

Factorial Optimization and Kinetics of Coal Washery Effluent Coag-Flocculation by *Moringa Oleifera* Seed Biomass

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Abstract

Factorial optimization and kinetics of coal washery effluent (CWE) coag-flocculation by *Moringa oleifera* seed has been investigated at room temperature based on standard method of bench scale jar test. *Moringa oleifera* coag-flocculant (MOC) was produced according to work reported by Ghebremichael. A 2^3 full factorial central composite design was employed for the experimental design and analysis of results with respect to optimization. The combined effects of pH, dosage and settling time on the particle (turbidity) removal were studied using response surface methodology. Kinetic data generated were confronted with specified kinetic models for the evaluation of functional kinetics parameters. The optimal values of pH, dosage and settling time were recorded at 8400 mg/l and 25 min, respectively. The results of the major kinetic parameters recorded are 20.002 l/mg·min, and 0.79 min for order of reaction, coag-flocculation reaction rate constant and coagulation period, respectively. The minimum removal efficiency recorded was 95% at 3 mins of coag-flocculation. The results, while re affirming MOC as efficient coag-flocculant, confirmed that theory of perikinetics holds for the studied system at the conditions of the experiment.

Keywords: *Moringa Oleifera*, Effluent, Coagulation/Flocculation, Optimization

1. Introduction

Coag-flocculation process is an established technology for the protection of environmental and human health with wide applications in water and waste water treatment facilities. Coag-flocculation is a core and usually the first unit process in water treatment and it is very important for the removal of suspended and dissolved particles (SDP). It is the act of destabilizing stable colloidal particles in suspension, such that they can agglomerate into settleable flocs. Readily, coag-flocculation is optimized for the removal of inorganic colloids, dissolved natural organic load, microbes and color which are typical composition of coal washery effluent [1-4]. Conventionally, coag-flocculation treatment technique entails the use of metal salts (Al and Fe salts) and synthetic organic polymers. Although the chemicals are very effective and widely used, there are inherent draw backs: they impact on the pH value of water, increase the solu-

ble residues volume and metal content of sludge. With Alum, there is risk of Alzheimer's disease and similar health related problems [5-7]. Obviously, the issues of cost and availability of the chemicals are major drawbacks since they have to be imported in hard currency.

In order to alleviate the prevailing challenges, approaches should focus on sustainable water treatment that are low cost, eco-friendly, robust and require minimal maintenance and operator skills. MOC among other natural materials such as *Brachestegia eurycoma*, *Afzelia bella* and *Mucuna* seeds possess these qualities and provide the required remedy for the identifiable deficiencies associated with non natural coagulants [8]. The *Moringa oleifera* is a small, fast growing drought deciduous tree that ranges in height from 5 - 12 m. The seed kernel contains positively charged water soluble proteins that act like magnets and attract the predominantly negatively charged particles (SDP) to form settleable flocs [9].

In recent years, there has been increasing advocacy for the research in and the use of natural coag-flocculants such as MOC, as an alternative to the synthetic ones, especially in developing country like Nigeria where portable water supply is highly limited. However, the major challenges have been the availability of research data that will promote the design of mini flocculator suitable for handling effluents such as CWE usually discharge into drinking aquifers of our communities. The focus is therefore directed towards the provision of kinetic data, the mathematical relationship that predicts the interaction of studied variables and the optimal values of the variables. The situation in Nigeria is typical of water system in developing countries and the results of this study can be applied to a number of similar situations in order to improve the quality of water supply and protect the environment.

2. Materials and Method

2.1. Material Collection, Preparation and Characterization

2.1.1. Coal Washery Effluent

The effluent used in this study was taken from the coal washery pond of moribund coal mine located in Akwuke, Enugu State, Nigeria. The physicochemical and biological characteristics of the effluent presented in **Table 1** were determined based on standard method [10].

2.1.2. Moringa Oleifera (MO) Sample

Dry *Moringa oleifera* (precursor to MOC) were sourced from Agulu, Anambra State, Nigeria and stored at room temperature. MOC was obtained as a by product of oil extraction procedure reported by [3]. The analysis of MO seed powder were performed by standard method [11] and the characteristics presented in **Table 2**.

