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# REMEDIATION AND REUSE OF RETTING FLAX WASTEWATER USING ASP FOLLOWED BY ADSORPTION ON AC

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**Key words:** Activated carbon, Activated sludge process, Remediation, Retting flax wastewater, Reuse.

**ABSTRACT**

Remediation and reuse of retting flax wastewater using activated sludge process (ASP) followed by adsorption on activated carbon (AC) were investigated. Wastewater produced (350 m3/d) is characterized by brownish to yellow color, bad odor and high organic contents due to lignin, cellulose and hemicelluloses in the flax fibers wastewater. Biodegradation of the organic contaminants was achieved successfully using ASP in presence of adapted culture containing pectin enzymes released by bacteria at a detention time of 6 h. The efficiency of ASP for chemical oxygen demand (COD), biological oxygen demand (BOD) and total suspended solids (TSS) were 95.86%, 94.53% and 97.6%, respectively. For wastewater reuse, post treatment was carried out using adsorption on AC with a dose of 2 g/L and at a contact time of 45 min. kinetics and adsorption isotherm was investigated. It was found that the sorption of residual COD from flax wastewater follows the second order kinetics and data fits both Langmuir and Freundlich isotherm models. The maximum adsorption capacity (qmax) of COD over AC was 333.33 mg/g. Residual COD, BOD and TSS were 38 mg O2/L, 9 mg O2/L and 2 mg/L, respectively which comply with the National Regulatory Standards for reuse.

## INTRODUCTION

Flax is a natural cellulosic fiber, non-lignified, lustrous, soft and pale yellow. Flax has a high economic value to produce flax fibers and linseed oil. The composition of flax is almost 92% cellulose, 2% hemicelluloses, 4% lignin and 2% other constituents (Amiri, *et al.,* 2015). The manufacturing process of flax, after cultivation, includes harvesting, rippling, water retting, drying, breaking and spinning. Water retting is the main source of pollution of flax manufacture process and it is highly polluted. It has a bad odor, high organic content, suspended particulates and refractory organic pollutants such as lignin (Abou-Elela, *et al.,* 2016). Also, the wastewater is characterized by the presence of an intense yellow color, which comes mainly from the lignin present in the flax fibers. Generally, retting is defined as the common technical term for 'rotting' used in natural fiber extraction process. The main retting process depends on the dual action of microorganisms and moisture on plants. These two parameters are the key factors responsible for the dissolve and/or rot away much of the cellular tissues and pectin surrounding the bast-fibre bundles. Accordingly, separation of fiber from the stem can be accomplished (Zhang, *et al.,* 2005; Yu and Yu, 2007). These processes are applied for the production of fibers from plant materials such as flax (*Linum usitatissimum*) and hemp stalks and coir from coconut husks (Zawani, *et al.,* 2013).

There are several types of retting such as mechanical decoration, chemical, heat, water, dew and enzymatic retting. The most widely practiced method of retting is water retting because it is simple and produce a good quality of fiber. However, huge amounts of water are used to obtain the fibers. The process takes place by submerging bundles of stalks in water. Then water penetrates to the central stalk portions, swells the inner cells, bursting the outermost layer,

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thus increasing absorption of both moisture and decay-producing bacteria. Flax fiber is decomposed by water retting which facilitates the degumming of pectin; the duration of retting time varies between 7-14 days according to the season (Tahir, *et al.*, 2011). Industrial wastewater discharged from retting process must be treated to protect the environment, human health and to minimize the risk for the contamination of ground water. Generally, most of organic compounds present in food industry and oil and soap wastewater can be easily biodegraded (El-Gohary, *et al.*, 1987). However, there are few research works dealing with the biodegradation of organic matters in flax wastewater. (Abou-Elela, *et al.*, 2016) studied the treatment of flax wastewater using advanced oxidation process followed by adsorption on granular activated carbon for the degradation of organic content, total suspended solids and oil and grease. They achieved percentage removals of 98.6% for BOD, 86.6% COD and 94.22% TSS. Also, (Hafez, *et al.*, 2012) studied the performance of Up-flow Anaerobic Sludge Blanket (UASB) reactor for treatment of flax retting wastewater at organic loading rates (OLRs) of 1.2 to 8.6 kg CODs $m^3.d^{-1}$, and hydraulic retention time (HRT) of 72 and 12 h. The chemical oxygen demand (COD) removal rates ranged from 64.5% to 90.5%.

