EFFECT OF MIXING SPEED AND SETTLING TIME ON THE TREATMENT OF INDUSTRIAL WASTE WATER WITH CELLULOSE ACETATE PRODUCED FROM RICE HUSK

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ABSTRACT
Industrial waste constitutes the major sources of various kinds of metal pollution and other pollutants in natural water. Waste waters generated from industrial treatment plant contain considerable contaminants which must be reduced to safe level before being released into the environment. The industrial waste water used in this work was collected from a Process industry in Benin. Cellulose Acetate was produced from rice husk containing 40-50% cellulose. Rice husk undergoes several processes like grinding, dewaxing, delignification, bleaching and acetylation. The Cellulose acetate produced was used to treat the waste water. The effect of mixing time was analyzed at a dosage of 4g/L, 200 rpm to 1200 rpm of mixing rate and settling time of 360 minutes maximum. The result obtained indicates that the industrial waste water is safe to be discharged into the environment. This study also revealed that rice husk is a very good and cheap source for making Cellulose acetate used in waste water treatment.

Keywords: *Cellulose acetate, Rice husk, Waste water, Mixing speed, Settling time.*

1.0 INTRODUCTION

Nigeria at the moment has established industries like petroleum refinery, soap and detergent, food and beverages, brewery, textiles and apparels, building materials, timber products, wood and leather works, metal works, chemicals and plastics industries. All these industries produce various effluents that are discharged into the environment. Most large cities in Nigeria e.g. Lagos, Port Harcourt, Ibadan, Kano etc. are experiencing the negative effects of pollution from industrial effluents. Tons of industrial effluents are disposed indiscriminately into the lagoons, rivers, streams and lands (Olaniyi et al., 2012). It has been realized that discharges of untreated wastes or partially treated wastes containing algae, non-biodegradable organics, heavy metals and other toxicants hasten the deterioration of receiving water bodies. There is growing awareness of the need for effective treatment of various effluents before discharging into any public water body (Olutayo et al, 2015). The effluents generated from domestic and industrial activities constitute the major sources of the natural water pollution. This is a great burden in terms of waste water management and can subsequently lead to a point where such pollution problem, which can only increase treatment cost and also introduce a wide range of chemical pollutants with microbial contaminants to those water bodies (Akpor, 2011). Industrial activities and urbanization in developing countries including Nigeria has gradually led to the deterioration of the environment in recent years. Increase in crude oil exploration, refining and activities of other industrial establishments in the Niger Delta has resulted in the wide scale contamination of most of its creeks, swamps and rivers with hydro carbon and dispersant products (Odjegba and Bamgbose, 2012).

In this study we investigate the effects of mixing speed and settling time on the treatment of industrial waste water with cellulose acetate produced from rice husk and the treatment parameters were: Ph, Electrical Conductivity, Chemical Oxygen Demand (COD) Biological Oxygen Demand (BOD) Total Dissolved Solids (TDS) Total Suspended Solids (TSS) and Total Solids (TS)

LITERATURE REVIEW

The main advantage of rice husk over other conventional methods is its strong affinity and high selectivity towards heavy metals and other pollutants; this is because of the presence of binding groups on its surface (Banerjee *et al.*, 2012). It is of low cost because of being generated from Agricultural waste; this can be easily processed, applied and recovered without any adverse impact on the environment. It is eco-friendly and innovative, sustainable waste management. In future it is expected to replace the traditional adsorbents used for decontaminating heavy metals from water as it has great advantage such as high efficiency even with low metal concentrations, low cost, no additional nutrients requirements and ease of operation (Boota and Bhatti, 2009). These waste material pose a serious environmental issue if not utilized, and thus the efficient use of these agricultural by products would critically reduce the environmental impact while also generating substantial benefit.(Khandanlou et al., 2016), (Roshanak, 2016).