2.2. Coag-Flocculation Experiment

Experiment were carried out in a conventional jar-test apparatus equipped with a six-unit multiple stirrer system. Appropriate dosage of MOC in the range 100 - 500 mg/l was added directly to 200 ml of CWE. The suspension, tuned to pH range of 2 - 10 using H₂SO₄ and NaOH were subjected to 2 minutes of rapid mixing (250 rpm), 20 min of slow mixing (40 rpm), followed by 30 minutes of settling. During settling, samples were withdrawn using pipette from 2 cm depth and analyzed for turbidity (converted to SDP in mg/l) changes with a view to determining optimal conditions (pH, dosage, settling time via 2³-CCD) and kinetics parameters. Independent variables range and levels for the coag-flocculation process optimization are given in **Table 3** while **Table 4** displays the full 2³-CCD factorial design matrix with output response.

Table 1. Characteristics of coal washery effluent.

Parameters	Values
pH	2.5200
Turbidity (NTU)	5387.0000
Total hardness (mg/l)	358.0000
Ca hardness (mg/l)	306.0000
Mg hardness (mg/l)	52.0000
Ca ²⁺ (mg/l)	122.4000
Mg ²⁺ (mg/l)	15.6000
Fe ²⁺ (mg/l)	0.2500
SO ₄ ²⁻ (mg/l)	72.0000
NO ₃ ²⁻ (mg/l)	Nil
Cl ⁻ (mg/l)	184.3400
E.cond (µm/m ²)	805.2000
TDS (mg/l)	450.9120
TSS (mg/l)	109.6000
T. Coliform	Nil
Plate Count	4.0000s
E-Coli	Nil
BOD5	1001.0110

Table 2. Characteristics of MOC precursor.

Parameter	MOC
Moisture content (%)	2.0200
Ash content (%)	2.1200
Lipid content (%)	30.4700
Crude protein (%)	39.3400
Carbohydrate (%)	23.7100
Crude fibre (%)	2.1600

Table 3. Experimental range and levels of independent process variables.

Independent Variable	Lower limit (-1)	Base level (0)	Upper limit (+1)
pH	2.0000	6.0000	10.0000
Dosage	100.0000	300.0000	500.0000
Settling time	10.0000	20.0000	30.0000

The experimental results of the 2³-CCD were studied and interpreted by software, MATLAB 7.0 to estimate the response of the dependent variable. The kinetics of coag-flocculation and extent of aggregation were monitored at optimal conditions at 3,5,10,15,20,25 and 30 min. The data were subsequently fitted in appropriate kinetic model. The experiments were carried at room temperature.

3. Theory

3.1. Coag-Flocculation Optimization

Optimization was studied specifically by central com-

Table 4. Process design matrix and output response.

S/NO	X ₁	X ₂	X ₃	Y ₁	Y ₂
1	0	0	0	163.71	164.01
2	-1	-1	-1	2045.67	2055.13
3	1	-1	-1	514.65	513.9
4	-1	1	-1	2134.97	2135.33
5	1	1	-1	467.56	460.05
6	0	0	0	165.86	163.81
7	-1	-1	1	1615.32	1613.33
8	1	-1	1	261.31	262.25
9	-1	1	1	847.24	849.31
10	1	1	1	195.51	197.71
11	0	0	0	164.73	164.67
12	-1	0	0	1386.5	1390.1
13	1	0	0	274.16	276.61
14	0	-1	0	157.48	158.84
15	0	1	0	232.65	235.51
16	0	0	-1	250.71	251.12
17	0	0	1	113.16	115.34

posite design (CCD). The parameters: pH, dosage and settling time were chosen as independent variables at two levels while particle (SDP) uptake is the output response. A 2³ full factorial experimental designs with three star points, six centre points and two replications generated 34 experiments employed in this study. The centre points replicates verify changes in the middle of the plan and measures of the degree of precision property, while star points verify the non linear suspected curvature. The behavior of the systems is explained by the multivariable polynomial equation presented below:

$$Y = b_0 + b_1X_1 + b_2X_2 + b_3X_3 + b_{12}X_1X_2 + b_{13}X_1X_3 + b_{23}X_2X_3 + b_{11}X_1^2 + b_{22}X_2^2 + b_{33}X_3^2 \tag{1}$$

X₁ is pH, X₂ is dosage, X₃ is settling time.

