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The Performance of Okra seed (*Hibiscus esculentus* L.) Extract in Removal of Suspended Particles from Brewery Effluent by Coag-Flocculation Process

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Authors' contributions

This work was carried out in collaboration between all authors. Authors BIO, PCN and MCM designed the study, performed the statistical analysis, wrote the protocol, and wrote the first draft of the manuscript and managed literature searches. Authors ODO and CCA managed the analyses of the study and literature searches. All authors read and approved the final manuscript.

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ABSTRACT

This work investigated the performance of Okra seed (*Hibiscus esculentus* L.) extract in removal of suspended dissolved particles from brewery effluent by coag-flocculation process at room temperature using various doses of okra seed extract. The study was carried out using nephelometric jar test. The perikinetic data generated were fitted to specific models for the determination of reaction order, rate constant, coagulation period. The maximum coag-flocculation performance is recorded at rate constant, K_m of 3.6×10^{-3}L (mg.min)$^{-1}$, dosage of 100 mg L^{-1}; pH of 6 and coagulation period of 0.38 min while the minimum is recorded at K_m of 2.95×10^{-5}L (mg.min)$^{-1}$, dosage of 500 mg L^{-1} , pH of 10 and period of 93 min. The optimum efficiency recorded at 92.6% for 30 min shows that the system is perikinetic controlled. The results indicate that okra extract can serve as a potential coagulant for the treatment of brewery effluent.

Keywords: Okra seed; brewery effluent; coag-flocculation; suspended dissolved particles; perikinetics.

NOMENCLATURE

K_m: Menkonucoag-flocculation rate constant; K: Coagulation-flocculation reaction rate constant; K_R: Smoluchowski coag-flocculation constant; β_{BR}: Collision factor for Brownian Transport; ε_p: Collision Efficiency; $\tau_{1/2}$: Coagulation Period/Half life; E: Coag-flocculation Efficiency; R^2: Coefficient of Determination; α: Coag-flocculation reaction order; -r: Coag-flocculation reaction rate; OSC: Okra Seed Coagulant; BRE: Brewery effluent; SDP: Suspended Dissolved Particles; N_o= Concentration of SDP at time t=0; N_t= Concentration of SDP at time, t.

1. INTRODUCTION

There has been an increasing demand of water usage in brewery industry; as a result, a large volume of wastewater is being generated. In brewery industry, the main section that consumes much water is the brew house, cellars and packaging [1].

The large quantity of wastewater produced from brewery industry contains significant amount of pollutants that usually pose serious risk to human beings, environment, and aquatic life if not properly treated prior to disposal. There are several ways to treat this wastewater. Coagulation-flocculation process is widely used for the treatment of water and wastewater. In this process, three important steps are considered: coagulant formation, colloid particles destabilization and inter particle collision and aggregation [2,3,4,5].

The coagulation/flocculation process involves the addition of inorganic/organic coagulants such as Aluminum sulphate, polyamine and *Xanthosoma spp.* When added in wastewater, they neutralize the negative charges in the dispersed non-settleable solids, resulting in the formation of micro flocs [6].

Since Alzheimer disease is associated with Aluminum sulphate, coupled with significant generation of recalcitrant sludge ,there is need to create a platform for the use of natural coagulant which are eco-friendly and locally abundant. Natural coagulants are promising and have attracted the attention of many researchers because of their abundance, low price and biodegradability [7,8,9,10].

Okra is a perennial shrub plant, which can reach three meters in height. It belongsto the family of Malvaceae, and is given the scientific name of *Hibiscus esculentus* L. and *Abelmoschus esclentus*. It is a crop of fast vegetative cycle and high yield [11]. Okra seed is mainly composed of oligomeric catechins (2.5 mg g^{-1} of seeds) and flavonol derivative (3.4 mg g^{-1} of seeds), while the mesocarp is mainly composed of hydroxycinnamic and quercetin derivatives (0.2 and 0.3 mg $g^{-1}$of skins). Pods and seeds are rich in phenolic compounds with important biological properties like quartering derivatives, caechin oligomers and hyhroxycinnamic derivatives [12,13]. Okra powder being rich in proteins, fat and polysaccharides shows a promising flocculation properties, an indication of the availability of active sites capable of adsorbing colloids during coagulation-flocculation process [14].

The objective of the current work is to investigate the natural coagulant (okra seed extract) using coagulation-flocculation processes on the removal of suspended dissolved particles from brewery effluent for a range of parameters (pH, doses, time).

2. COAGULATION THEORY AND MODEL DEVELOPMENT

2.1 The Theory of Rapid Coagulation

For an aqueous coagulating suspension, where Brownian particle collision is in force, the number of collision occurring per unit time per unit volume, K_{ij}, for two species of particles of volumes $v_i$and v_j is expressed as [15,16,17].

$$K_{ij} = \beta_{BR}(v_i v_j) n_i n_j \tag{1}$$

Where $\beta_{BR}(v_i v_j)$ is collision frequency
$n_i n_j$ is the number of concentration of particles

The formation rate r_f of particles of volume V_k, resulting from collisions of volumes V_i and V_j is given by:

$$r_f = {}^1\!/_2 \sum_{i+j=k} K_{ij} \tag{2}$$

The summation of *i +j* implies an overall collision for which

$$V_k = V_i + V_j \tag{3}$$

Conversely, the rate of particle loss by collision is

$$r_l = \sum_{i=1}^{\infty} K_{ik} \tag{4}$$

Hence the rate of change due to Brownian motion in the fluid medium is:

$$\frac{dn_k}{dt} = {}^1\!/_2 \sum_{i+j=k} K_{ij} - \sum_{i=1}^{\infty} K_{ik} \tag{5}$$

Putting equation 1 into equation 5, yields

$$\frac{dn_k}{dt} = {}^{1}/_{2} \sum_{i+j} \beta_{BR}\,(V_iV_j)n_in_j - n_k \sum_{i=1}^{\infty} \beta_{BR}\,(V_iV_k)n_i \tag{6}$$

