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Comparative Studies on the Adsroption Properties of Powdered Activated Carbon and Propenoic Acid Modified Sawdust in the Treatment of Secondary Palm Oil Mill Effluent

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Abstract. Propenoic acid monomer was used to modify pulped cellulosic materials (sawdust). The sorption properties of the propenoic acid modified sawdust (PAMS) were compared with those of powdered activated carbon (PAC) in the tertiary treatment of palm oil mill effluent, previously clarified with iron(III) chloride plus lime (secondary effluent). The adsorption processes were effected in a fluidized bed reactor (FBR) at a pressure of 80 kiloNewton per meter square (kNm^{-2}). Optimum amount of PAC and PAMS used for the fluidized adsorption of contaminants from the secondary palm oil mill effluent (POME) were 2.5 g/l and 4.0 g/l, respectively. These sorption processes were found to be optimum at 10 min and 50 min for PAC and PAMS, respectively. At optimum sorption conditions, removal differentials of 28.6%/g chemical oxygen demand, 19.1%/g suspended solids, and 19.3%/g colour in favour of PAC were established. The application of optimum conditions for adsorption, for both adsorbents, to the bulk treatment of the palm oil mill effluent yielded a clear effluent with wider reuse applicability.

Keywords: metal adsorption, palm oil mill effluent, propenoic acid treated sawdust, fluidized bed reactor, activated carbon, wastewater treatment

Introduction

Adsorption processes are becoming viable options for the removal of non-biodegradable substances from conventionally treated wastewaters (Osuide *et al.*, 2003; Lopez-Delgado *et al.*, 1998; Allen and Brown, 1995; Lo and Lin,1989; AWTR-19, 1967). These substances exist as fine colloids or true solutes in these wastewaters, even after the wastewaters have been subjected to primary and secondary treatment processes.

Large-scale palm oil processing industries use waste stabilization ponds (WSPs) for the secondary treatment of their effluents. These ponds stabilize wastewaters through biodegradation processes. It has been observed that these pond systems, in typical cases, are quite elaborate in design and construction. The effluents so produced, do not fully meet the effluent discharge guidelines as stipulated by the Ministry of Environment, Nigeria (FEPA, 1988). With an average chemical oxygen demand (COD) value of 1096 mg/l, dissolved oxygen (DO) of 2.0 mg/l, and an oil and grease (O-G) value of 19,446 mg/l, in a typical study case, the usual treatment methods still leave a lot to be desired (Osuide, 2004).

Dependence on the natural purification schemes to continue the stabilization processes does not guarantee the protection of the environment owing to the large volume of wastewater involved. It also does not provide for the extension of available water resources via the possibility of recycling.

Studies have generally shown the removal differentials of between 45% and 75% for most water quality parameters in favour of chemical treatment of industrial wastewaters as against treatment by waste stabilization pond systems (AWTR-19, 1967). Besides, chemical treatment provides for the possibility of recycling of industrial effluents.

Powdered activated carbon (PAC) is the most popular adsorbent for removing such recalcitrant pollutants from solutions in tertiary treatment steps (Lo and Lin,1989; AWTR-19,1967). However, the cost of preparing PAC is becoming quite prohibitive. In recent times, alternative materials are being sought to replace PAC in adsorption processes. Cellulosic materials available in the form of agricultural byproducts are now the choice. The capacity of these cellulosic materials to take up contaminants, particularly heavy metal ions from synthetic and natural standard solution have been examined variously by people working in this area (Saeed *et al.*, 2005; Saeed and Iqbal, 2003; Saeed *et al.*, 2002; Pelekani and Snoeyink, 1999; Lopez-Delgado *et al.*,1998; Allen and Brown, 1995). The results show not much of a success story, though indicate the inherent potential. More recently, however, some workers have used active monomers and other reactive groups to modify cellulosic materials before application to sorption processes.

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Such monomers include cyanoethene, 2-propenoic acid, and other monomers of acidic and basic formaldehyde or thioglycollic groups. These have been found to considerably enhance the uptake capacities of the cellulosic materials thus modified. By and large, the applications of these have been also exterded to the treatment of standard synthetic metal ions solutions (Okieimen *et al.,* 1991; 1986; Randall *et al,* 1978). Owing to the continuous enlargement of agricultural produce processing industries, there is a need to investigate for cheeper alternative sorption substrates that can be employed in the tertiary treatment of industrial effluents in anticipation for a wider range of effluent reuse, or at least for a safer discharge into the environment.

Sorption properties of propenoic acid modified sawdust (PAMS) and those of powdered activated carbon (PAC) were investigated for their comparative ability for the tertiary treatment of chemically clarified palm oil mill effluent (POME).