Upon the determination of polynomial coefficients (b₀, b₁, b₃₃ e.t.c.) by the following relationships expressed below, statistical analysis (CSI, G-test, F-test, T-test e.t.c) were performed to developed model that is adequate, significant and homogenous (variance wise) [12].

$$b_0 = a \sum_{u=1}^N Y_u + P \sum_{j=1}^M \sum_{u=i}^N X_{ju}^2 \tag{2}$$

$$b_i = e \sum_{u=1}^N X_{iu} Y_u \tag{3}$$

$$b_{ij} = g \sum_{u=1}^N X_{iu} X_{ju} Y_u \tag{4}$$

$$b_{ii} = c \sum_{u=1}^N X_{iu}^2 Y_u + d \sum_{j=1}^M \sum_{u=i}^N X_{ju}^2 + P \sum_{u=i}^N Y_u \tag{5}$$

where a(0.40625), e(0.10), g(0.125), c(0.40625), d(-0.093750), P(-0.15625)

3.2. Coag-Flocculation Kinetic

For a coag-flocculating phase, the rate of successful collision between particles of sizes i and j to form particle of size k is [13-16].

$$\frac{dn_k}{dt} = \frac{1}{2} \sum_{i+j=k} \beta_{BR}(i,j)n_i n_j - \sum_{i=1}^{\alpha} \beta_{BR}(i,k)n_i n_k \tag{6}$$

where β_{BR}(i,j) is Brownian aggregation factor for flocculation transport mechanism, n_in_j is particle aggregation concentration for particles of size i and j, respectively. It has been established that [15-17].

$$\beta_{BR} = \frac{8}{3} \varepsilon_p \frac{K_B T}{\eta} \tag{7}$$

and

$$K_R = 8\pi a D' \tag{8}$$

where K_R is the Von Smoluchowski rate constant for rapid coagulation. K_B, T and η are Boltzmann constant, temperature and viscosity, respectively. ε_p is collision efficiency factor, D' is the diffusion coefficient and a is particle radius.

Equations (7) and (8) can be transformed to

$$\frac{1}{2} \beta_{BR} = K_m \tag{9}$$

where K_m is defined as Menkonu coag-flocculation rate constant accounting for Brownian coag-flocculation transport of destabilized particles at αth order. It can also be shown that coag-flocculation is governed by [18-21].

$$-\frac{dN_t}{dt} = \varepsilon_p K_R N_t^\alpha \tag{10}$$

where ε_p K_R = 0.5 β_{BR}

Thus

$$-\frac{dN_t}{dt} = K_m N_t^\alpha \tag{11}$$

N_t is the concentration of SDP at time, t.

Empirical evidence shows that in real practice, 1 < α < 2 [8,22-26]. Graphical representation of linear form of Equation (11) at α = 2 provides for K_m from the slope of linear equation below:

$$\frac{1}{N} = K_m t + \frac{1}{N_0} \tag{12}$$

where N₀ is the initial N_t at time = 0; N is N_t at upper time limit > 0

Equation (12) can be solved to obtain coag-flocculation period, τ_{1/2}:

$$\tau_{1/2} = (0.5N_0K_m)^{-1} \tag{13}$$

Equation (6), solved exactly, results in generic expression for microscopic aggregation:

$$\frac{N_{m(t)}}{N_0} = \frac{\left[\frac{1}{\tau_{1/2}} \right]^{m-1}}{\left[1 + \frac{t}{\tau_{1/2}} \right]^{m+1}} \tag{14}$$

$m = 1$ (monomers), $m = 2$ (dimers), $m = 3$ (trimers)

Efficiency of coag-flocculation is expressed as:

$$E(\%) = \left[\frac{N_0 - N_t}{N_0} \right] 100 \tag{15}$$

4. Results and Discussion

4.1. Optimization Studies

The optimization of the coag-flocculation process with respect to pH, dosage and settling time was achieved by response surface methodology via 2³-CCD. The analysis focused on how the SDP uptake (dependent output variable) is influenced by independent variables, *i.e.* CWE pH, (X₁) coag-flocculant dosage (X₂) and settling time (X₃). The pH range studied was between 2 and 10, dosage varied between 100 and 500mg/l and settling time in the range of 10 to 20 minutes as shown in **Table 3**. In order to study the combined effect of these factors, experiments were performed at different combinations of the physical parameters using statistically designed (design matrix shown in **Table 4**) experiments. The main effects of the parameters and response behavior of the system was explained by Equation (16) shown below:

$$Y_u = 486.04 - 1.62X_1 + 7.05X_2 + 0.92X_3 - 9.69X_1X_2 + 2.89X_1X_3 + 2.85X_2X_3 - 24.04 X_1^2 + 4.62 X_2^2 + 0.57 X_3^2 \tag{16}$$