For kernel like particles,

$$K_{ij} = 4\pi\left(D_i + D_j\right)\left(a_i + a_j\right) \tag{7}$$

Also,

$$K_{ij} = {}^{2}/_{3}\left(\frac{K_BT}{\eta}\right) \tag{8}$$

Combining equations 7 and 8 yields

$$\left(D_i + D_j\right) = \frac{K_BT}{6\pi\eta}\left(\frac{1}{a_i} + \frac{1}{a_j}\right) \tag{9}$$

Putting ($D_i + D_j$) of equation 9 into equation 7 yields

$$K_{ij} = \frac{2}{3}\frac{K_BT}{\eta}\frac{\left(a_i+a_j\right)^2}{a_ia_j} \tag{10}$$

For particles of difference radius with dimmer formation of an initially monodispersed suspension:

$$\frac{\left(a_i+a_j\right)^2}{a_ia_j} = \begin{pmatrix} 4\, for a i = a_j \\ >4\, for a_i \neq a_j \end{pmatrix} \tag{11}$$

Hence, for the case of $a_i = a_j$, equation 10 becomes:

$$K_{11} = \frac{8}{3}\frac{K_BT}{\eta} \tag{12}$$

Where K_{11} is the Smoluchowski coagulation rate constant.

If $K_{ij} \rightarrow K_{11} = \beta_{BR}\left(v_iv_j\right)n_in_j$, equation 6 solves exactly to yield:

$$\frac{N_t}{N_o} = \frac{\left(K_{11}N_o{}^{t}/_{2}\right)^{n-1}}{\left(1+K_{11}N_o{}^{t}/_{2}\right)^{n+1}} \tag{13}$$

It has been established that for aggregating system at n=1; extending into flocculation regime, such that $K_{11} \approx K_m$ [16] equation 13, transforms to equation 14:

$$\frac{1}{\sqrt{N_L}} = \left(\frac{K_mN_o}{\sqrt[2]{N_o}}\right)t + \frac{1}{\sqrt{N_o}} \tag{14}$$

Where $K_m = {}^{1}/_{2}\,\beta_{BR} = \frac{2}{3}\varepsilon_p\frac{K_BT}{\eta}$

$K_m$is defined as Menkonu coag-flocculation rate constant accounting for Brownian coag-flocculation transport of destabilized particle at α^{th} order, $\varepsilon_p$is collision efficiency

From equation 13, coag-flocculation period is obtained as

$$\tau_{1/2} = \frac{1}{(0.5K_M N_o)} \tag{15}$$

There is, however, a reasonable relationship between Turbidity and total suspended solid for the settled effluent. The general form of the relationship is as follows:

$$\text{TSS (mg L}^{-1}\text{)} = \text{Tss}_f \text{ x (T)}$$

where TSS is total suspended solid, mg L^{-1}

Tss_f =2.3 is a factor used to convert turbidity reading to total suspended solids [18]
T is turbidity (NTU)

The concentration of singlets, doublets and triplets during coagulation can be evaluated by solving equation 13.

Hence, for singlets (n=1)

$$N_1 = N_o\left[\frac{1}{1+{}^{t}/_{\tau'}}\right] \tag{17}$$

For doublets (n=2)
.

$$N_2 = N_o\left[\frac{{}^{t}/_{\tau'}}{\left(1+{}^{t}/_{\tau'}\right)^3}\right] \tag{18}$$

For triplets (n=3)

$$N_3 = N_o\left[\frac{\left({}^{t}/_{\tau'}\right)^2}{\left(1+{}^{t}/_{\tau'}\right)^4}\right] \tag{19}$$

Where $\tau' = 2\tau$

Evaluation of coagulation –flocculation efficiency is given as

$$\text{E(\%)} = \left[\frac{N_o - N_t}{N_o}\right] \text{x100} \tag{20}$$

3. MATERIALS AND METHODS

3.1 Material Sampling, Preparation and Characterization

3.1.1 Brewery effluent

The effluent was collected from brewery plant located in Enugu, Enugu State Nigeria. The characterization of the effluent and the analysis presented in Table 1 were determined based on standard method [19].

Table 1. Characterization of brewery effluent

Parameters	Values
Temperature	27
pH	7.68
Turbidity (NTU)	316.63
Electrical Conductivity (µ/cm)	5290.0
Total hardness (mg L^{-1})	41.0
Ca hardness (mg L^{-1})	36.0
Mg hardness (mg L^{-1})	14.0
Ca^{2+} (mg L^{-1})	15.6
Mg^{2+}(mg^{-1})	0.6
Fe^{2+} (mg L^{-1})	0.178
So_4^{2-} (mg L^{-1})	46.224
No_3^{2-} (mg L^{-1})	0.178
Cl^- (mg L^{-1})	80.826
TDS (mg L^{-1})	3438.5
TSS (mg L^{-1})	30.406
E. coil	Nil
BOD_5 (mg L^{-1})	640

3.1.2 Okra seed sample

The Okra seed pods were bought from Ogbete market, Enugu State Nigeria. The Okra seed pods were prepared according procedure reported by Agarwal et al. [20]. The characteristics of okra seed were determined based on standard method AOAC [21]. This is presented in Table 2.

Table 2. Characterization of okra seed

Parameters	% values
Protein	23.0
Moisture content	12.0
Crude fiber	13.5
Fat content	11.0
Ash content	7.2
Carbohydrate	33.3

3.2 Coagulation-flocculation Experiment

Experiments were conducted using conventional jar test apparatus. Desired dosages of Okra coagulant between 100-500 mg L^{-1} were directly dosed into 300 mL of Brewery Effluent contained in standard 1000 mL beaker (G.C17). The suspension, tuned to pH 2-10 using H_2SO_4/NaOH, was subsequently subjected to 2 minutes of rapid mixing (200 rpm), 20 minutes of slow mixing (30rpm) using a magnetic stirrer. After agitation, the suspended solution was transferred into a cylinder (JayTec.Uk) of height 33.5cm at an angle so that the flocs will not break [22]. During settling, 10mL of the supernatant was withdrawn from 2 cm depth and changes in total turbidity particles (mg L^{-1}) measured at 3, 5, 10, 15, 20, 25 and 30 min.

