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Research Article

Remediation and Reuse of Retting Flax Wastewater Using Activated Sludge Process Followed by Adsorption on Activated Carbon

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Abstract

Background and Objective: Water retting is the most widely process used to produce flax fibers and linseed oil. The manufacturing process in this industry consumes a huge amount of fresh water. Wastewater produced is characterized by brownish to yellow color, bad odor and high organic contents due to lignin, cellulose and hemicelluloses. In this study retting wastewater (350 m³/d) was discharged directly without treatment to a nearby agricultural drain and consequently aggravates the problem of pollution. To avoid the high contamination of the drainage water and to overcome the high consumption of water in this industry, remediation and reuse of treated wastewater using Activated Sludge Process (ASP) followed by adsorption on Activated Carbon (AC) were carried out. **Materials and Methods:** The ASP followed by sedimentation and then adsorption on AC were investigated. Determination of the optimum conditions for both ASP (time) and AC (time and dose) were determined. Also, kinetics and adsorption isotherm were investigated. **Results:** Operation of the ASP at MLSS of 3-4 g L⁻¹ and optimum detention time of 6 h produced a good quality effluent. The percentage removal of Mixed Liquor Suspended Solids (MLSS), Chemical Oxygen Demand (COD), Biological Oxygen Demand (BOD) and Total Suspended Solids (TSS) were 95.86, 94.53 and 97.6%, respectively. Post treatment using adsorption on AC with a dose of 2 g L⁻¹ and at a contact time of 45 min improved greatly the quality of the treated effluent. Residual values of COD, BOD and TSS were 38 mg O₂ L⁻¹, 9 mg O₂ L⁻¹ and 2 mg L⁻¹, respectively. Kinetic studies showed that adsorption process follow second order kinetic and data fits both Langmuir and Freundlich isotherm models. The maximum adsorption capacity (q_{max}) of COD over AC was 333.33 mg g⁻¹. **Conclusion:** The use of ASP followed by AC adsorption produced a high-quality effluent which can be either disposed safely into the agricultural drain or reuse in the industrial processes in the manufacturing plant.

Key words: Activated carbon, activated sludge process, remediation, retting flax wastewater, reuse

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Data Availability: All relevant data are within the paper and its supporting information files.

INTRODUCTION

Flax is a natural cellulosic fiber, non-lignified, lustrous, soft and pale yellow. Flax has a high economic value for the production of flax fibers and linseed oil. The composition of flax is almost 92% cellulose, 2% hemicelluloses, 4% lignin and 2% other constituents¹. 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 lignin². 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^{3,4}. These processes are applied for the production of fibers from plant materials such as flax (*Linum usitatissimum*) and hemp stalks and coir from coconut husks⁵.

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, 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 season⁶. 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 biodegraded⁷. However, there is little research work dealing with the biodegradation of organic matters in flax wastewater. Abou-Elala *et al.*² 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.*⁸ studied the performance of Up-flow Anaerobic Sludge Blanket (UASB) reactor for treatment of flax retting wastewater at Organic Loading Rates (OLRs) of 1.2-8.6 kg CODs m³/d and Hydraulic Retention Time (HRT) of 72 and 12 h. The Chemical Oxygen Demand (COD) removal rates ranged from 64.5-90.5%.

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)⁹. 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 uses¹⁰. In the current study, wastewater produced from a large flax company provides the material of this study. The wastewater produced (350 m³/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 m³/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 sub-surface 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 (APHA)"¹¹ were carried out for raw wastewater as well as the treated effluents. The analysis included pH, color, total and soluble COD, BOD, TSS, Total Phosphorous (TP), Total Kjeldahl Nitrogen (TKN), ammonia nitrogen (NH₄-N), oil and grease and all extractable

matters by chloroform (O and 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. The BOD was measured according to 5 days BOD test method (5210-B). The pH was measured using bench pH meter model Jenway 3505. The TKN was measured using mercuric sulfate digestion method followed by titration method (4500-Norg). Ammonia was measured according to method (4500-NH₃). The TP was measured according to the method (4500-C) while O and 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 L Plexiglas laboratory column¹¹. At the starting period, the columns were fed with pre-aerated sludge from a nearby wastewater treatment plant with an initial concentration of a(MLSS) 3-4 g L⁻¹, containing almost 75% of volatile matters. Air supply using air pump was adjusted to produce 2-3 mg L⁻¹ 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 (NH₄H₂PO₄) 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 1 h 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 micro-organisms were identified according to APHA¹¹.