METHODOLOGY

MATERIALS

The major materials and equipment used in this work are as follows:

1. Chemicals and Reagent used are pure and analytical grade purchased from a Science Laboratory in Edo State. They include: Acetic acid, Acetic anhydride, Sodium hydroxide, Hydrogen peroxide, Hexane, Sulphuric acid, Ferrous ammonium sulfate, Chloroform, Ethyl alcohol, Potassium dichromate, Ferroin Indicator, Methylene chloride, Distilled water and Industrial waste water collected from Seven up company in Benin City.
2. Glassware and apparatus used are research materials for settling up experiment and were made by Jinotech and Pyrex. They include; Beakers, Measuring Cylinder and Burette
3. Equipment and other sensitive research machines made by Jinotech and Pyrex. They includes; Heating mantle, magnetic stirrer, pH and Conductivity meters, Analytical weighing balance, Air dry oven, TDS meter and Bunsen burner.
4. The Rice husk used was collected from a rice mill in Agenebode Edo State and the industrial waste water was collected from a process industry in Edo State.

METHODS

The methods employed in this work are as described below:

Rice Husk treatments.

The rice husk treatment comprises of milling, dewaxing, delignification and bleaching (Das et al., 2014). Approximately 49g of rice husk was washed with distilled water and some impurities and dirt particles were separated. The rice husk was then oven dried at 80°c for 24hrs using electro- heating standing temperature Air dry oven. The dried rice husk was then milled to pass through a 250 micro meter sieve. The milled rice husk was weighed to be 46.61 g.

Degumming/dewaxing was carried out to remove the oil/wax from the rice husk (Biswas *et al.*, 2006), the experiment was setup using a Soxhlet apparatus. After dewaxing, the rice husk was oven dried at 80°C for four hours. The degummed rice husk was weighed to be 45.23g. After dewaxing, delignification process was carried out. It is a hydrolysis process of breaking the structure of the lignocellulose and separating the lignin. 40g of sodium hydroxide is dissolved in 500 ml of water. This solution was then mixed homogenously in a conical flask and heated for about 42 minutes using Bunsen burner. Then it changes to dark brown indicating that the lignin has been separated from the cellulose. The pH was tested and was found to be alkalinity and the rice husk was washed with some water to maintain the pH at neutral and then the rice husk was filtered and kept in the oven to dry. After drying at 80°C, it was weighed to be 14.43g. Bleaching was also carried out. 30 ml of 50% H_2O_2 was added using a measuring cylinder and heat was applied using heating mantle at 55°c for 2hours with the pH adjusted to 11 by NaOH. Then the white cellulose powder was filtered from the suspension and washed with an adequate amount of distilled water until the filtrate became neutral (Rosa *et al.*, 2012; Fan *et* al., 2013).

CELLULOSE ACETYLATION.

The cellulose acetylation is the conversion from Cellulose into Acetylation (Chen *et al.*, 2016). 10g of the prepared cellulose simply called rice husk cellulose along with 2.4g of acetic acid, 25g of acetic anhydride, 150 ml of methylene chloride and 0.38g of sulfuric acid were added to a 1000ml round bottom flask. The mixture was heated to 80°c while stirring with a magnetic stirrer. 400 millilitres of chloroform was added and the mixture was stirred for 30 minutes at room temperature and then sieved. The filtrate was collected in a large beaker and combined with previous filtrates. The overs on the screen were discarded. The combined filtrates were further filtered into a round bottom flask using #54 whatman filter paper. Ethyl alcohol was used to remove the acetate film from the flask. Filtration through a coarse filter paper yielded cellulose acetate as residue and ethyl alcohol as filtrate. (Khandanlou et al.,2016; Salam et al., 2007).

RESULTS AND DISCUSSION

Effluent from Process industry commonly contains high concentrations of organic and inorganic chemicals (caustic soda, sodium citrate, sodium benzoate, sulfuric acid, chlorine, barium chloride, methylene blue, phenolphthalein etc.) and are characterized by high Chemical Oxygen Demand (COD), Biochemical oxygen Demand (BOD), Total Dissolved Solids (TDS), pH, and Total Suspended Solids (TSS) values. There are many methods of effluent treatment (Aluyor and Badmus, 2008). They includes; ion exchange, coagulation and flocculation, oxidation, reverse osmosis, biological decolonization and adsorption to reduce pH, color and TDS from beverage effluent, among them use of coagulants has been applied traditionally.