The optimization results obtained from the Equation (16) as interpreted by MATLAB 7.0 are presented in **Table 5**. With the objective of minimizing SDP, the optimal pH, dosage and settling time were recorded at 8,400 mg/l and 25 min, respectively. It can be deduced that at optimal operation, the SDP was reduced from

12657.85 mg/l to 65.0587mg/l. This translates to about 99.48% SDP removal from the CWE. The corresponding optimized interactive surface response plots are presented in **Figures 1-3**. **Figure 1** shows the interaction effects of pH and dosage on the SDP removal. In respect of **Figure 2**, the interaction effect of pH and settling time is presented while **Figure 3** shows the interaction effect of settling time and dosage. It is pertinent to point out that the value of output responses are tied to the intensity of the color of the 3-D plots. Hence, the optimal values recorded for **Figures 1-3** are 80, 80 and 75 mg/l, respectively. In general, the 3-D plots provide avenue to observe the surface area of the curve within which the process can perform at optimal level based on the effects of the interaction of the variables under consideration. The significance of these interaction effects between the variables would have been lost if the experimental were carried out by conventional methods of analysis.

Coag-flocculation efficiency E, (%) calculated from Equation (15) is graphically depicted as **Figure 4**. It shows the temporal variation of SDP removal at varying dosage and optimum pH 8 and 25 minutes settling time. It is apparent that at 3 minutes, all the dosages had achieved up to 95% efficiency. The dosage with best performance is 400 mg/l, at E, (%) > 98%. This is in agreement with result recorded in **Table 5**. Furthermore, the performance of MOC was compared to that of alum as shown in **Figure 5** at optimum pH and settling time. Apparently, MOC performed better than alum for all the dosages considered. This re-affirms the existing assertion that MOC is a highly efficient coag-flocculant that are eco-friendly [3,27].

4.2. Coag-Flocculation Kinetics

A summary of the coag-flocculation functional parameters at optimum conditions as determined in this study is shown in **Table 6** for varying dosages. The accuracy of the fit of the studied model (Equation (12)) with the experimental data was based on squared linear regression coefficient (R²). **Table 6** indicates that experimental data (with R² > 0.90) were significantly described by the linearised form of Equation (12). K_m is determined from the slope of Equation (12) on plotting 1/N Vs. time. The results posted in **Table 6** indicate that K_m (and β_{BR}) are in-

Table 5. Optimization results of CWE coag-flocculation based on 2³ CCD.

Sample	X ₁ (pH)		X ₂ (Dosage)		X ₃ (Settling time)		Y (SDP removal) (mg/l)
	CV*	RV**	CV*	RV** (mg/l)	CV*	RV** (min)	
MOC	0.500	8.0000	0.5000	400.0000	0.5412	25.4120	65.0587

*Coded value
**Real value

Table 6. Coag-flocculation kinetic parameters of MOC in CWE @varying dosage and pH of 8.

Parameters	100 mg/l	200 mg/l	300 mg/l	400 mg/l	500 mg/l
Y	$0.0002 X + 0.0018$	$0.0002 X + 0.001$	$0.0002X + 0.0018$	$0.0001X + 0.0018$	$0.0001X + 0.0015$
α	2.0000	2.0000	2.0000	2.0000	2.0000
R^2	0.9692	7420.0000	0.9194	0.9701	0.9096
$K_m \left(\frac{1}{\text{mg}\cdot\text{min}} \right)$	0.0002	0.0002	0.0002	0.0002	0.0002
$\beta_{BR} (1/\text{mg}\cdot\text{min})$	0.0004	0.0004	0.0004	0.0004	0.0004
$K_R (1/\text{min})$	7.0819×10^{-12}	7.5095×10^{-12}	7.70909×10^{-12}	7.5829×10^{-12}	7.6525×10^{-12}
$\epsilon_P (1/\text{mg})$	5.6481×10^7	5.3265×10^7	5.1887×10^7	5.2749×10^7	5.2269×10^7
$\tau_{1/2} (\text{min})$	0.7900	0.7900	0.7900	0.7900	0.7900
$N_0 (\text{mg/l})$	555.5600	1000.0002	555.5600	555.5600	666.6700
$(Np)_0$	3.3455×10^{23}	6.022×10^{23}	3.3455×10^{23}	3.3455×10^{23}	4.0146×10^{23}