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⁻¹ at different time intervals namely; 15, 30, 45, 60, 90 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 APHA¹¹.

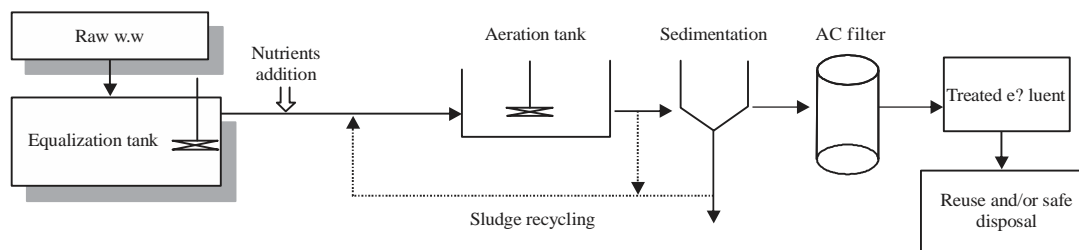


Fig. 1: Flow diagram of the treatment process

Statistical analysis: Statistical analysis of the collected data was carried out using Microsoft Excel, 2013 version. It is a measure of how widely values are dispersed from the average value (the mean). In other words; the mean is the statistic used most often to characterize the center of the data. Standard Deviation (SD) is commonly used to measure the variability of the data.

RESULTS AND DISCUSSION

Wastewater characteristics: Characteristics of the end-off-pipe were 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 wastewater¹². It is brownish to yellow in color, has bad odor and high organic content. The total COD ranged from 1272-6718 mg O₂ L⁻¹, while the higher BOD concentration was 3300 mg O₂ L⁻¹ and with a minimum value of 365 mg O₂ L⁻¹. The high organic content in wastewater is mainly due to cellulose, hemicelluloses and lignin in the flax. The TSS reached at 2800 mg L⁻¹. This is due to the presence of some residues of plant fibers. The O and G concentration reached 519 mg L⁻¹. This value is attributed to the presence of lignin and its derivatives released during retting process¹³. The mechanism of retting is mainly achieved by pectin enzymes produced by bacteria¹⁴. 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 occurs¹⁵.

Treatability study: An equalization tank was used before treatment due to the variation of wastewater composition as shown in Table 1. It homologized the wastewater 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.*¹⁶. The results depicted in Table 1 showed 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 oligoamines and monomers and (5) Reduction of the degree of polymerization and production of degradable products. The final products of assimilation process are shown in Eq. 1:



Determination of the optimum operating conditions for ASP:

The retting flax wastewater was acclimatized for a period of two weeks at a retention time of 24 h 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 O₂ L⁻¹, 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

Table 1: Characteristics of retting flax wastewater from different basins

Samples	Parameters						Minimum	Maximum	Mean ± SD
	Closed basins			Open ponds					
	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 ⁻¹)	3995	2060	3187	1272	6718	1565	1272.0	6718.0	3132.8 ± 2033.3
COD _s (mg O ₂ L ⁻¹)	2775	1680	3075	952	5820	947	947.0	5820.0	2541.5 ± 1838.4
BOD (mg O ₂ L ⁻¹)	1200	365	1480	675	3300	765	365.0	3300.0	1297.5 ± 1057.7
TSS (mg L ⁻¹)	122	140	63	192	2800	100	63.0	2800.0	569.5 ± 1093.6
TP (mg P L ⁻¹)	6	5	8	2	5	1.1	1.1	8.0	4.5 ± 2.6
NH ₃ (mg N L ⁻¹)	1.6	7.8	1.96	N.D	28	61	1.6	61.0	20.1 ± 25.3
TKN (mg N L ⁻¹)	45.6	30.2	39.2	20	33.6	213	20.0	213.0	63.6 ± 73.69
H ₂ S (mg L ⁻¹)	4	4	8.8	N.D	14	4	4.0	14.0	7.0 ± 4.5
O and G (mg L ⁻¹)	66	96	86.5	114	519	134.5	66.0	519.0	169.3 ± 127.9