4. RESULTS AND DISCUSSION

4.1 Plot of Efficiency E% vs Time, pH and Dosage

The removal efficiency E (%) with time, pH and dosage is obtained based on the evaluation of equation 20. This is presented in Figs. 1-7. This was obtained at 100,200,300, 400 and 500 mg L^{-1} dosage for pH 2, 4, 6, 8 and10. The time of macrofloc formation is one of the operating parameters that is given great consideration in any water treatment that involves coag-flocculation operation. The result presented in Figs. 1-7, shows that efficiency increases with increase in time, but the magnitude differs for particular pH and dosage. The efficiency at 3 minutes was between 15 and 72%, at pH 2 and 10 respectively. At 30 minutes, the least efficiency is more than 90%. This justified the theory of fast coagulation [15]. Also coagulation dosage is used to determine the optimum condition for the performance of the coagulant in coagulation/flocculation. The highest removal of suspended dissolved particles was 92.5% at 200 mg $L^{-1}$of okra at 30 minutes. From the Figs. 1-7, effective treatment was achieved with relatively lower doses, increasing the dosage from 100 to 500 mg L^{-1}, shows a decrease in SDP removal efficiency. This could be attributed to the excess polymer being adsorbed on the colloidal surface and producing restabilized colloids. From Figs. 1-7, shows that the effect pH on SDP removal decrease with increase in pH. The highest removal using 200 mg L^{-1} at pH 2 was 92.6%. It can be seen that the optimum range of pH is between 2 and 4. In the present work, it is recommended the pH 2 is the best. Good coagulation at a lower pH could be attributed to a higher degree of protonation of amino groups of the okra [10].

4.2 Nephelometric Kinetic Result

This test was performed for a sample of BRE with an initial SDP of 137.66 mg L^{-1} for 100-500 mg L^{-1} dosage of OSC dosage and pH 2-10 range, respectively.

Assuming perikinetic aggregation condition, where α=2, solving equation 13, gives equation 14, which is presented in the selected linear plots in Fig. 8. The plots of $\frac{1}{\sqrt{N_t}}$ Vs t yields, K_m and $\frac{1}{\sqrt{N_o}}$, as the slope and intercept, respectively. The results presented in Tables 3-7, show that majority of R^2values are high, which shows that the reaction is a second order.

Meanwhile, the value of$K_m = 0.5\beta_{BR}$ can be obtained from equation 15. However, the highest value of K_m of $3.6 \times 10^{-3} L(mg.\min)^{-1}$ for the process is recorded at pH of 6 and 100 mg L^{-1} OSC. The least of K_m of $2.95 \times 10^{-5} L(mg.\min)^{-1}$ is recorded at pH of 10 and 500 mg L^{-1}. In this study, the lowest $\tau_{1/2}$(0.38 min) is recorded at high $K_m\, of\; 3.6 \times 10^{-3}$L (mg.min)$^{-1}$ which favors fast coag-flocculation process [9]. Generally, K_m values were obtained from equation 14 and the representative kinetic plot presented in fig. 8. In the Tables 3-7, the variation of K_R is minimal, following insignificant changes in values of temperature and viscosity of the effluent medium. Using equation 14, the values of β_{BR}, $and\ \varepsilon_p$ were calculated. The coag-flocculation period, $\tau_{1/2}$ which indicates the time taken for the initial concentration of turbidity particles to reduce by half, is calculated from equation 15.

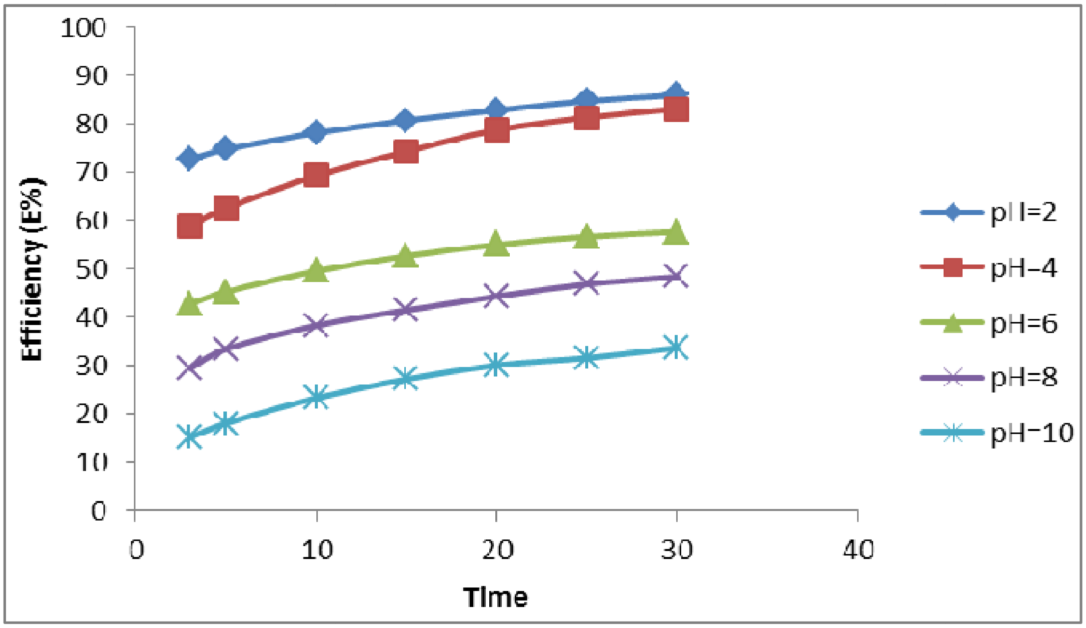

Fig. 1. Plot of efficiency (E %) vs coag-flocculation time for 100 mg L^{-1} okra at varying pH