Materials and Methods

The wastewater material. Palm oil mill processing effluent (POME) obtained from a palm oil mill industry in Benin City, Nigeria, was treated using experimentally determined optimum conditions in a preliminary study. The preliminary study included: determination of optimum time of sedimentation for POME over a time range of 10-100 min at 10 min intervals (60 min was found optimum); determination of the amount of $FeCl_3$ required as coagulant over a concentration range of 200-700 mg/l at 50 mg/l interval (350 mg/l $FeCl_3$ was found to be optimum at pH 4.6); and the amount of CaO (lime) required as flocculant over a range of 25-150 mg/l at 25 mg/l interval, in constant mixture with 350 mg/l $FeCl_3$ (75 mg/l CaO was found to be optimum at a mixture pH of 5.2). These optimum conditions were then applied in the treatment of raw POME via integrated physicochemical unit treatment processes. The unit treatment processes included sieving and sedimentation (primary treatment), coagulation and flocculation using 350 mg/l $FeCl_3$ plus75 mg/l CaO (lime) (secondary treatment) (Osuide, 2004; AWWA, 1989; Ademoroti, 1985). This effluent was sand-filtered to yield the secondary effluent used in the present study.

Preparation of the sorbent. The powdered activated carbon (PAC; Nentech product) was reactivated at 110 °C for 24 h before use. Sawdust, obtained from a local sawmill in Ekpoma town, Nigeria, was modified using 2-propenoic acid. The modification process was as follows: the raw sawdust was dried in the sun to constant weight. Methanol was used to extract excretory and fatty materials from the dried sawdust. 50.0 g portions of the extracted cellulose material was pulped for 1 h in 800 ml hot water using 18.0 ml ethanoic acid and 22.5 g sodium chlorite, which were added in three portions of 6.0 + 7.5 g, respectively, in a well ventilated hood (fume cupboard). The pulped sawdust (holocellulose) was filtered and air-dried, and then placed in a three-necked round bottom flask containing 36.0 ml of 0.1% (v/v) nitric acid. The flask was placed in a waterbath maintained at 40 °C. Ceric ammonium sulphate (0.674 g; equivalent to 0.26 g ceric ion, Ce^{4+}) was dissolved in 6.0 ml of 0.1% (v/v) nitric acid, which acted as the initiator, and added to the contents of the flask. The flask was shaken to ensure good dispersion. 2.0 g propenoic acid monomer was added to the flask, stoppered and shaken continuously for 5 min, and then shaken briefly at 10 min interverls for a total period of 1 h. After 1 h, the pulp was washed, filtered and air-dried at room temperature. The copolymer or homopolymer was later extracted using dimethylformamide over a period of 24 h, after which it was filtered and air-dried at room temperature to constant weight (Okieimen *et al.*, 1986). This yielded the propenoic acid modified sawdust (PAMS) used in the present studies.

Fluidized bed reactor. A portable laboratory model of a fluidized bed reactor (FBR) was designed and built using Perspex sheets, following the Pillai proceedure described in detail elsewhere with (Ademoroti, 1979), as shown in Fig 1. The main compartment of the FBR is divided into two parts: the lower and the upper chambers. The lower chamber is fitted with a funnel through which enters the compressed air into the system, then passing into the upper chamber through perforated partition. The upper chamber is the fluidization compartment where the chemically treated effluent and sorbents are mixed and fluidized by the compressed air from a compressor. The optimum fluidization pressure was earlier determined as 80 kilo Newton per meter square (kNm^{-2}) from trial runs (Osuide, 2004).

Sand filtration bed. Loose fine sand of aggregates of 0.5-1.0

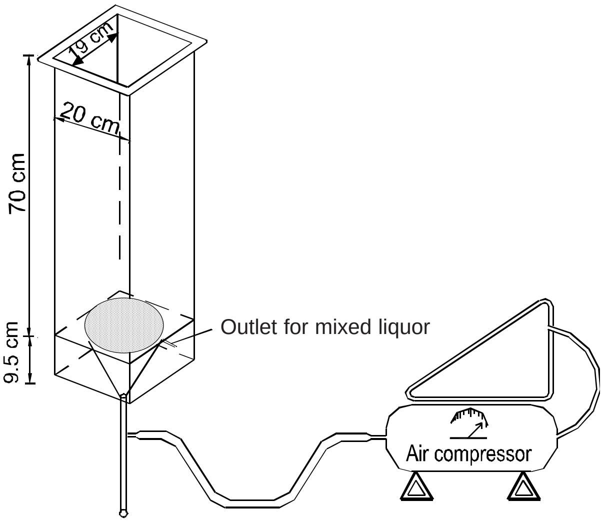

Fig. 1. Fluidized bed reactor (FBR) developed for the treatment of secondary palm oil mill effluent.

mm gradings was obtained from river dredgings. It was thoroughly washed with water and rinsed several times with distilled water. The washed sand was further leached with 1% (v/v) of hydrochloric acid at a rate of 1 ml per min using 300 ml of the acid in a glass column. It was then rinsed with distilled-deionised water until it became free of acid, after which it was sun-dried. The leached sand was packed into glass column of internal dia 6.71 cm. An optimum height of the sand column for the filtration of the sorptively fluidized palm oil mill effluent was obtained from test run as 90.0 cm. The total length of the glass column from tip of the tap to top was 81.0 cm. Two glass columns were used to accommodate the required 90.0 cm of sand column for filtration, by allowing each of the glass column to accommodate 45.0 cm height of the sand column. The total filtered liquid was passed through the two glass columns in series.