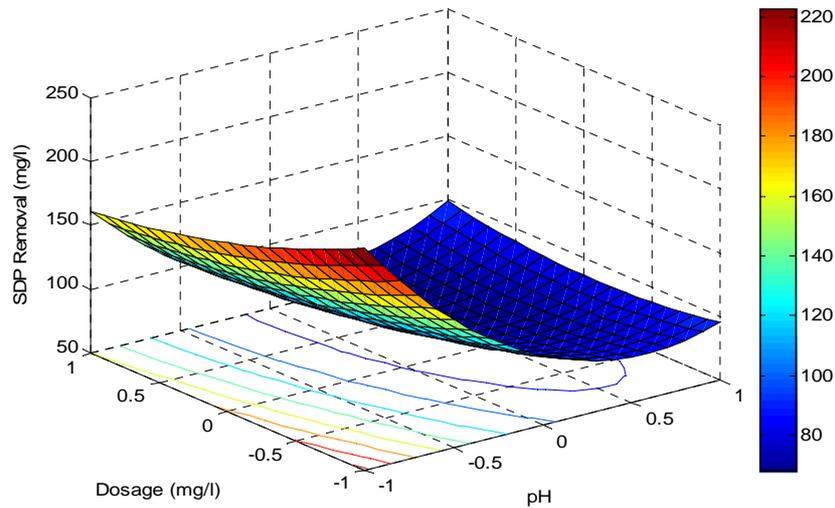


Figure 1. Coag-flocculation surface/contour plots of MOC in CWE showing interaction effects of pH and dosage.

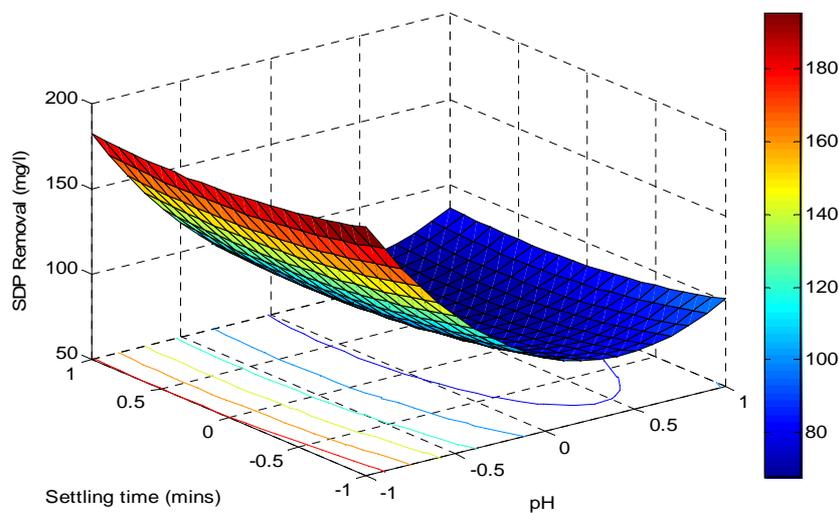


Figure 2. Coag-flocculation surface/contour plots of MOC in CWE showing interaction effects of pH and settling time.

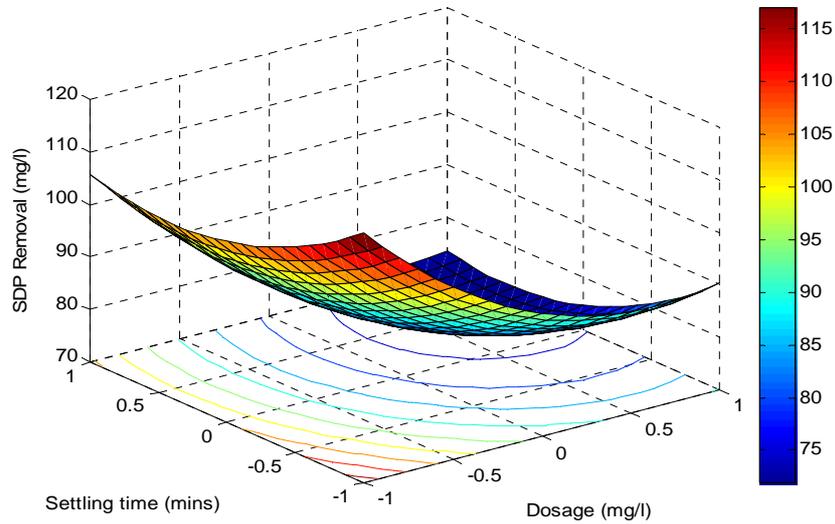


Figure 3. Coag-flocculation surface/contour plots of MOC in CWE showing interaction effects of Dosage and settling time.