Now-a-days, the strategies all over the world are not limited to the wastewater treatment only. Reuse of treated effluents is significantly increasing due to the scarcity of fresh water resources as stated in water resource management plan of Egypt (2017-2037) (El-Shafai, *et al.*, 2016). Treated wastewater can be used in irrigation (both landscape and agriculture), recharge of aquifers, seawater barriers, industrial applications, dual-distribution systems for toilet flushing, and other urban uses (Angelakis and Snyder, 2015).

In the current study, wastewater produced from a large flax company provides the material of this study. The wastewater produced (350 $m^3$/day) are discharged directly to a nearby agricultural drain without any treatment. Therefore, the aim of this work was to treat the retting flax wastewater to a level amenable for reuse and/or safe disposal into agricultural drains.

## MATERIALS AND METHODS

### Sources of wastewater

The annual production of flax fiber in the company was huge (5000 Tons/year). The retting process in water was carried out in 32 open ponds and 32 closed basins and for a duration of 15 days in winter and 10 days in summer. The wastewater discharged from these basins was almost 350 $m^3$/day. After accomplishing the desired retting period, wastewater is discharged alternatively from the assigned basins/ ponds to a nearby agricultural drain without any treatment. The wastewater was highly polluted with organic and inorganic constituents, which aggravate the pollution load in the drain. In order to utilize this wastewater, combined treatment was carried out to fulfill the requirements for reuse and/or safe disposal into agricultural drains.

### Samples collection and analysis

Composite samples were collected from the subsurface of the end-off-pipe using peristaltic dosing pump and during the working shifts. Physico-chemical analysis according to (Standard Methods for Water and Wastewater Examination, 2012) were carried out for raw wastewater as well as the treated effluents, (APHA, 2012). The analysis included pH, color, total and soluble COD, BOD, TSS, total phosphorous (TP), total kjeldahl nitrogen (TKN), ammonia nitrogen ($NH_4$ - N),oil and grease and all extractable matters by chloroform (O&G) and total sulphide ($H_2S$). The COD was measured according to dichromate method 5210-D. Soluble COD was measured after sample filtration using a membrane filter paper. BOD was measured according to 5 days BOD test method (5210-B). pH was measured using bench pH meter model Jenway 3505. TKN was measured using mercuric sulfate digestion method followed by titration method (4500-Norg). Ammonia was measured according to method (4500-$NH_3$).TP was measured according to the method (4500-C) while O&G was measured using the gravimetric partitioning method (5520-B). Total suspended solids were measured gravimetrically after sample filtration using GF/C paper, method (2540-D). Total sulfides were measured according to method (4500-E). Sludge analysis was also carried out and it includes the TSS and volatile suspended solids (VSS), sludge volume index (SVI) and microscopical examination.

### Treatability study

Treatment of retting flax wastewater was carried out using activated sludge process followed by sedimentation and then adsorption on activated carbon as shown in (Fig. 1).

