, there is no energy barrier to aggregation. Thus, the process of aggregation extends to a strong link to cluster – cluster aggregation or bridging mechanism [17]. Aggregation taking place between MSS and TDSP in the effluent presented to be more effective in pH 11.3. This indicated at the experimental conditions that MSS can act as an aggregating agent.

Effect of coagulant dosage on efficiency

Figures 1 to 4, also show the variation coagulant efficiency with dosage. Figures 1 and 3, show that at 30 minutes MSS was found to be most effective for TDSP removal at a dosage of $0.3 \times 10^{-3} \text{ kg/m}^3$ (pH 4.3) and $0.4 \times 10^{-3} \text{ kg/m}^3$ (pH 11.3), respectively. The results obtained are product of gentle destabilization (WST, 2005). However, figure 2, shows that alum was not very effective at low dose, but at a higher dose of $0.6 \times 10^{-3} \text{ kg/m}^3$ pH 4.3, while figure 4, shows that alum was very effective at low dose of $0.4 \times 10^{-3} \text{ kg/m}^3$ (pH 11.3). This supports the fact that alum is known to perform best under alkaline medium [18]. Similarly, it was observed that the organic coagulant MSS was equally good under alkaline medium.

Coagulation-Flocculation Kinetic Parameters

The values of coagulation – flocculation Kinetic parameters are presented in tables 2 to 5. Broadly, the values of R^2 obtained for MSS relatively represent the existence of perikinetic at $\alpha=2$. It was generally observed in tables 2 to 5, that relatively higher values of k_m were obtained, at pH(11.3) than that of pH(4.3). In theoretical terms $\tau_{1/2}$, ϵ_p , and K_R are particle coagulation effectiveness factors, known to be responsible for the coagulation efficiency prior to particle aggregation. The $\tau_{1/2}$ values obtained in tables 2 – 5 are relatively satisfactory, though milliseconds has been reported in systems without stabilizing repulsive interactions (Smoluchowski, 1917). ϵ_p , which is particle collision efficiency is proportional to the kinetic energy acquired by the colliding particles. Hence, high ϵ_p , results in high kinetic energy to overcome the repulsive forces by stabilization. Also observation from tables 2 to 5, show that the highest value of k_m is recorded in pH 11.3, for MSS (Table 4). Meanwhile, k_m relates proportionally to β_{BR} . This is an indication that increase in k_m leads to greater collisions between TDSP particles in effluent as seen in equation (4) ($k_m = \frac{1}{2} \beta_{BR}$) and tables 2 to 5.

In addition, equation (12) represented in figs (5 – 8), show that k_m values are obtained as the slope, and it relates inversely to the fractional final particle concentration ($\frac{1}{N_t}$) or ($\frac{1}{TDSP}$). Similarly, the rate equation values reported in tables 2 to 5 were obtained from equation (5) and it relates proportionally to k_m and N_t (TDSP), which accounts for the rate of depletion of TDSP in the effluent. Thus, higher value of rate equation leads to a higher reduction of TDSP in the effluent.



Particle size distribution behavior as a time dependent function

Equations 14a – 14c are associated with the pattern and distribution of ions/particles as they floc into visible blobs and are graphically presented in figs 9 – 12. These figures actually represent the plots of particle aggregation concentration (N_m) versus time obtained for MSS and alum at pH (4.3 and 11.3) respectively. The figures generally, exhibit similar trend, which is an indication of a process being controlled by a similar mechanism. Critical observation of figure 10 shows that the concentrations of the TDSP for N_{m1} , N_{m2} and N_{m3} are constant at 30 minutes and that of figure 11 are constant at 20 and 30 min, an indication of coagulation process dominated by colloidal destabilization mechanism. Generally, critical observation of the figures shows that there is apparent sharp decrease in the concentrations (N_{m1} , N_{m2} and N_{m3}) between time zero and five minutes. This is in agreement with other similar work [13, 19].

Table 1: Characteristics of wastewater sample before treatment.