*N.D: Not detected, COD: Chemical oxygen demand, BOD: Biological oxygen demand, TSS: Total suspended solids, TP: Total Phosphorous, , NH₃: Ammonia nitrogen, TKN: Total kjeldahl nitrogen, H₂S: Total Sulphide, O and G: Oil and grease

and rotifers (higher life forms) in the sludge floc is an indicator of a 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^{17,18}.

To determine the optimum time for biodegradation, the system was operated at different time ranged from 1 h up to 24 h. Results in Table 2 showed that the best removal rate of COD was achieved after 6 h. detention time. The mean residual value was 124 mg O₂ L⁻¹ 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 microorganisms¹⁹.

Efficiency of activated sludge process (ASP) at the optimum detention time:

The data in Table 3 showed 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 mg O₂ L⁻¹, 70 mg O₂ L⁻¹ and 12.5 mg L⁻¹. The total weight of sludge was 5.7 g L⁻¹ and with 3.3 g L⁻¹ volatile organics. The average SVI increased up to 75 which indicate the good settle ability 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 adsorption on activated carbon (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⁻¹) with different detention times were examined and the results were shown in Fig. 3. The adsorption capacity increased with time and COD levels decreased as well². Results revealed that the optimal contact time was 45 min with a residual COD concentration of 38 mg L⁻¹. To determine the optimum dose of adsorbent, several batches were carried out using different doses (1-6 g L⁻¹). The results illustrated in Fig. 4 showed that at a dose of 2 g L⁻¹, the best percentage removal of COD was 69.35%. Increasing the dose of adsorbent up to 4 g L⁻¹ resulted in a slight increase in capacity and

removal rate of COD. From the economic point of view, 2 g L⁻¹ at 45 min was selected as the optimum dose and optimum time.

Kinetics of biosorption: For realizing more data about the mechanism of adsorption and rate determining step, kinetic

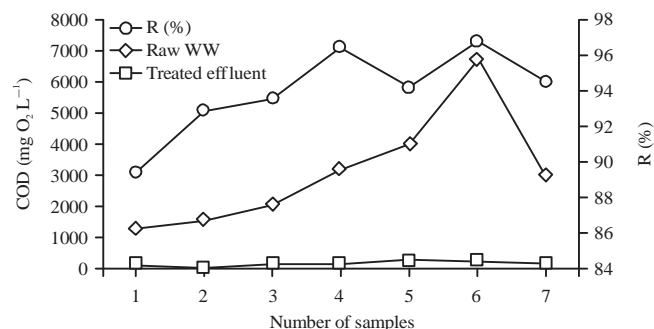


Fig. 2: Variation of COD values after acclimatization period and at 24 h aeration