### Aerobic treatment using ASP

### Start-up and operation

Bench scale experiments were carried out using 2.5 liters Plexiglas laboratory column (Abou-Elela, *et al.*, 2010). At the starting period, the columns were fed

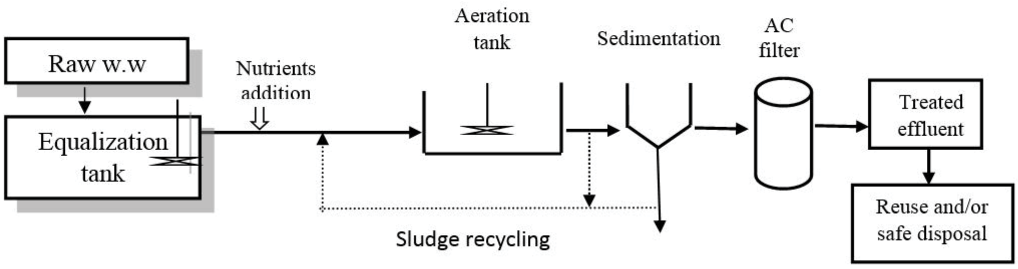


**Fig. 1** Flow diagram of the treatment process.

with pre-aerated sludge from a nearby wastewater treatment plant with an initial concentration of a mixed liquor suspended solids (MLSS) of 3-4 g/L, containing almost 75% of volatile matters. Air supply using air pump was adjusted to produce 2-3 mg/L dissolved oxygen. The pH was adjusted to ~ 7.0 using 0.1 N NaOH and the COD, TKN, TP concentrations were measured to assure that there is no deficiency in nutrients requirements (C: N: P ratio is 300:5:1 based on COD concentration). Ammonium dihydrogen phosphate ($NH_4H_2PO_4$) was added when required to compensate any nutrients deficiency. Gradual addition of retting flax wastewater to the aerated column was carried out to adapt the sludge to this type of wastewater. At the beginning of the experiment the ratio of industrial wastewater to sewage water was 1:1 for 7 days, then this ratio was increased to 1.25:0.75 for further 7 days and finally full feeding (100%) of retting flax wastewater was added till the steady state was achieved as indicated by almost constant COD values.

After acclimatization of the aerobic sludge to the industrial wastewater, a growth rate experiment was carried out to determine the optimum detention time needed for biological degradation. Based on the results obtained, the column was fed with raw wastewater at the predetermined optimum detention time; the effluent quality was monitored through daily analysis of COD and TSS after one hour sedimentation.

### Microscopic examination

Sludge was examined microscopically using microscope Model number (B-180, Optika). This was done to identify the mixed microbial communities; including bacteria, rotifers and protozoa which are responsible for the biological degradation process. The microorganisms were identified according to (APHA, 2012).

### Post treatment by adsorption on AC

The biologically treated effluent was then subjected to a polishing step using adsorption on AC. This was carried out to assure color removal and to improve the quality of treated effluent to comply with the permissible limits of wastewater discharge into drains and/or reuse. Batch adsorption experiments at ambient temperature (approximately 21°C) were conducted by shaking a series of five glass reagent bottles containing 100 ml of biologically treated effluent with adsorbent dosage of (AC) equal 2 g/L at different time intervals namely; 15 min, 30 min, 45 min, 60 min, 90 min and 120 min. Also, the same batch experiments were carried out using different adsorbent dosage to obtain the optimum dose of GAC at the pre-determined optimum contact time. The shaking process proceeded for 45 min to establish equilibrium, after which the mixture was left to settle for 10 min and then filtered using Whatman, No. 42. The final treated effluent was characterized according to APHA, (2012).

## RESULTS AND DISCUSSION

### Wastewater characteristics

Characteristics of the end-off- pipe are shown in Table 1. Results indicated that the intermittent discharge of wastewater from the retting basins and the variation in retting time resulted in great variations in the quality of wastewater. It is brownish to yellow in color, has bad odor and high organic content. The total COD ranged from 1272 to 6718 mg $O_2$/L, while the higher BOD concentration was 3300 mg $O_2$/L and with a minimum value of 365 mg $O_2$/L. The high organic content in wastewater is mainly due to cellulose, hemicelluloses, and lignin in the flax. The TSS reached 2800 mg/L. This is due to the presence of some residues of plant fibers. O&G concentration reached 519 mg/L. This value is attributed to the presence of lignin and its derivatives released during retting process (Perez, *et al.*, 2002). The mechanism of retting is mainly achieved by pectin enzymes produced by bacteria (Sharma, *et al.*, 2011). In retting process, the breakdown of the bonds between the constituent layers of the stem through the removal of pectin and hemicelluloses that binds the layers together occurs (Foulk, *et al.*, 2011; Gomez, *et al.*, 2007).