Parameter	Values
Temperature (°C)	27.1
Electrical Conductivity $\mu\text{S/cm}$	0.294
TSS (mg/l)	880
TDS (mg/l)	1000
pH	4.92
phenols (mg/l)	Nil
Odour	acidic
Total hardness (mg/l)	220
Ca^{2+} (mg/l)	0.5
Mg^{2+} (mg/l)	0.8
Calcium (mg/l)	177.5
Magnesium (mg/l)	250
Chlorides (mg/l)	100
Dissolved oxygen (mg/l)	16.7
Biochemical Oxygen Demand (mg/l)	26.7
Chemical Oxygen Demand (mg/l)	10
Fe^{2+} (mg/l)	1.49
Turbidity (NTU)	262
Iron mg/l	Nil
nitrate mg/l	Nil
Total acidity (mg/l)	180
Total viable count (cfu/ml)	9×10^1
Total coliform MPN/100ml	0
Total Coliform count cfu/ml	1×10^1
Faecal count MPN/ml	Nil
Clostridium perfringens MPN/ml	Nil

Table 2: Coagulation-Flocculation functional parameters for a second order system at pH 4.3 (MSS).

Coagulant dosage $\times 10^{-3}$ (m^3/kg)	K_m ($\text{m}^3/\text{kg}\cdot\text{S}$)	Rate Equation (-r)	R^2	β_{br} ($\text{m}^3/\text{kg}\cdot\text{S}$)	$\tau_{1/2}$ (S)	ϵ_p (kg^{-1})	K_R (m^3/S)	Reaction order
0.1	6×10^{-6}	$6 \times 10^{-6} N_t^2$	0.897	1.2×10^{-5}	162.9	7.849×10^{13}	1.529×10^{-19}	2
0.2	7×10^{-6}	$7 \times 10^{-6} N_t^2$	0.934	1.4×10^{-5}	139.6	9.157×10^{13}	1.529×10^{-19}	2
0.3	6×10^{-6}	$6 \times 10^{-6} N_t^2$	0.754	1.2×10^{-5}	162.9	7.835×10^{13}	1.532×10^{-19}	2
0.4	6×10^{-6}	$6 \times 10^{-6} N_t^2$	0.505	1.2×10^{-5}	162.9	7.822×10^{13}	1.534×10^{-19}	2
0.5	6×10^{-6}	$6 \times 10^{-6} N_t^2$	0.843	1.2×10^{-5}	162.9	7.804×10^{13}	1.538×10^{-19}	2
0.6	5×10^{-6}	$5 \times 10^{-5} N_t^2$	0.952	1×10^{-5}	195.5	6.497×10^{13}	1.539×10^{-19}	2

Table 3: Coagulation-Flocculation functional parameters for a second order system at pH 4.3 (ALUM).

Coagulant dosage $\times 10^{-3}$ (m^3/kg)	K_m ($\text{m}^3/\text{kg}\cdot\text{S}$)	Rate Equation (-r)	R^2	β_{br} ($\text{m}^3/\text{kg}\cdot\text{S}$)	$\tau_{1/2}$ (S)	ϵ_p (kg^{-1})	K_R (m^3/S)	Reaction order
0.1	5×10^{-6}	$5 \times 10^{-6} N_t^2$	0.794	1×10^{-5}	195.5	6.475×10^{13}	1.544×10^{-19}	2
0.2	6×10^{-6}	$6 \times 10^{-6} N_t^2$	0.504	1.2×10^{-5}	162.9	7.715×10^{13}	1.556×10^{-19}	2



0.3	7×10^{-6}	$7 \times 10^{-6} N_t^2$	0.571	1.4×10^{-5}	139.6	8.991×10^{13}	1.557×10^{-19}	2
0.4	9×10^{-6}	$9 \times 10^{-6} N_t^2$	0.205	1.8×10^{-5}	108.6	1.156×10^{14}	1.557×10^{-19}	2
0.5	8×10^{-6}	$8 \times 10^{-6} N_t^2$	0.167	1.6×10^{-5}	122.2	1.022×10^{14}	1.558×10^{-19}	2
0.6	1×10^{-6}	$1 \times 10^{-5} N_t^2$	0.389	2×10^{-5}	97.8	1.283×10^{14}	1.559×10^{-19}	2

Table 4: Coagulation-Flocculation functional parameters for a second order system at pH 11.3(MSS).