The experimental results are presented as follows;

Table 1 Effect of Setting Time

Time (min)	*pH*	*TSS (ppm)*	*TDS (ppm)*	*TS (ppm)*	*Conductivity (µS/cm)*	*BOD (mg/L)*	*COD (mg/L)*
0	6.70	3.26	442.00	445.26	884.00	185.00	2440.00
60	6.75	2.67	436.00	438.67	868.00	144.00	2040.00
120	6.85	2.00	406.00	408.00	821.00	128.00	1660.00
180	6.97	1.33	371.00	372.33	776.00	90.00	1120.00
240	7.09	0.93	323.00	324.67	704.00	72.00	912.00
300	7.31	0.67	270.00	270.67	608.00	55.00	640.00
360	7.94	0.00	214.00	214.00	513.00	23.00	415.00

Table 2 Effect of Mixing speed

RPM	**pH**	**TSS (ppm)**	**TDS (ppm)**	**TS (ppm)**	**Conductivity (µS/cm)**	***BOD (mg/L)***	***COD (mg/L)***
0	6.7	3.26	442.00	445.26	884.00	185.00	2440.00
200	6.72	2.13	436.00	438.13	871.00	177.00	2040.00
400	6.84	1.53	406.00	407.53	837.00	163.00	1820.00
600	6.91	0.67	371.00	371.67	792.00	146.00	1615.00
800	7.02	0.46	323.00	323.46	711.00	146.00	1521.00
1000	7.12	0.33	270.00	270.33	625.00	126.00	1506.00
1200	7.86	0.33	214.00	214.33	545.00	124.00	1506.00

Effect of Mixing Speed and Contact time on Electrical conductivity, BOD, COD, TSS, TDS and TS.

Mixing speed and time play an important role on flocs and the growth in flocculation process. Polymeric flocculent such as cellulose acetate disperses throughout the medium and absorb onto the colloidal particles surfaces for interparticle bridging or charge neutralization during the mixing period. Longer mixing time will lead to an increase in flocs breakage and this will decreases the flocculation rate. On the other hand, if the mixing time is too short, the collisions between the flocculants and colloids are not efficient to precipitate suspended solids in wastewater. (Abu Hassan, 2009)

The effect of mixing time was analyzed at a dosage of 4g/L, 200 rpm to 1200 rpm of mixing rate and settling time of 360 minutes maximum. It was observed from figure 4.1 that Electrical conductivity, BOD and COD reduction of 38%, 33% and 38% respectively at mixing speed of 1200 rpm of cellulose acetate on the waste water was less effective when compared with overall reduction in Figure 2 with Electrical conductivity, BOD and COD of 42%, 88% and 83%. This observation was also recorded in Figure 4 with TSS 100% reduction at 360 minutes of settling compare with Figure 3, TSS was 90% reduction after 1200 rpm of mixing speed. TDS and TS did not follow this trend as contact time had minimal effects on the waste water (figure 4.4). This is a result of excessive mixing time which lead to an increase in flocs breakage and thus decreases flocculation rate (Abu Hassan, 2009),(Ahmed et al., 2007). It was observed as well that the trend for all parameters are almost identical for values obtained for contact time and mixing speed but with different percentage of reduction for TSS (Ariffin and Hassan, 2007). The 100% reduction of TSS from the waste water shows that the waste water is free from suspended solids at the maximum settling time which is 360 minutes while the 90% reduction shows that at the maximum mixing speed suspended solids is almost free from the waste water.

When we carefully examine Figure 1, 2, 3 and Figure 4 consisting of TSS, TDS, Conductivity, BOD and COD reduction respectively, it can be drawn that the contact time and mixing speed of the industrial waste water has an influence on coagulation and flocculation using cellulose acetate. The trend that had been illustrated in Figure 2 shows that longer settling time would result in the high percentage reduction of Electrical conductivity, BOD and COD values of the waste water.

At higher mixing speed above 1000 rpm, the collisions between the flocculants and suspended particles are high and lead to the breakage of the flocculate chains thereby limiting the size of the flocs formed.