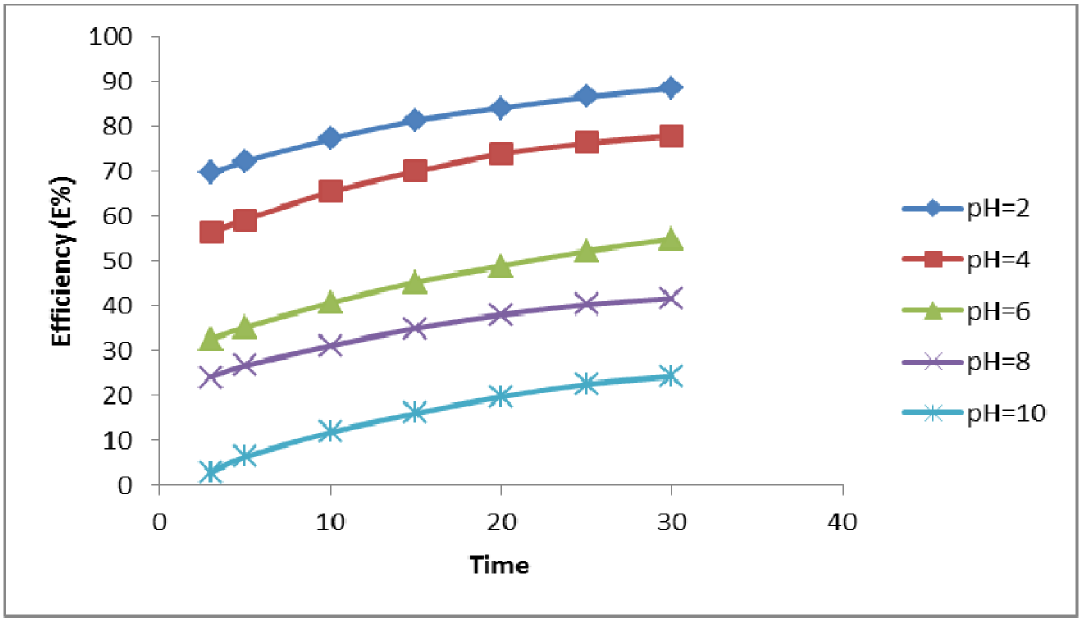

Fig. 2. Plot of efficiency (E %) vs coag-flocculation time for 200 mg L^{-1} okra at varying pH

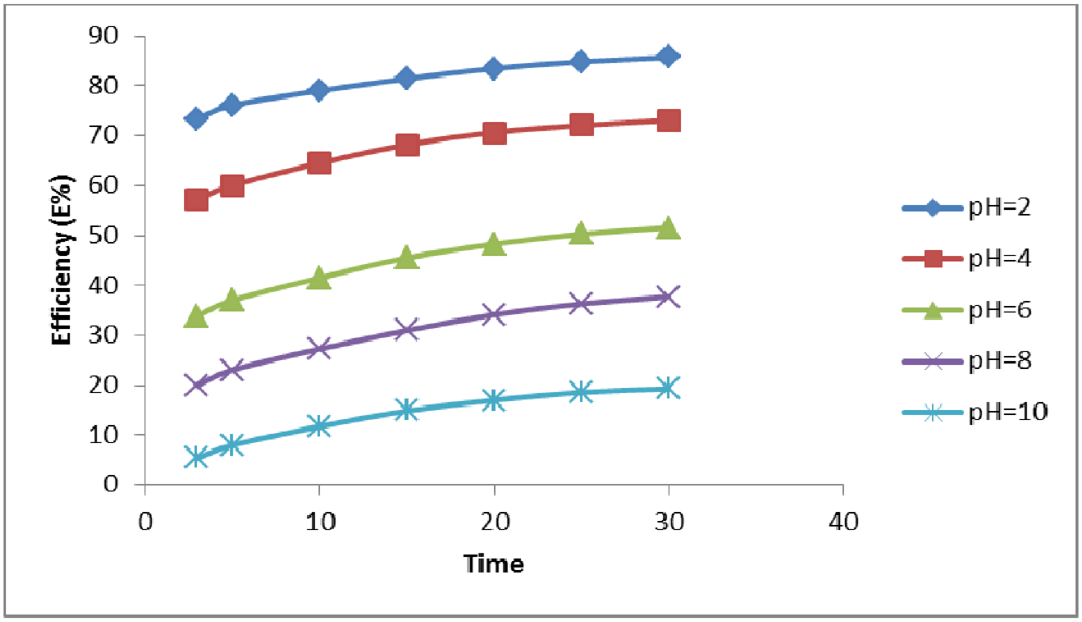

Fig. 3. Plot of efficiency (E %) vs coag-flocculation time for 300 mg L^{-1} okra at varying pH