Coagulant dosage $\times 10^{-3}$ (m ³ /kg)	K _m (m ³ /kg.S)	Rate Equation (-r)	R ²	β_{BR} m ³ /kg.S	$\tau_{1/2}$ (S)	ϵ_p (kg ⁻¹)	K _R m ³ /S	Reaction order
0.1	8×10^{-6}	$8 \times 10^{-6} N_t^2$	0.7932	1×10^{-5}	166.67	1.033×10^{14}	1.549×10^{-19}	2
0.2	1×10^{-6}	$1 \times 10^{-6} N_t^2$	0.776	2×10^{-5}	133.33	1.289×10^{14}	1.551×10^{-19}	2
0.3	2×10^{-6}	$2 \times 10^{-6} N_t^2$	0.856	1.4×10^{-5}	68.67	2.579×10^{14}	1.551×10^{-19}	2
0.4	3×10^{-6}	$3 \times 10^{-6} N_t^2$	0.807	1.8×10^{-5}	44.44	3.862×10^{14}	1.554×10^{-19}	2
0.5	8×10^{-6}	$8 \times 10^{-6} N_t^2$	0.906	1.6×10^{-5}	33.33	5.146×10^{14}	1.554×10^{-19}	2
0.6	4×10^{-6}	$4 \times 10^{-5} N_t^2$	0.851	2×10^{-5}	66.67	2.572×10^{14}	1.555×10^{-19}	2

Table 5: Coagulation-Flocculation functional parameters for a second order system at pH 4.3 (ALUM).

Coagulant dosage $\times 10^{-3}$ (m ³ /kg)	K _m (m ³ /kg.S)	Rate Equation (-r)	R ²	β_{BR} m ³ /kg.S	$\tau_{1/2}$ (S)	ϵ_p (kg ⁻¹)	K _R m ³ /S	Reaction order
0.1	3×10^{-6}	$3 \times 10^{-6} N_t^2$	0.657	1.6×10^{-5}	444.44	3.859×10^{14}	1.555×10^{-19}	2
0.2	8×10^{-6}	$8 \times 10^{-6} N_t^2$	0.706	1.4×10^{-5}	166.67	1.094×10^{14}	1.556×10^{-19}	2
0.3	7×10^{-6}	$7 \times 10^{-6} N_t^2$	0.725	1.4×10^{-5}	190.48	8.994×10^{13}	1.557×10^{-19}	2
0.4	7×10^{-6}	$7 \times 10^{-6} N_t^2$	0.290	1.8×10^{-5}	190.48	8.980×10^{13}	1.559×10^{-19}	2
0.5	1×10^{-6}	$1 \times 10^{-6} N_t^2$	0.793	2×10^{-5}	133.33	1.283×10^{14}	1.559×10^{-19}	2
0.6	1×10^{-6}	$1 \times 10^{-5} N_t^2$	0.810	2×10^{-5}	133.33	1.282×10^{14}	1.560×10^{-19}	2

Conclusion

The ability of MSS coagulant to achieve 82% TDSP removal after 30 minutes of coagulation-flocculation, presents it as an effective natural organic coagulation – flocculants for wastewater treatment. The coagulation-flocculation process were found to be influenced by settling time, coagulant dosage, pH (of sample), and reaction rate constant. The optimum dosage, pH and $\tau_{1/2}$ recorded are $0.4 \times 10^{-3} \text{kg/m}^3$, 11.3 and 33.33S respectively after 30 minutes of coagulation – flocculation. In general, the results obtained are in accordance with previous works [10, 13-14, 19].