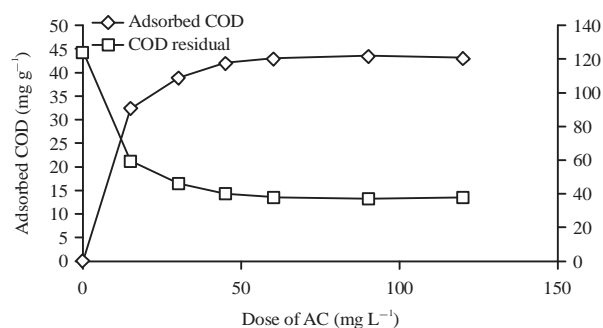


Fig. 3: Effect of detention time on adsorbed COD

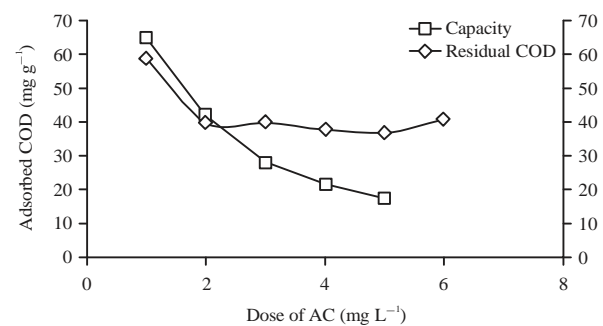


Fig. 4: Effect of adsorbent dosage on COD removal

Table 2: Determination of the optimum detention time for ASP

Parameters	Raw W.W	Time (h)									
		0	1	2	3	4	5	6	8	24	
COD _t (mg O ₂ L ⁻¹)	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 ⁻¹	35	35	36	38	41	45	45	48	40	

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 Eq. 2 and 3:

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

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

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

Figure 5 and 6 showed the kinetics model of COD adsorption on AC. The results revealed 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 $\text{COD}_{\text{residual}}$ from flax 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 Freundlich²⁰.

The Langmuir model was used to fit the adsorption of COD on AC. Figure 7 revealed that 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 $\text{COD}_{\text{residual}}$ 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^{-1} .

The isothermal model of Freundlich is established by the plot of $\log q_e$ vs. $\log \text{COD}_{\text{residual}}$ 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^{-1} , which is equal to q_e at $\text{COD}_{\text{residual}} = \text{unity}$. 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 revealed that both isotherm models are suitably represented the COD adsorption by AC.

The hydrophobic nature of AC due to complexation, ion exchange and hydrogen bonding increased the adsorption capacity which resulted in good quality effluent²¹. 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 O and G

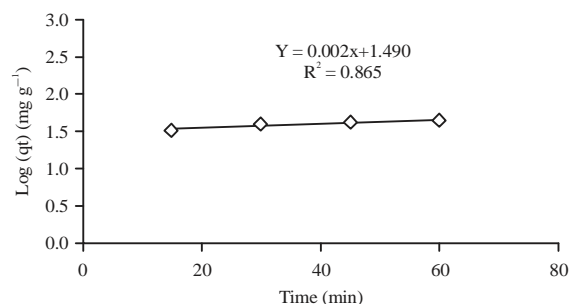


Fig. 5: First order kinetic of adsorption of $\text{COD}_{\text{residual}}$ from Flax wastewater

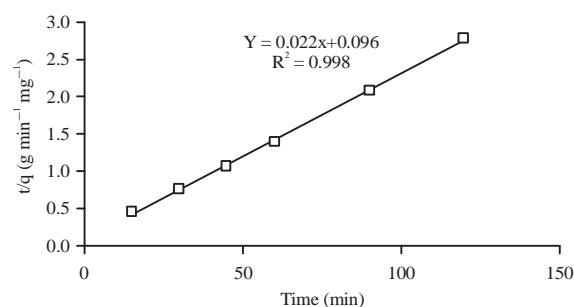


Fig. 6: Second order kinetic of adsorption of $\text{COD}_{\text{residual}}$ from flax wastewater

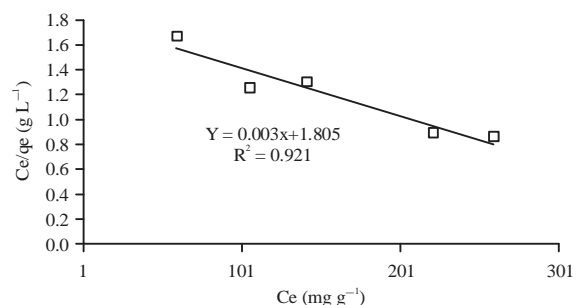


Fig. 7: Langmuir isotherm model of COD removal over AC

Table 3: Physico-chemical analysis of ASP effluent

Parameters	Raw WW	Treated effluent	R (%)
pH	6.1	8.8	-
Color (Co/Pt units)	138	Colorless	100.0
COD_t ($\text{mg O}_2 \text{ L}^{-1}$)	3000	124	95.86
BOD ($\text{mg O}_2 \text{ L}^{-1}$)	1280	70	94.53
TSS (mg L^{-1})	524	12.5	97.6
TP (mg N L^{-1})	4.1	0.8	80.48
NH_3 (mg N L^{-1})	20	N.D	100.0
TKN (mg N L^{-1})	61	5.8	90.49
H_2S (mg L^{-1})	4	0.8	80.00
O and G (mg L^{-1})	169	8.0	95.26