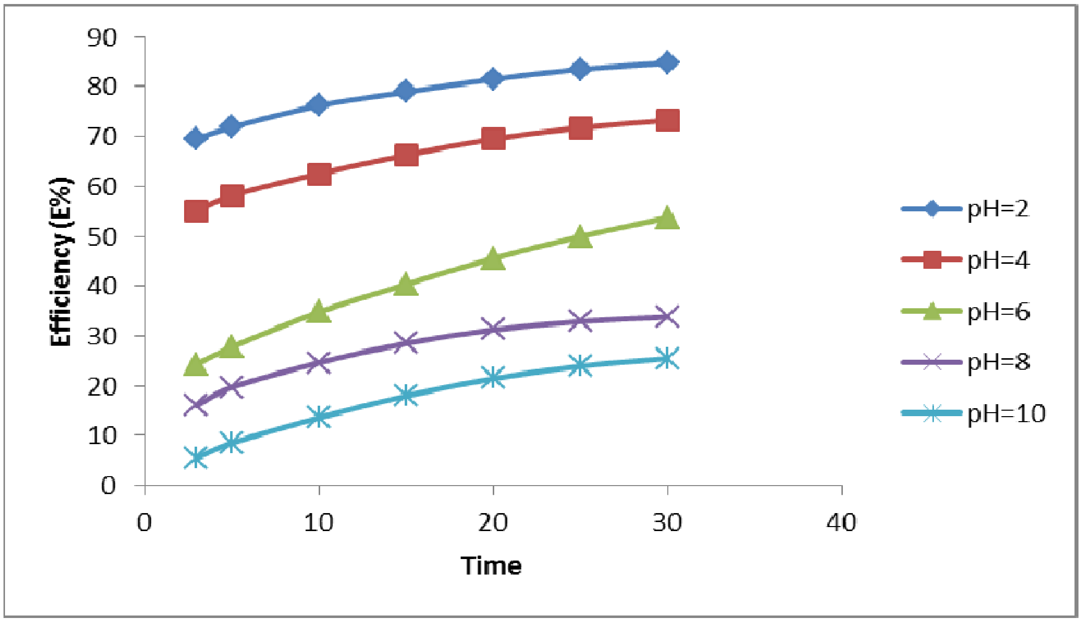

Fig. 4. Plot of efficiency (E %) vs coag-flocculation time for 400 mg L^{-1} okra at varying pH

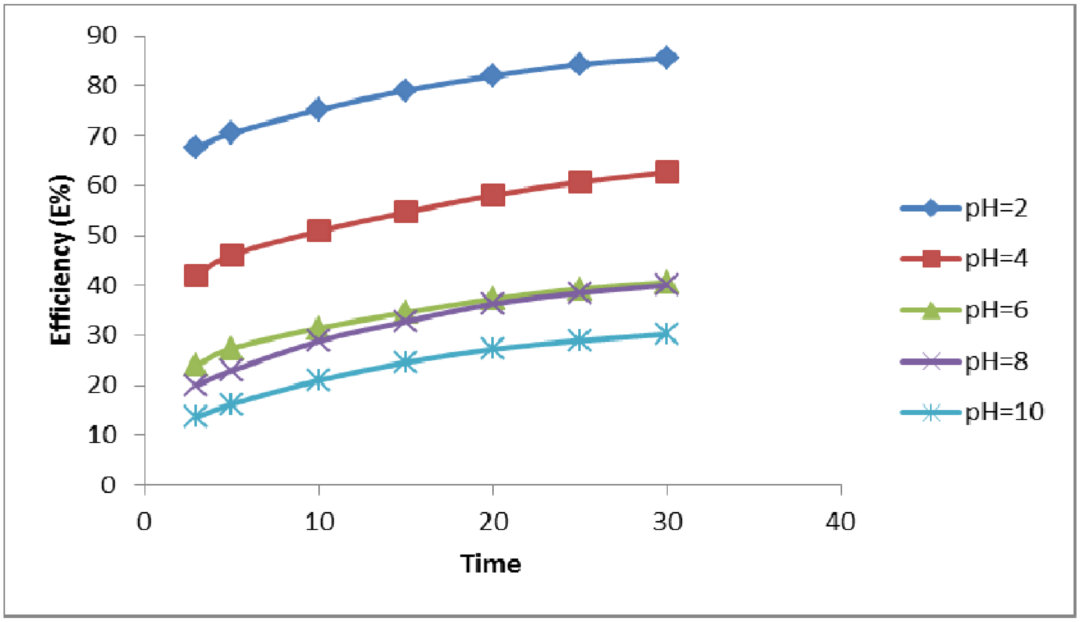

Fig. 5. Plot of efficiency (E %) vs coag-flocculation time for 500 mg L^{-1} okra at varying pH