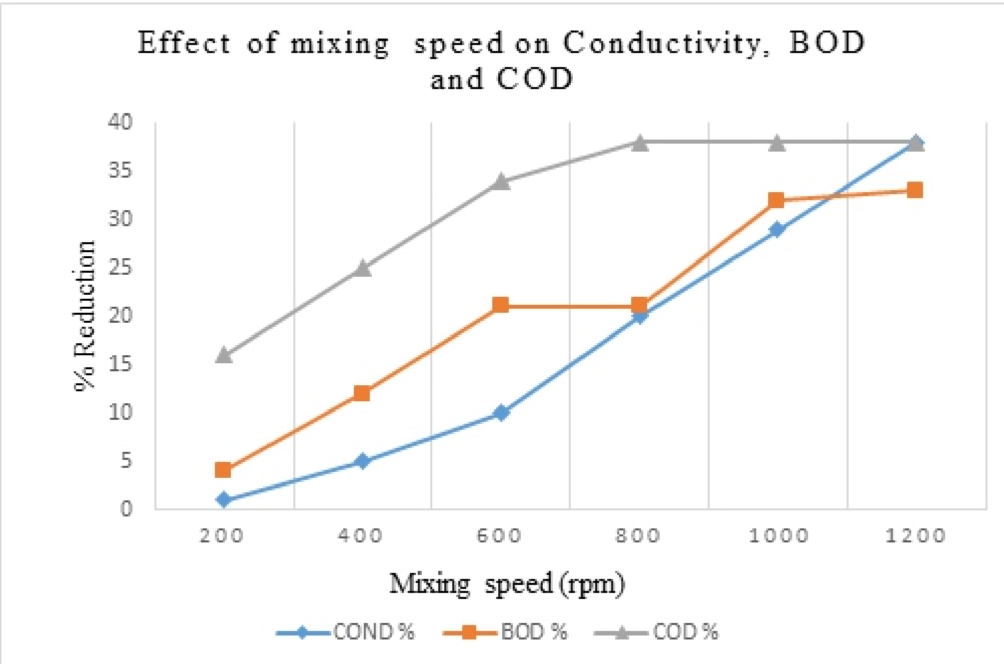

Figure 1: Effect of mixing speed on the reduction of Electrical conductivity, BOD and COD of waste water.

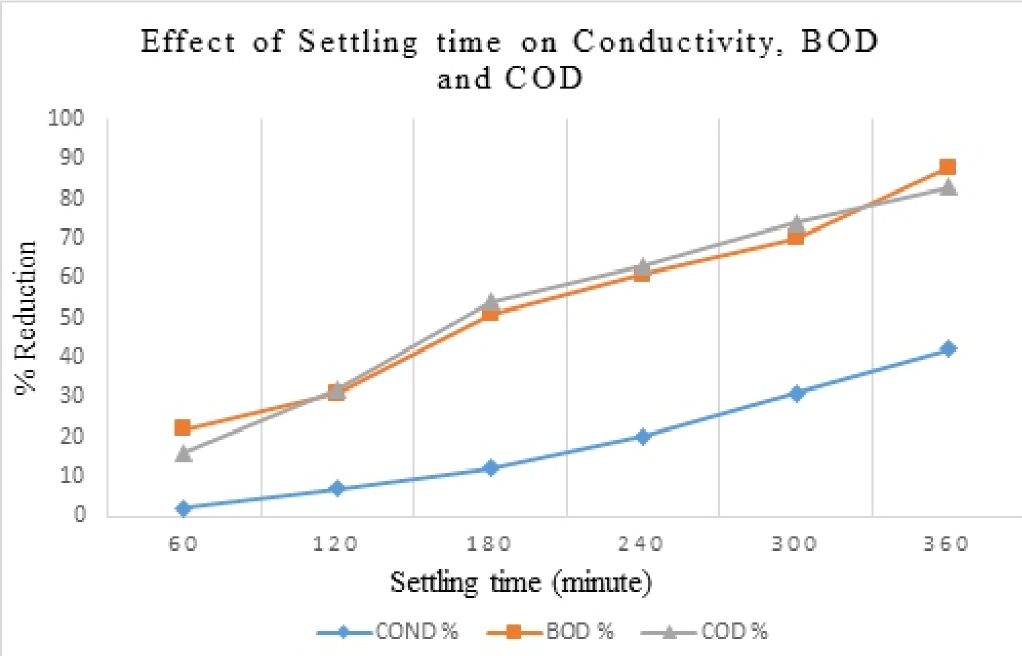

Figure 2: Effect of contact time on the reduction of Electrical conductivity, BOD and COD.