**Treatability study**

An Equalization tank was used before treatment due to the variation of sample composition as shown in Table 1. It increases the efficiency of biological process resulting in a uniform flow characteristic, minimize the impact of shock loads during operation and improve odor removal due to the presence of mixing in the equalization tank as stated by (Abou-elela, *et al.*, 2017). The results depicted in Table 2 show that BOD/COD ratio ranged from 0.4- 0.5 which indicates the high biodegrade ability of wastewater. Accordingly, ASP was proceeded. There are three simultaneous stages takes place in biodegradation namely; biodeterioration, biofragmentation and assimilation.

In presence of oxygen and organic matters:

1) Microorganisms stick onto the surface of material by aggregation or adhesion,

2) Proliferation of attached microbial cells,

3) Enzyme production,

4) Biodegradation and breakdown of macromolecules to oligomonomers and monomers,

5) Reduction of degree of polymerization and production of degradable products.

The final products of assimilation process are shown in equation (1).

$$C_{polymer} + O_2 \rightarrow CO_2 + H_2O + C_{Residue} + C_{Biomass} + Salts \qquad (1)$$

The flax wastewater was acclimatized for a period of two weeks at a retention time of 24 hrs. until a constant removal rate of COD was achieved. After reaching the steady state, the performance of activated sludge process was improved. The percentage removal of COD ranged between 89.46% and 96.76% with residual concentrations of 217 and 134 mg $O_2$/L, respectively (Fig. 2).

Microscopic examinations of the MLSS were carried out during the acclimatization period. Many tolerant species of microorganisms were found, simple life forms such as amoebas, free swimming ciliates, then multi-celled animals such as the rotifers and flat worms. The presence of the ciliates and rotifers (higher life forms) in the sludge floc is a sign of good sludge quality and the efficiency of the treatment process. These microorganisms including prokaryotic and eukaryotic plays an important role in purifying the wastewater, consuming bacteria and small particulates in wastewater (Abdelfattah, *et al.*, 2016; Metcalf and Eddy, 2013).

**Table 1.** Characteristics of retting flax wastewater from different basins

| Parameters / Samples | Units | Closed Basins | | | | | Open Ponds | Min | Max | Mean | St. Dev. |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | 1 | 2 | 3 | 4 | 5 | 6 | | | | |
| PH | -- | 4.6 | 4.82 | 4.6 | 4.5 | 4.7 | 6.1 | 4.5 | 6.1 | -- | -- |
| Color | Co/Pt units | 187 | 94 | 154 | 56 | 344 | 61 | 56.0 | 344.0 | 149.3 | 108.5 |
| $COD_t$ | mg$O_2$/L | 3995 | 2060 | 3187 | 1272 | 6718 | 1565 | 1272 | 6718 | 3132.8 | 2033.3 |
| $COD_S$ | mg$O_2$/L | 2775 | 1680 | 3075 | 952 | 5820 | 947 | 947 | 5820 | 2541.5 | 1838.4 |
| BOD | mg$O_2$/L | 1200 | 365 | 1480 | 675 | 3300 | 765 | 365 | 3300 | 1297.5 | 1057.7 |
| TSS | mg/L | 122 | 140 | 63 | 192 | 2800 | 100 | 63.0 | 2800 | 569.5 | 1093.6 |
| TP | mg P/L | 6 | 5 | 8 | 2 | 5 | 1.1 | 1.1 | 8.0 | 4.5 | 2.6 |
| $NH_3$ | mg N/L | 1.6 | 7.8 | 1.96 | N.D | 28 | 61 | 1.6 | 61 | 20.1 | 25.3 |
| TKN | mg N/L | 45.6 | 30.2 | 39.2 | 20 | 33.6 | 213 | 20 | 213 | 63.6 | 73.69 |
| $H_2S$ | mg/L | 4 | 4 | 8.8 | N.D | 14 | 4 | 4.0 | 14.0 | 7.0 | 4.5 |
| O&G | mg/L | 66 | 96 | 86.5 | 114 | 519 | 134.5 | 66.0 | 519.0 | 169.3 | 172.9 |
| *N. D= Not Detected | | | | | | | | | | | |