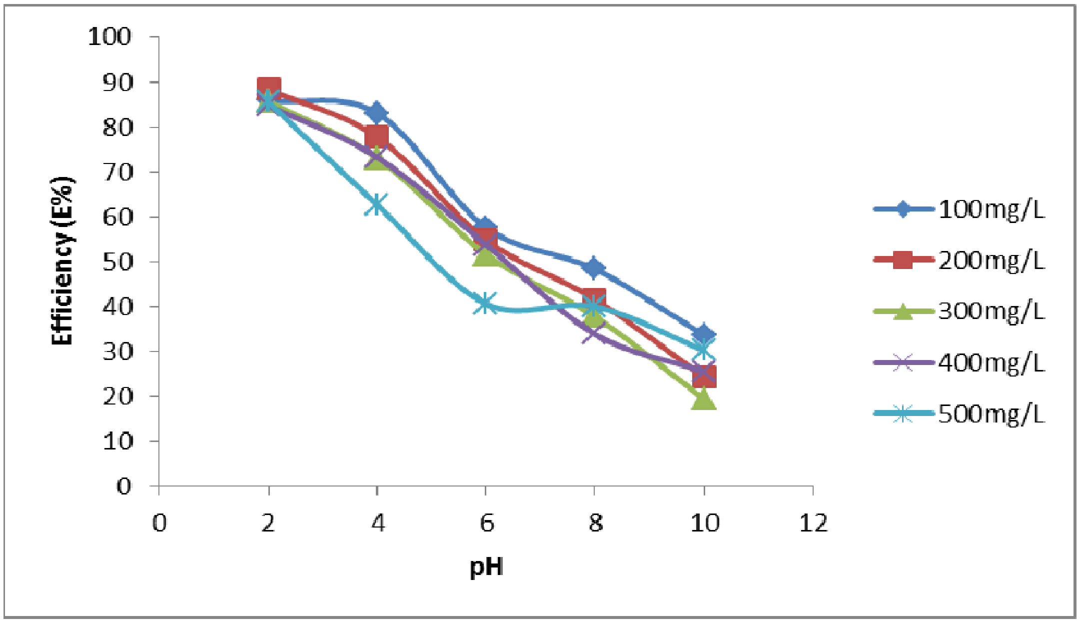

Fig. 6. Plot of efficiency (E %) vs pH at varying okra dosage

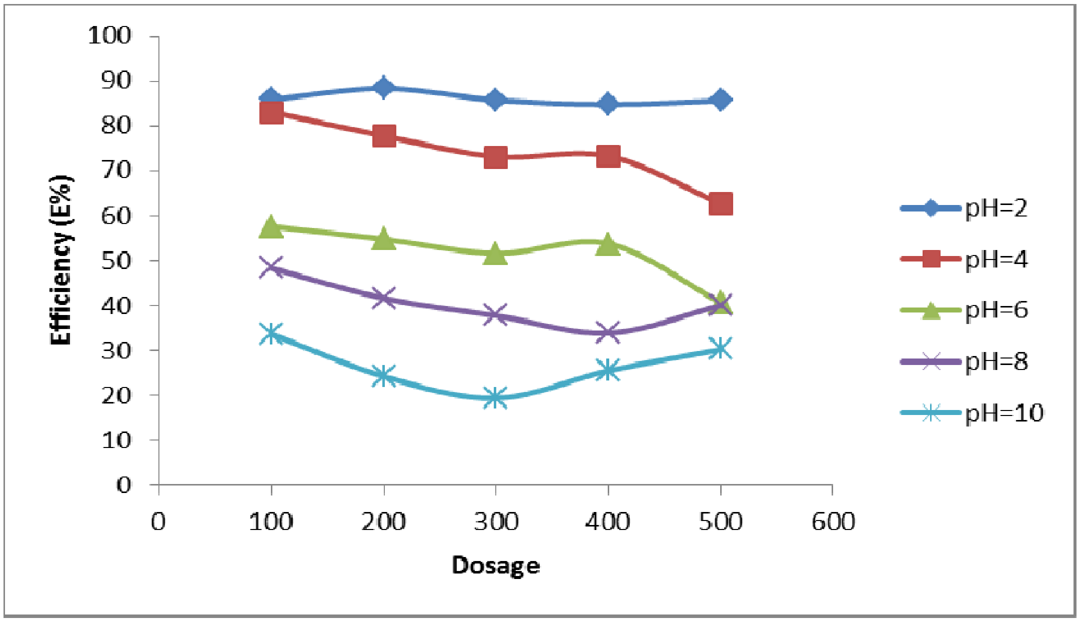

Fig. 7. Plot of efficiency (E %) vs dosage at varying pH

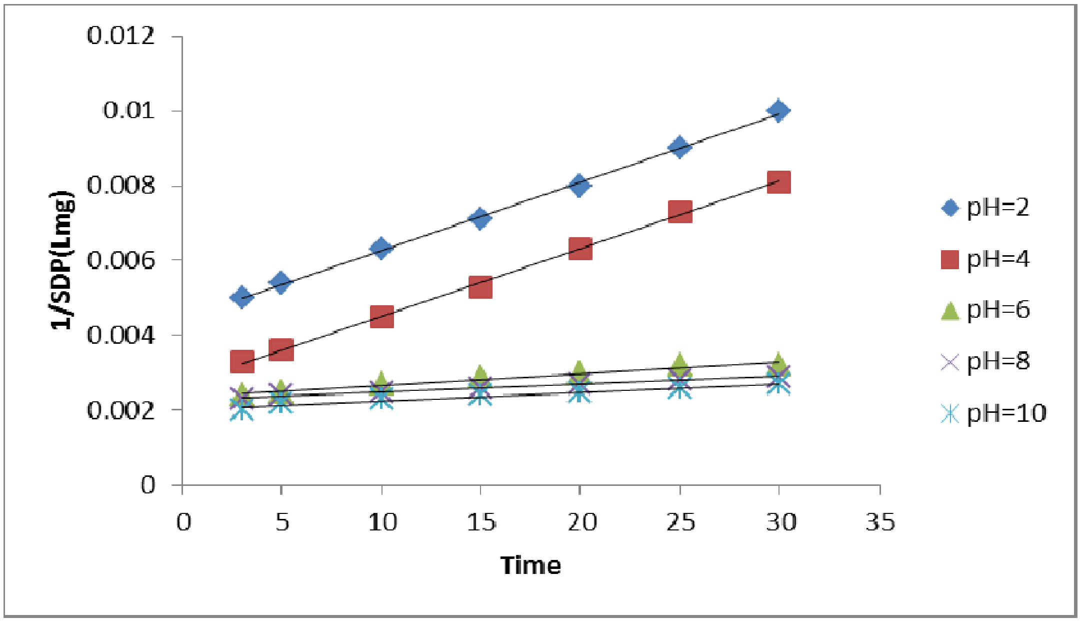

Fig. 8. Representative kinetic plot of 1/SDP as a function of time for 100 mg L^{-1} okra dosage

4.3 Particles Distribution Plots

The particle distribution plots are presented in figs. 9 and 10 for $\tau_{1/2}$=0.38 min and $\tau_{1/2}$=9.3min respectively. By substituting $\tau_{1/2}$ from equation 15 into 17, 18, 19, the microscopic particles aggregation can be graphically illustrated. In Fig. 9, the number of singlet particle decreases more rapidly than the sum particles. This indicate that early stage of this coag-flocculation is affected by colloidal destabilization and rapid sweep [10,14].