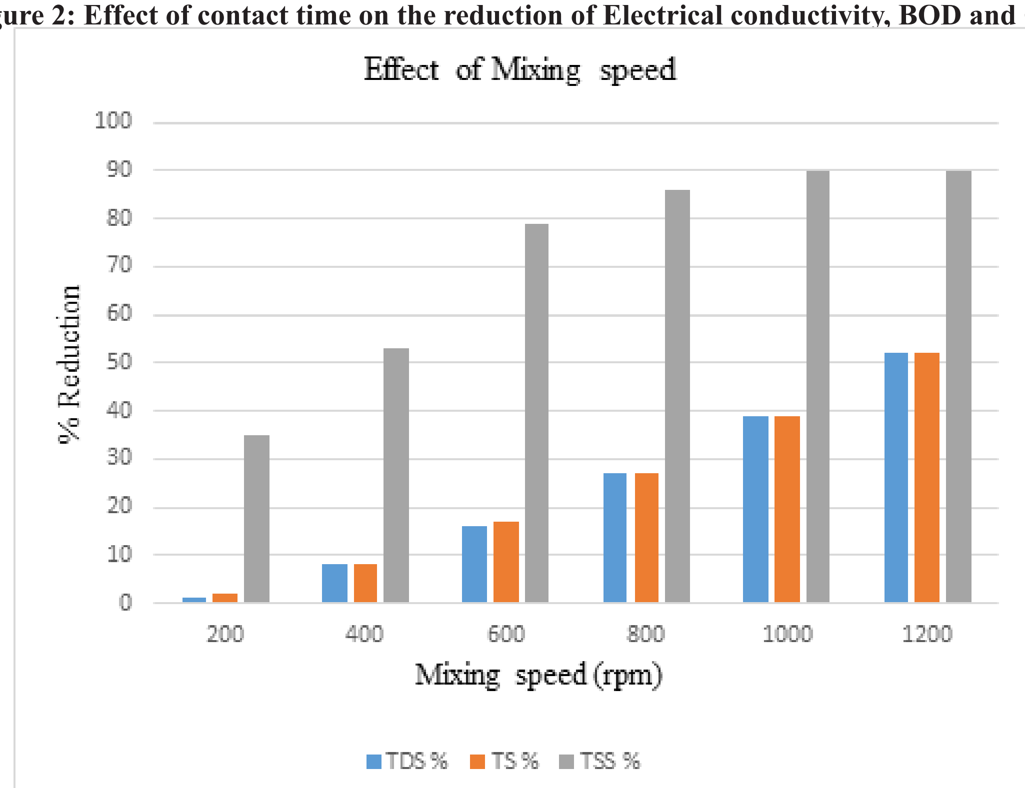

Figure 3: Effect of mixing speed on coagulation and flocculation.