**Table 2.** Determination of the optimum detention time for ASP

| **Parameters** | **Raw W.W** | **Time (h)** | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | | **Zero** | **1** | **2** | **3** | **4** | **5** | **6** | **8** | **24** |
| $COD_t$, mg $O_2$/L | 3000 | 1800 | 1208 | 675 | 333 | 270 | 184 | 124 | 127 | 165 |
| % Removal | 0 | 40 | 59.73 | 77.5 | 88.9 | 91 | 93.86 | 95.86 | 95.76 | 94.5 |
| Sludge volume | ml/L | 35 | 35 | 36 | 38 | 41 | 45 | 45 | 48 | 40 |

### Determination of the optimum operating conditions for ASP

Table 2 shows that the best removal rate of COD was achieved after 6 h detention time. Their corresponding residual values were 124 mg $O_2$/L with a removal rate of 95.86%. At zero time, 40% of $COD_t$ was removed. This is attributed to the instant adsorption on the sludge and degradation by microorganisms (Rúa-Gómez, *et al.,* 2012). Table 3 shows complete analysis of the treated effluent at the optimum detention time (6 h). The removal rates of COD, BOD and TSS reached 95.86%, 94.5% and 97.6% with residual values of 124 $mgO_2$/L, 70 $mgO_2$/L and 12.5 mg/L. The total weight of sludge analysis was 5.7 g/L and with 3.3 g/L volatile organics. The average SVI was 75 which indicate the good setlability of sludge. However, for safe disposal into a nearby drain and/or reuse post treatment is required for further improvement of the quality of the effluent.

### Post treatment using AC

Application of AC for color adsorption and micro pollutants removal is the most well-liked technique used in industry. Adsorption is influenced by adsorbent dose and contact time. To determine the optimal time for adsorption, a fixed dose of AC (2 g/L) with different detention times were examined and the results are shown in (Fig. 3). The adsorption capacity increased with time and COD levels decreased as well (Abou-Elela, *et al.,* 2016). Results revealed that the optimal contact time was 45 min with a residual COD concentration 38 mg/L. To determine the optimum dose of adsorbent, several batches were carried out using different doses (1-6 g/L). The results illustrated in (Fig. 4) shows that at a dose of 2 g/L, the best percentage removal of COD was 69.35%. Increasing the dose of adsorbent up to 4 g/L resulted in a slight increase in capacity and removal rate of COD. From the economic point of view, 2 g/L at 45 min was selected as the optimum dose and optimum time.

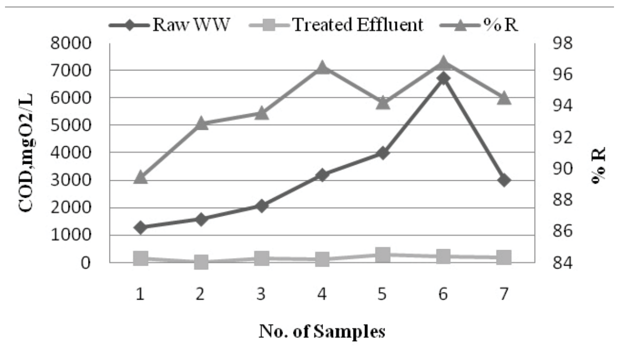


**Fig. 2** Variation of COD values during extended aeration.

### Kinetics of biosorption

For realizing more data about the mechanism of adsorption and rate determining step, kinetic models were applied. Pseudo-first- or pseudo-second-order are kinetic models used to estimate the adsorption process mechanism. The two models can be expressed in linear forms as in equations 2 and 3:

Pseudo-first-order: $$\log(q_e - q) = \log q_e - \frac{k_1}{2.303}t \quad (2)$$

Pseudo-second-order: $$\frac{t}{q} = \frac{1}{q_e}t + \frac{1}{k_2 q_e^2} \quad (3)$$

Where t is the contact time (min), $q_e$ and q, in mg/g, are the adsorption capacity amount of COD adsorbed at equilibrium and time t and $k_1$, in $min^{-1}$, and $k_2$, in g/mg min, are the pseudo-first-order and pseudo-second-order rate constants, respectively.

(Fig. 5 and 6) show the kinetics model of COD adsorption on AC. The results is revealing that the kinetics of COD adsorption by AC is well fitted with kinetic model of pseudo- second-order ($R^2$=0.998) rather than pseudo-first –order ($R^2$=0.865).

### Adsorption isotherm of residual COD

The equilibrium isotherms for $COD_{residual}$ from Flax

**Table 3.** Physico-chemical analysis of ASP effluent

| Parameters | Units | Raw WW | Treated Effluent | % R |
|---|---|---|---|---|
| PH | mg $O_2$/L | 6.1 | 8.8 | - |
| Color | Co/Pt units | 138 | Colourless | 100 |
| $COD_t$ | mg $O_2$/L | 3000 | 124 | 95.86 |
| BOD | $mgO_2$/L | 1280 | 70 | 94.53 |
| TSS | mg/L | 524 | 12.5 | 97.6 |
| TP | mgN/L | 4.1 | 0.8 | 80.48 |
| $NH_3$ | mgN/L | 20 | N.D | 100 |
| TKN | mgN/L | 61 | 5.8 | 90.49 |
| $H_2S$ | mg/L | 4 | 0.8 | 80 |
| O&G | mg/L | 169 | 8 | 95.26 |