Table 3. Coag-flocculation kinetic parameter of okra in BRE at varying pH and 100 mg L^{-1} dosages

Parameters	pH=2	pH=4	pH=6	pH=8	pH=10
A	2	2	2	2	2
R^2	0.9977	0.9938	0.969	0.9864	0.9601
K(L/mg.min)	5.09×10^{-3}	4.04×10^{-3}	3.6×10^{-3}	3.23×10^{-3}	2.89×10^{-3}
β_{BR}(L/mg.min)	1.0×10^{-2}	8.16×10^{-3}	7.2×10^{-3}	6.46×10^{-3}	5.78×10^{-3}
K_r(L/min)	7.3×10^{-19}	7.65×10^{-19}	7.45×10^{-19}	7.45×10^{-19}	7.45×10^{-19}
$\varepsilon_p(mg^{-1})$	1.37×10^{16}	1.08×10^{16}	9.67×10^{15}	8.67×10^{15}	7.76×10^{15}
$\tau_{1/2}$(min)	0.54	0.68	0.38	0.85	0.95
τ'(min)	1.08	1.36	0.76	1.7	1.9
(-r)mg/min	$5.09 \times 10^{-3} N_t^2$	$4.04 \times 10^{-3} N_t^2$	$3.6 \times 10^{-3} N_t^2$	$3.23 \times 10^{-3} N_t^2$	$2.89 \times 10^{-3} N_t^2$

Table 4. Coag-flocculation kinetic parameter of okra in BRE at varying pH and 200 mg L^{-1} dosage

Parameters	pH=2	pH=4	pH=6	pH=8	pH=10
A	2	2	2	2	2
R^2	0.997	0.9911	0.9896	0.9498	0.9671
K(L/mg.min)	4.61×10^{-3}	$4.o5 \times 10^{-3}$	3.27×10^{-3}	3.15×10^{-3}	2.78×10^{-3}
β_{BR}(L/mg.min)	9.22×10^{-3}	8.1×10^{-3}	6.54×10^{-3}	6.3×10^{-3}	5.56×10^{-3}
K_r(L/min)	7.44×10^{-19}	7.43×10^{-19}	7.45×10^{-19}	7.45×10^{-19}	7.5×10^{-19}
ε_p (mg^{-1})	1.24×10^{16}	1.09×10^{16}	8.78×10^{15}	8.46×10^{15}	7.4×10^{15}
$\tau_{1/2}$ (min)	0.59	0.68	0.84	0.87	0.98
τ'(min)	1.18	1.36	1.68	1.74	1.98
(-r)mg/min	$4.61 \times 10^{-3} N_t^2$	$4.05 \times 10^{-3} N_t^2$	$3.27 \times 10^{-3} N_t^2$	$3.15 \times 10^{-3} N_t^2$	$2.78 \times^{-3} N_t^2$

Table 5. Coag-flocculation kinetic parameter of okra in BRE at varying pH and 300 mg L^{-1} dosage

Parameters	pH=2	pH=4	pH=6	pH=8	pH=10
A	2	2	2	2	2
R^2	0.9873	0.9639	0.9574	0.9407	0.881
K(L/mg.min)	5.22×10^{-3}	4.2×10^{-3}	3.37×10^{-3}	3.03×10^{-3}	2.82×10^{-3}
β_{BR} (L/mg.min)	1.04×10^{-2}	8.4×10^{-3}	6.74×10^{-3}	6.06×10^{-3}	5.64×10^{-3}
K_r (L/mg.min)	7.43×10^{-19}	7.43×10^{-19}	7.6×10^{-19}	7.45×10^{-19}	7.4×10^{-19}
ε_p (mg^{-1})	1.4×10^{16}	1.13×10^{16}	8.86×10^{15}	8.14×10^{15}	7.57×10^{15}
$\tau_{1/2}$ (min)	0.53	0.65	0.83	0.91	0.97
τ'(min)	1.06	1.3	1.66	1.82	1.95
(-r)mg/min	$5.22 \times 10^{-2} N_t^2$	$4.2 \times 10^{-3} N_t^2$	$3.37 \times 10^{-3} N_t^2$	$3.03 \times 10^{-3} N_t^2$	$2.82 \times 10^{-3} N_t^2$

Table 6. Coag-flocculation kinetic parameter of okra in BRE at varying pH and 400 mg L^{-1} dosage

Parameters	pH=2	pH=4	pH=6	pH=8	pH=10
A	2	2	2	2	2
R^2	0.9977	0.9941	0.9998	0.9167	0.9514
K (L/mg.min)	4.78×10^{-3}	3.97×10^{-3}	3.1×10^{-3}	2.98×10^{-3}	2.82×10^{-3}
β_{BR} (L/mg.min)	9.4×10^{-3}	7.94×10^{-3}	6.2×10^{-3}	5.96×10^{-3}	5.64×10^{-3}
K_r (L/mg.min)	7.34×10^{-19}	7.45×10^{-19}	7.45×10^{-19}	7.45×10^{-19}	7.46×10^{-19}
$\varepsilon_p\ (mg^{-1})$	1.28×10^{16}	1.06×10^{16}	8.32×10^{15}	8.0×10^{15}	7.56×10^{15}
$\tau_{1/2}$ (min)	0.57	0.69	0.88	0.92	0.97
τ' (min)	1.14	138	1.76	1.84	1.95
(-r)mg/min	$4.78 \times 10^{-3} N_t^2$	$3.97 \times 10^{-3} N_t^2$	$3.1 \times 10^{-3} N_t^2$	$2.98 \times 10^{-3} N_t^2$	$2.82 \times 10^{-3} N_t^2$

Table 7. Coag-flocculation kinetic parameter of okra in BRE at varying pH and 500 mg L^{-1} dosage

Parameters	pH=2	pH=4	pH=6	pH=8	pH=10
A	2	2	2	2	2
R^2	0.9981	0.9836	0.9719	0.9719	0.8947
K(L/mg.min)	4.58×10^{-3}	3.58×10^{-3}	3.03×10^{-3}	3.03×10^{-3}	2.95×10^{-5}
β_{BR} (L/mg.min)	9.0×10^{-3}	7.16×10^{-3}	6.06×10^{-3}	6.06×10^{-3}	5.9×10^{-5}
$K\_r\ (\ L/mg.min)$	7.32×10^{-19}	7.45×10^{-19}	7.45×10^{-19}	7.45×10^{-19}	7.45×10^{-19}
$\varepsilon_p\ (mg^{-1})$	1.23×10^{16}	9.61×10^{15}	8.14×10^{15}	8.14×10^{15}	7.92×10^{13}
$\tau_{1/2}$ (min)	0.6	0.76	0.91	0.91	9.3
τ'(min)	1.2	1.52	1.82	1.82	186
(-r)mg/min	$4.58 \times 10^{-3} N_t^2$	$3.58 \times 10^{-3} N_t^2$	$3.03 \times 10^{-3} N_t^2$	$3.03 \times 10^{-3} N_t^2$	$2.95 \times 10^{-5} N_t^2$