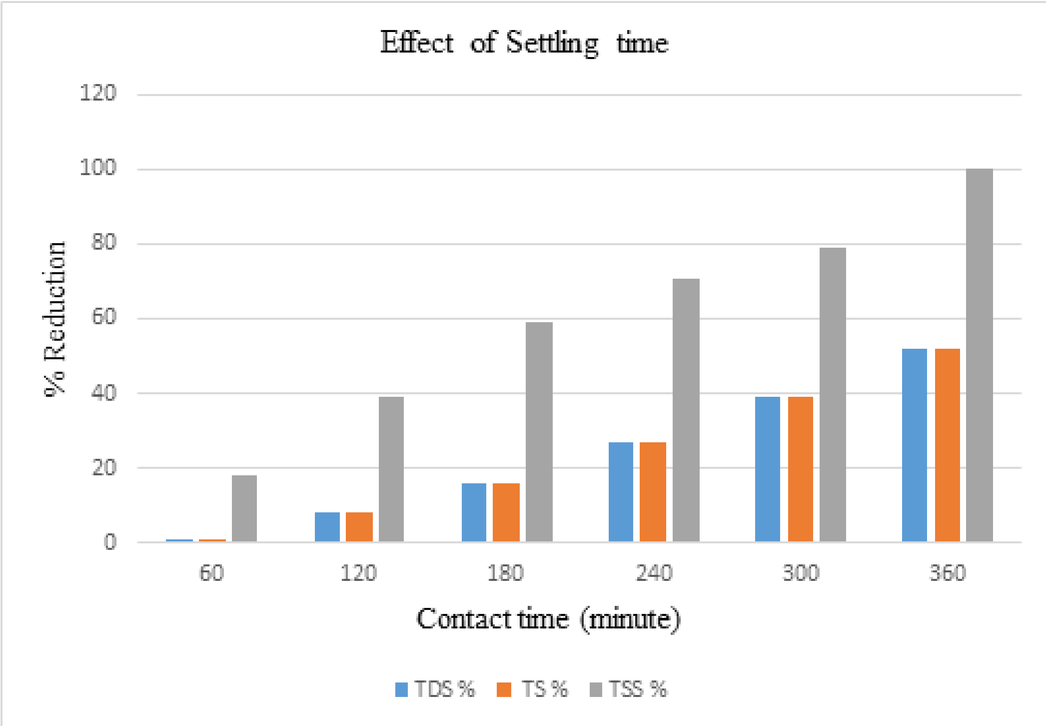

Figure 4: Effect of setting time on coagulation and flocculation.

Effect of mixing speed and setting time on pH.

Water with a pH of 7 is considered to be a *neutral* solution. If a solution is *acidic*, the concentration of H+ ions exceeds that of the OH– ions. In a *basic* solution, the concentration of OH– ions exceeds that of the H+ ions (WHO, 2007).

The pH will not only affects the surface charge of coagulants, but also affects the stabilization of the suspension. The study of pH was essential to determine the optimum pH condition of the treatment system. The effect of pH was analyzed at a dosage, 4g/L, 200 rpm of mixing speed, 400 rpm of mixing speed and 600 rpm of mixing speed,800 rpm, 1000rpm and 1200 rpm and 360 minutes maximum of settling time for a range of pH which varied from pH 6 to pH 8.

The results were presented in Figure 5 and 6 which showed the effects of pH on settling time and mixing speed in flocculation processes. Figure 5 shows how settling time affect pH while figure 4.6 shows how mixing speed affect pH. Moreover, it was observed that the trends for all the pH were almost identical.

By analyzing every curve in Figure 5 and 6 which for settling time against pH and Mixing speed against pH respectively; it can be stated that the pH of the wastewater has an influence on coagulation using Cellulose Acetate. Furthermore, the figures demonstrate the waste water which is acidic, after treatment is gradually moving to alkalinity according to the increase of the settling time, it therefore shows that the acidic content has been removed completely from the waste water. The same trend also follows for the mixing speed, at maximum mixing speed the waste water is very closed to alkalinity or at alkalinity, therefore, the optimum pH condition of the treatment system was pH 7.94

The pH value of streams and lakes is usually between pH 7 and 8. Levels between 6.5 and 8.5 pH are acceptable for most drinking water standards. Areas with higher levels of water hardness (high concentrations of Mg^{2+}, Ca^{2+}, and HCO_3^-) often have water with higher pH values (between 7.5 and 8.5).

Therefore, the values gotten from the experiment shows that the treated waste water is safe to be discharge into the environment or water bodies.