Abbreviation

TDSP	:	Total Dissolved solid particles
MSS	:	Mucuna Seed Coagulant
(-r)	:	Rate of depletion of TDSP
N_o	:	Initial concentration of the dissolved solid particles
N_t	:	final concentration of the dissolved solid particles
K _m	:	MenkonuCoag – flocculation rate constant
PHE	:	Pharmaceutical Effluent
β_{BR}	:	Collision factor for Brownian Transport
α	:	Coagulation-Flocculation reaction order
ϵ_p	:	Collision Efficiency
$\tau_{1/2}$:	Coagulation period / Half Life
R ²	:	Coefficient of Determination



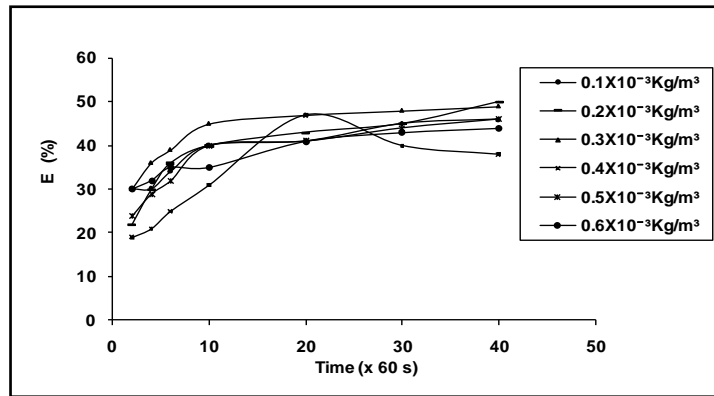


Figure 1: E (1%) VS Coag-flocculation time for MSS at pH 4.3

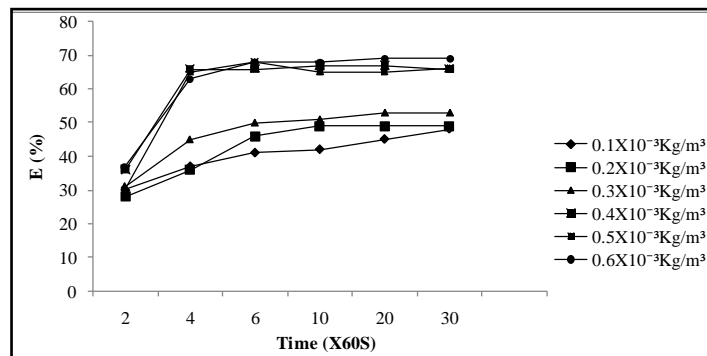


Figure 2: E1%VS Coag-flocculation time for Alum at pH 4.3

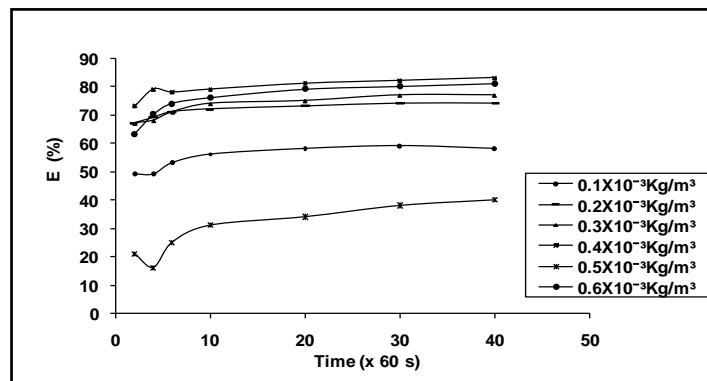


Figure 3: E1%VS Coag-flocculation time for MSS at pH 11.3 (Experimental initial concentration No=1500 mg/l)

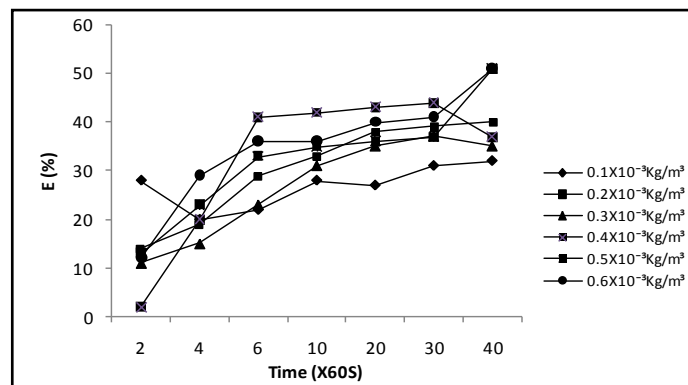


Figure 3: E1%VS Coag-flocculation time for Alum at pH 11.3 (Experimental initial concentration No=1500 mg/l)