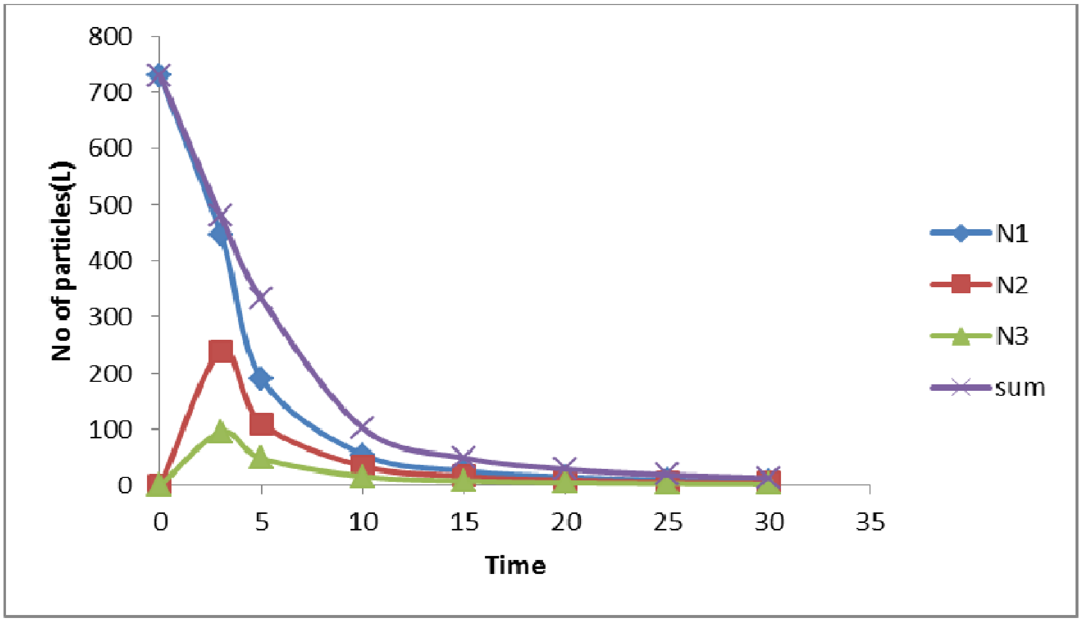

Fig. 9. Particles distribution behavior for half life of 0.38 min

In Fig. 10, the plot exhibits opposing profile to that of Fig. 9. Here there is decrease in rapidity of flocculation resulting from destabilization inertia. The curve clearly demonstrates that there is slow colloidal destabilization, resulting to low particle entrapment and low bridging mechanism.

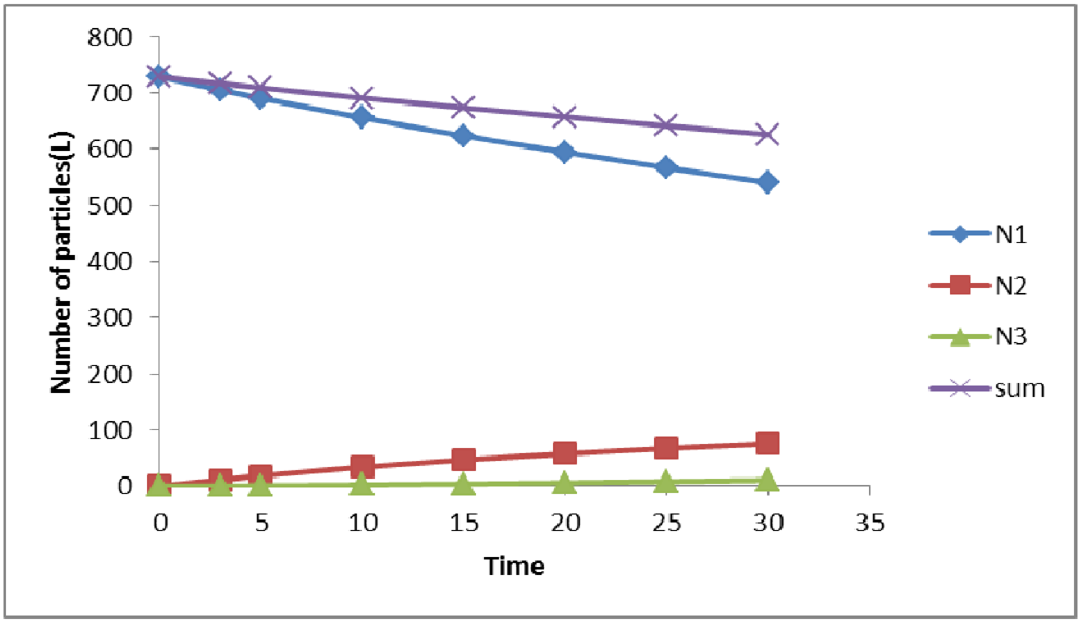

Fig. 10. Particles distribution behavior for half-life of 9.3 min

5. CONCLUSION

The performance of Okra seed extract as an effective plant coag-flocculation has been established. The process effectively removed 92.5% SDP from the effluent. However, the jar test conducted was sensitive to pH and dosage. 200 mg L^{-1} dose of Okra seed coagulant and pH 2 were found to be optimal conditions for the effluent treatment. It could be inferred at the experimental conditions that coag-flocculation process using okra seed was feasible for the treatment of brewery effluent.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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