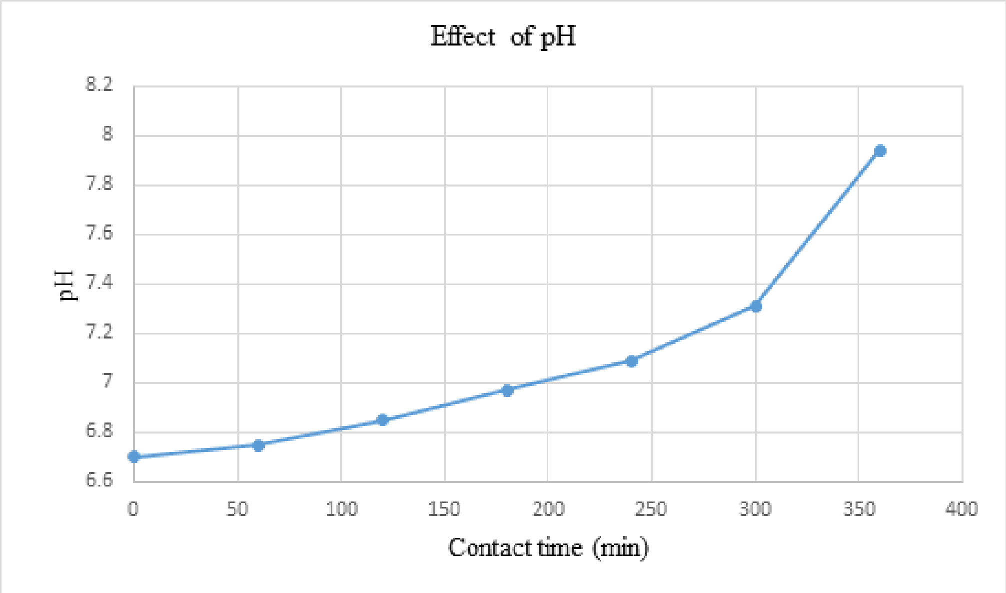

Figure 5: Effect of contact time on pH

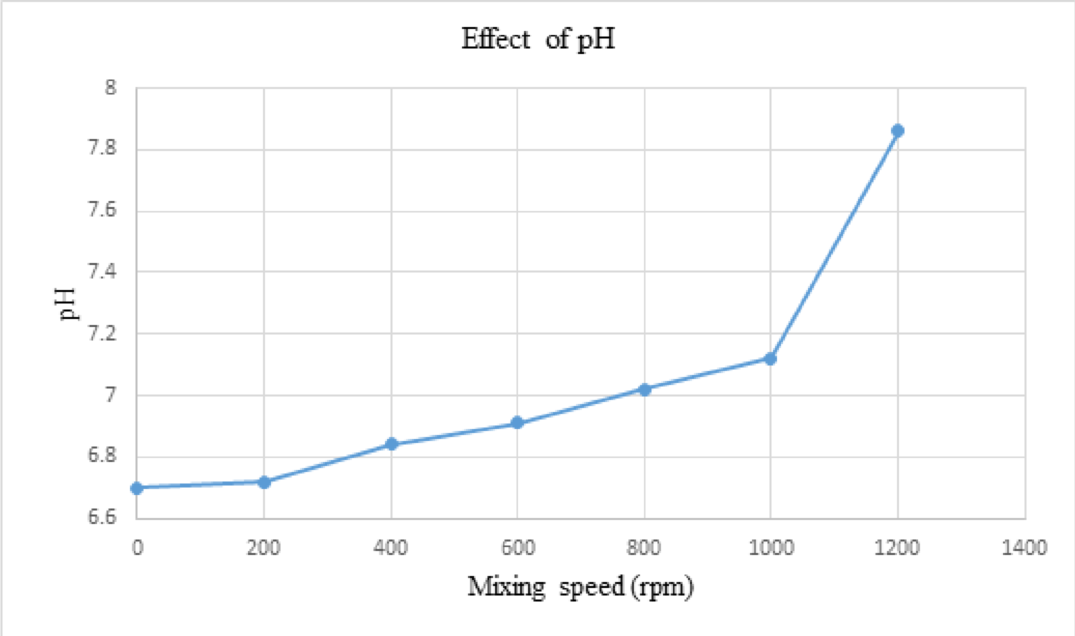

Figure 6: Effect of mixing speed on pH

CONCLUSIONS.

The concentration of Electrical conductivity, total dissolved solids and total solids in waste water collected from a Process industry are very high. The COD and BOD of untreated wastewater varied from 2000 to 2500 mg/L and from 170 to 200 mg/L respectively. The range of pH varies from 6.5 to 6.8 which indicates the acidic nature of the effluent. The present study indicates that mixing speed and setting time using Cellulose acetate is useful for the reduction and elimination of Electrical conductivity, BOD, COD, pH, total dissolved solids, total solids and total suspended solids in industrial waste water.

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