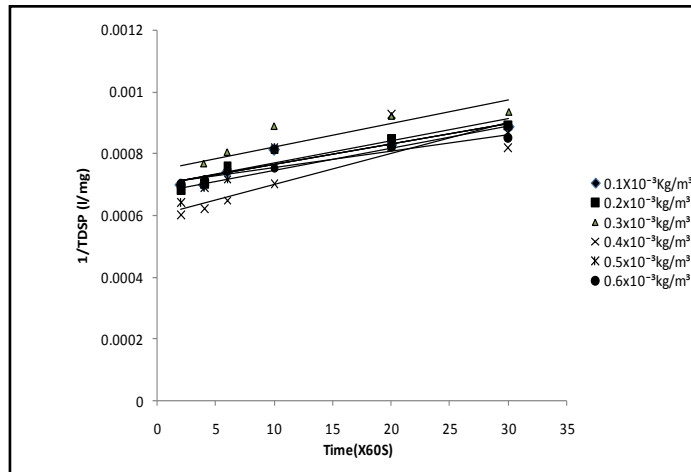


Figure 5: Plot of 1/TDSP Vs time for MSS at pH 4.3

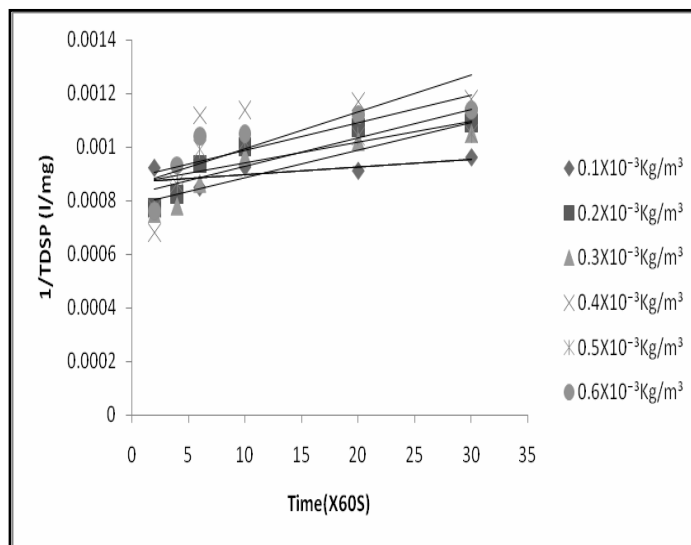


Figure 6: Plot of 1/TDSP Vs time for Alum at pH 4.3

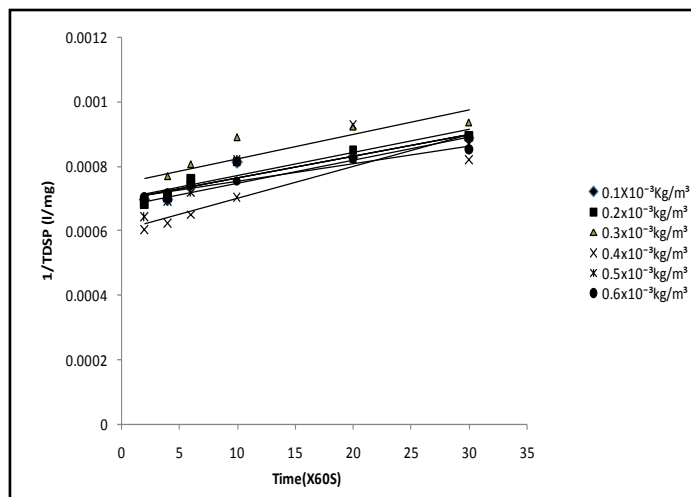


Figure 7: Plot of 1/TDSP Vs time for MSS at pH 4.3

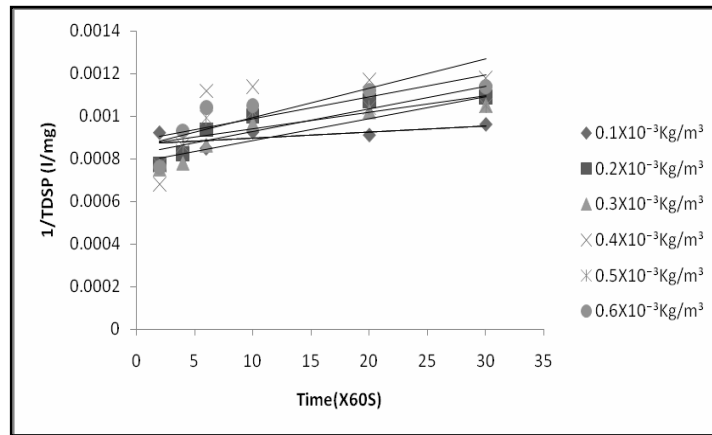


Figure 8: Plot of 1/TSDP Vs time for Alum pH 11.3