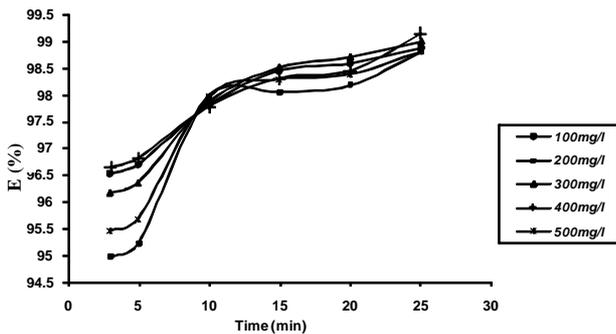


Figure 4. Coag-flocculation efficiency profile for varying MOC dosage in CWE at Ph of 8.

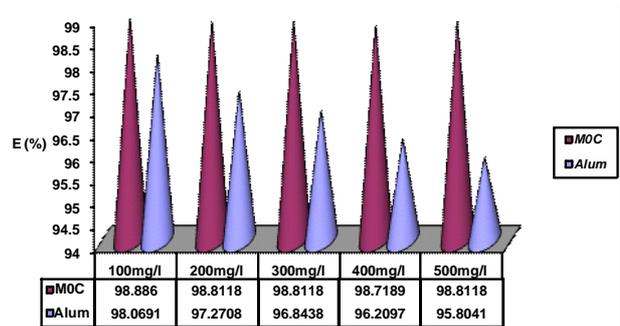


Figure 5. Comparative coag-flocculation performance at 25 mins for varying MOC and Alum dosage in CWE at Ph of 8.

variant with dosage variation. This may be explained by the near equal efficiency (equal aggregation rate) achieved by the varying MOC dosages depicted in Figure 4.

Generally, the variation of $K_R = f(n, T, \eta)$ is minimal, following insignificant changes in the values of temperature and viscosity of the effluent medium. At approximately invariant K_R , ϵ_p relates directly to $2K_m = \beta_{BR}$. Thus high ϵ_p results in high kinetic energy to overcome the repulsive forces. The coagulation period, $\tau_{1/2}$ is determined from Equation (13). Here, $\tau_{1/2} = f(n, N_0)$. The higher the N_0 , the smaller the $\tau_{1/2}$. This accounts for high settling rate associated with highly turbid water. From theoretical point of view, $\tau_{1/2}$, ϵ_p and K_R are considered as effectiveness factor, understood to be accounting for the coag-flocculation efficiency before flocculation sets in.

Equation (14) predicts the time evolution of aggregation (monomers, dimmers, trimmers for $m = 1, 2, 3$ respectively) at microscopic/discrete level. The aggregations profile as a function of time are depicted in Figure 6.

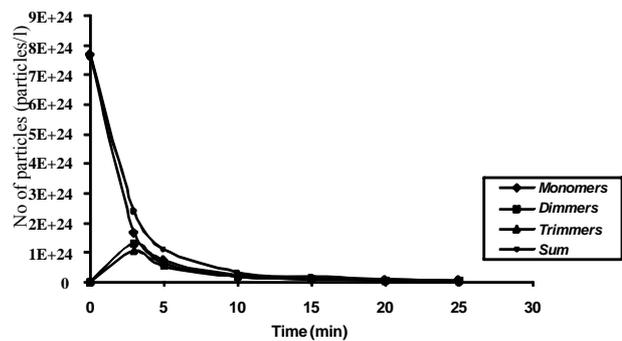


Figure 6. Time evolution of the cluster size distribution for MOC in CWE at minimum half life of 0.79 min.

The primary particles (monomers) and total number of particles can be seen to decrease more rapidly. This can be accounted on the basis of sweep-floc or massive instantaneous destabilization of the particles. With negligible repulsion in the system, the MOC sweeps away the SDP from the CWE [8].

5. Conclusions

The application of MOC as an effective coag-flocculant in the treatment of high turbid effluent such as CWE has been established. The removal of 90% of initial value of SDP within the first three minutes of treatment justifies that the process was rapid with high rate constant and low coagulation period. The system can operate optimally at pH 8, 400 mg/l dosage and 25 min settling time.

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