N.D: Not detected, *Average of 6 samples, COD: Chemical oxygen demand, BOD: Biological oxygen demand, TSS: Total suspended solids, TP: Total Phosphorous, NH_3 : Ammonia Nitrogen, TKN: Total kjeldahl nitrogen, H_2S : Total sulphide, O and G: Oil and grease

grease (Table 4). The treated effluent can be either disposed safely into the agricultural drain or reuse in the industrial processes in the manufacturing plant²².

Table 4: Overall efficiency of ASP followed by AC

Parameters	Raw WW	Biologically treated effluent	R (%)	Final effluent	R (%)	ECP code (501/2015)
pH	6.1	8.8	-	8	-	Not defined
Color (Co/Pt units)	138	10	92.75	Colorless	100	Not defined
COD (mgO ₂ L ⁻¹)	3000	124	95.86	38	98.73	Not defined
BOD (mgO ₂ L ⁻¹)	1280	70	94.53	9	99.29	<30
TSS (mg L ⁻¹)	524	12.5	97.6	2	99.61	<30
NH ₃ (mg N L ⁻¹)	20.0	N.D	100	N.D	100	Not defined
H ₂ S (mg L ⁻¹)	4.0	0.8	80	N.D	100	Not defined
O and G (mg L ⁻¹)	169	8.0	95.26	1	99.4	Not defined

N.D: Not detected, COD: Chemical oxygen demand, BOD: Biological oxygen demand, TSS: Total suspended solids, TP: Total Phosphorous, , NH₃: Ammonia nitrogen, H₂S: Total sulphide, O and G: Oil and grease

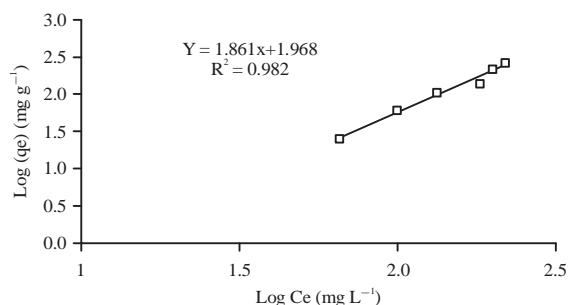


Fig. 8: Freundlich isotherm model of COD removal over AC

CONCLUSION

The production of flax fibers by water retting process is one of the industrial processes which consumes large amount of water. Wastewater produced contains high concentration of organic matters, TSS, color and has a bad odor. In this study wastewater was treated using ASP followed by adsorption on AC. The operation of ASP at a detention time of 6 h and MLSS 3-4 g L⁻¹ removed 95.86% COD, 94.53% BOD and 97.6% TSS with corresponding residual values of 124 mg O₂ L⁻¹, 70 mg O₂ L⁻¹ and 12.5 mg L⁻¹. To achieve a quality of effluent amenable for reuse, AC was used at a dose of 2 g L⁻¹ and contact time of 45 min. The quality of treated effluent improved greatly and the residual values of COD, BOD and TSS reached 38 mg O₂ L⁻¹, 9 mg O₂ L⁻¹ and 2 mg L⁻¹, respectively. The use of ASP followed by adsorption on AC proved to be a very promising technique for remediation and reuse of flax wastewater. The treated effluent can be disposed safely into agricultural drain according to the National Regulatory Standards. Furthermore, the treated effluent can be reused in the appropriate places in such industry.

SIGNIFICANCE STATEMENT

Water scarcity now-a-days is a major problem facing the developing countries especially Egypt. New sources of water must be found to reserve the fresh water resources.

Treated effluent from industrial processes is a very interesting solution. Remediation and reuse of retting flax wastewater (350 m³/day) was carried out successfully using ASP followed by adsorption on AC. This will help the decision makers to apply such solution for similar industries to save fresh water and protect the water resources from pollution.

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