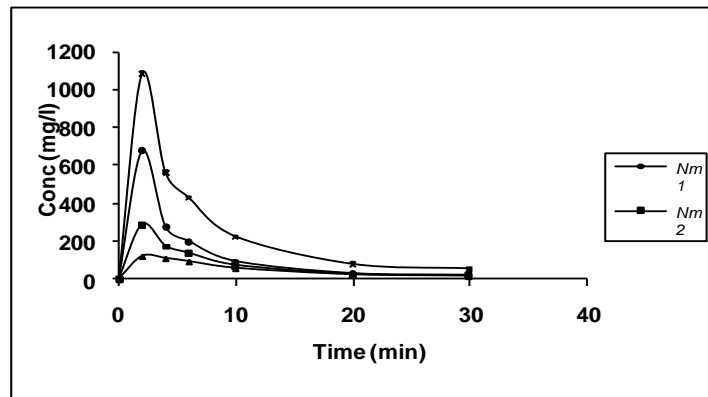


Figure 9: Theoretical particle distribution plot for MSS at pH 4.3

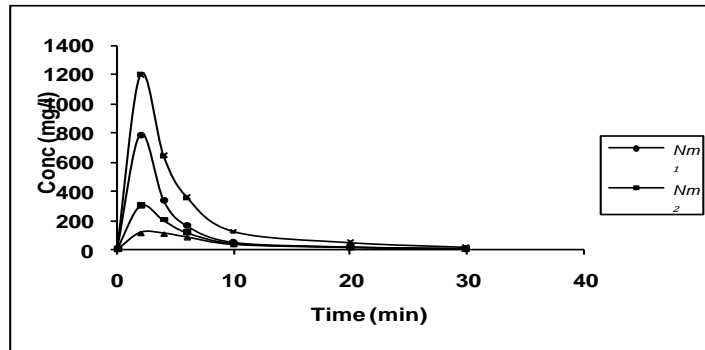


Figure 10: Theoretical particle distribution plot for Alum at pH 4.3

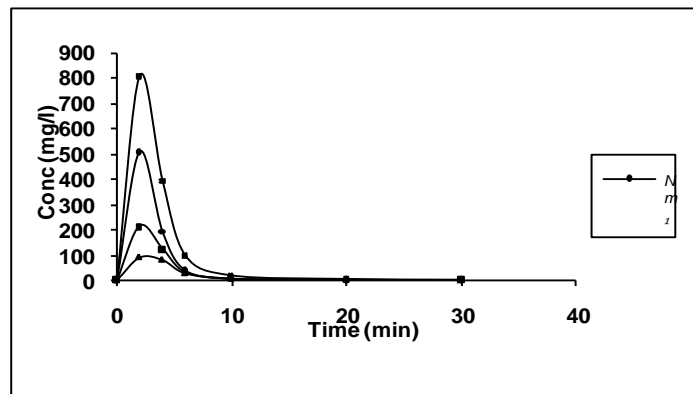


Figure 11: Theoretical particle distribution plot for MSS at pH 11.3

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