*N.D= Not Detected
*Average of 6 samples

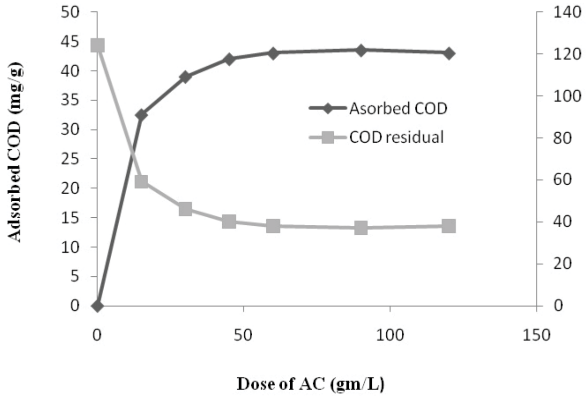


**Fig. 3** Effect of detention time on adsorbed COD.

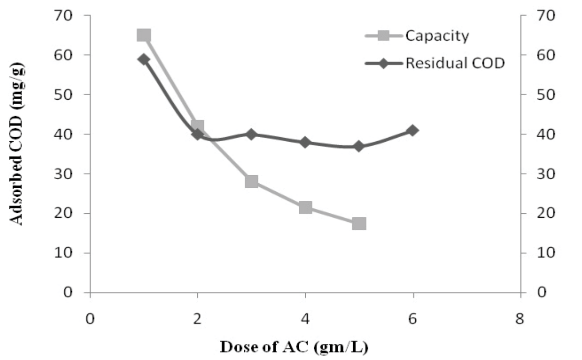


**Fig. 4** Effect of adsorbent dosage on COD removal.

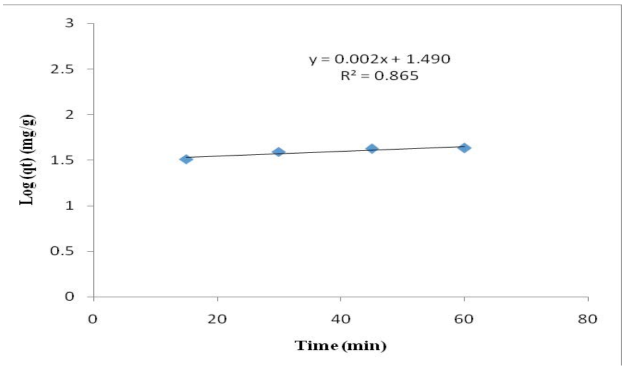


**Fig. 5** First order kinetic of adsorption of $COD_{residual}$ from Flax wastewater.

wastewater sorption were applied. The data are used to find out the relationship between $q_e$ and $COD_e$. Two isothermal models were developed; Langmuir, and Freundlich (Abdel-Aty *et al.* 2015).

Langmuir model assume that: (1) proceeding of adsorption at specific uniform sites on surface of the adsorbent (2) all adsorption sites on surface have constant energy (3) and the capability of independent adsorption of molecules regardless the occupancy of neighbouring sites. Isotherm models were used for of the sorption of $COD_{residual}$ on AC bio sorbent at equilibrium. The Langmuir model was used to fit the adsorption of COD on AC and (Fig. 7) revealed Langmuir model. The Langmuir model is moderately succeeded for ascribing the experimental data. But adsorption mechanism of COD by AC is not limited to monolayer adsorption of $COD_{residual}$ on AC surface. Since all available surface sites for adsorption are not alike, thus the maximum adsorption capacity ($q_{max}$) was 333.33 mg/g.

The isothermal model of Freundlich is established by the plot of log $q_e$ verus log $COD_{residual}$ has a slope with a value of 1/n and an intercept magnitude of log $K_F$. Freundlich isotherm was validated in (Fig. 8). The correlation coefficient ($R^2$) value was 0.98. The biosorption capacity (KF), was 94.82 mg/g, which is equal to $q_e$ at $COD_{residual}$=unity. Freundlich model gave 1/n where amounted with 1.86 referring to the capability of explaining the uptake of residual COD by heterogeneous bio sorbents. The obtained correlation coefficient of the two models reveals that both isotherm models are suitably represented the COD adsorption by AC.

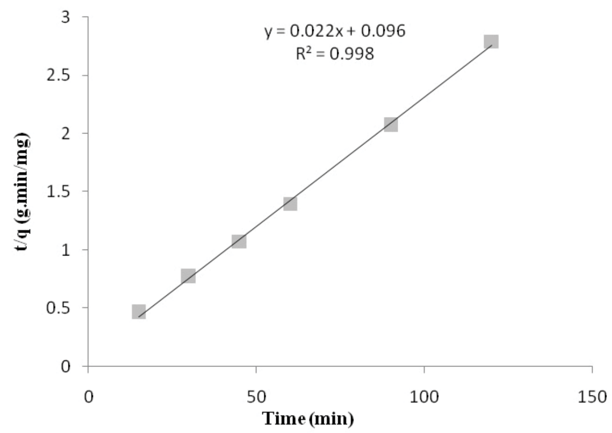


**Fig. 6** Second order kinetic of adsorption of $COD_{residual}$ from flax wastewater.

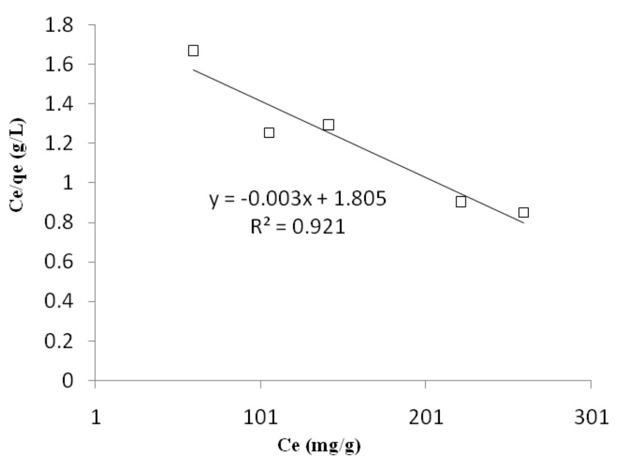


**Fig. 7** Langmuir isotherm model of COD removal over AC.

**Table 4.** Overall efficiency of ASP followed by AC

| Parameters | Units | Raw WW | Biologically Treated Effluent | % R | Final Effluent | % R | ECP Code (501/2015) |
|---|---|---|---|---|---|---|---|
| pH | $mgO_2/L$ | 6.1 | 8.8 | - | 8 | - | Not defined |
| Color | Co/Pt units | 138 | 10 | 92.75 | colorless | 100 | Not defined |
| COD | $mgO_2/L$ | 3000 | 124 | 95.86 | 38 | 98.73 | Not defined |
| BOD | $mgO_2/L$ | 1280 | 70 | 94.53 | 9 | 99.29 | <30 |
| TSS | mg/L | 524 | 12.5 | 97.6 | 2 | 99.61 | <30 |
| $NH_3$ | mg N/L | 20 | N.D | 100 | N.D | 100 | Not defined |
| $H_2S$ | mg/L | 4 | 0.8 | 80 | N.D | 100 | Not defined |
| O&G | mg/L | 169 | 8 | 95.26 | 1 | 99.4 | Not defined |
| *N.D= Not Detected | | | | | | | |

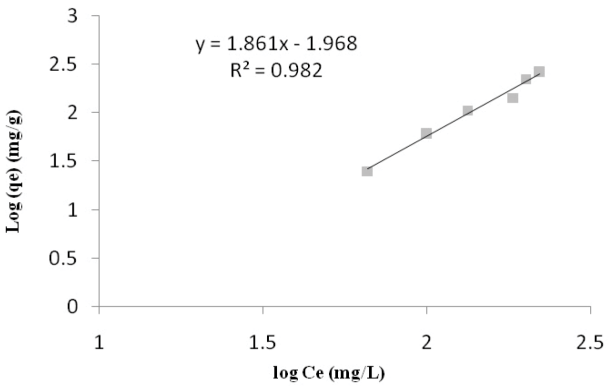


**Fig. 8** Freundlich isotherm model of COD removal over AC.

The quality of the final treated effluent was very satisfactory for the removal of 98.73% COD, 99.61% of TSS and 99.4% of oil & grease Table 4. The adsorption capacity of AC, which is hydrophobic in nature and mainly occurs due to complexation, ion exchange and hydrogen bonding are all contributed to the good quality of effluent (Grassi, *et al.,* 2012).

## CONCLUSION

The production of flax fibers is one of the industrial processes which consumes a huge amount of water. Consequently, the wastewater produced contains high amount of organic matters, TSS, color and has a bad odor. This is due to the retting of flax fibers in water. Remediation of flax wastewater can be efficiently treated using ASP followed by adsorption on AC. Using ASP at a detention time of 6 hrs. The removal rate of pollutants reached 95.86% COD, 94.53% BOD and 97.6% TSS with corresponding residual values of 124 mg O2/L, 70 mg O2/L and 12.5 mg/L. Reuse of the huge amount of wastewater necessitated post treatment using AC. The use of 2 g/L of AC and at 45 min contact time produced a good quality of effluent amenable for reuse in agriculture according to the National Code of Practice (ECP 501, 2015).

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