Batch procedure. Batch tests were carried out on one litre each of the secondary palm oil mill effluent to establish the optimum time (min) and the amount of adsorbents required for sorption fluidization in the FBR. Three important and quickly determinable parameters were used to ascertain optimum conditions for adsorption, which included: chemical oxygen demand (COD), suspended solids (SS), and colour (as absorbance at 465 nm). The optimal removal levels were based on percentage levels of the parameters. Four readings were taken in each case and the average value was calculated.

Optimum time for sorption fluidization in the FBR. With moderate amount of PAC or PAMS (2.0 g) the sample was fluidized for a given time (5, 10, 20, 30, 40, 50 and 60 min), at an optimum pressure of 80 kNm^2, for both the adsorbents. Following the sorption pattern observed for PAMS, sorption time for this sorbent was extended to 80 min (Ademoroti, 1979).

Optimum amounts of adsorbents for fluidization in the FBR. Different amounts of the adsorbents, PAC or PAMS, were used at the constant pressure of 80 kNm^{-2}, at the optimum condition of time for the respective adsorbents.

Analysis. The analytical procedures used for this study were as described by APHA (1989). Typical analysis of the secondary effluent (FeCl3 + lime pre-treated and sand filtered palm oil mill effluents is shown in Table 1.

Abbreviations used: TS (total solids), DS (dissolved solids), SS (suspended solids), DO (dissolved oxygen), BOD$_5$ (5-day biochemical oxygen demand), COD (chemical oxygen demand), NO$_3^-$-N (nitrate-nitrogen), SO$_4^{-2}$(sulphate), Cl$^-$ (chloride), PO$_4^{-3}$(phosphate), O and G (oil and grease), TBC (total bacterial count).

Results and Discussion

The secondary POME FeCl$_3$ (coagulated + lime treated) had a light orange colour and could only manifest minimal sediments when left to stand overnight. The data shown in Table 1 depict a better quality effluent in comparison with what the processing industries usually let out into the environment. This, however, still falls short of the guidelines set out by the Ministry of Environments, Nigeria (Table 1). The reuse possibility of

Table 1. Analysis of the FeCl$_3$ + lime pre-treated and sand filtered palm oil mill effluent (secondary effluent)

Parameters	Range of values	Mean	SD	SE	FEPA limits*
Colour (nm)	0.199-0.26	0.202	0.060	0.027	-
Odour	slightly rancid, oily odour	-	-	-	-
Temperature (°C)	23-28	25.75	2.217	0.992	-
pH	5.0-5.9	5.6	22.236	9.944	6.9
Turbidity (NTU)	41.6-59.2	51.28	7.352	3.288	-
Conductivity (µS/cm)	(0.5-0.7)x10^3	0.61 x10^3	0.098	0.044	-
Total solids (mg/l)	360-520	440.00	67.330	30.111	2000
Suspended solids (mg/l)	306-501	367.7	52.009	23.259	30
Dissolved solids (mg/l)	153-189	172.25	15.435	6.903	2000
Dissolved oxygen (mg/l)	2.8-3.4	3.08	0.250	0.112	-
BOD$_5$ (mg/l)	72-101	89.50	13.429	6.006	30
COD (mg/l)	300-306	305.00	15.055	6.733	50
Nitrrate-N (mg/l)	1.09-1.25	1.193	0.071	0.032	10
Sulphate (mg/l)	0.3-1.32	0.780	0.435	0.195	500
Chloride (mg/l)	120-150	136.75	13.841	6.190	600
Phosphates (mg/l)	1.3-1.32	1.318	0.024	0.011	5.0
Oil and grease (mg/l)	600-620	611.75	20.156	9.014	10
TBC (per 100 ml)	(0.60-1.60) x 10^7	1.100 x 10^7	4.761x 10^6	5.214 x 10^6	-

* = FEPA (1988); TBC = total bacterial count; SD = standard deviation; SE = standard error; NTU = nephelometric turbidity unit

this effluent are still limited, hence the necessity for tertiary treatment. The values of SS (367.7 mg/l), BOD_5 (89.50 mg/l), COD (305 mg/l), and O-G (611.0 mg/l), are above the mandatory limits (FEPA, 1988). All the other values, however, fall below the guideline limits for the respective parameters.

The TBC test was aimed at highlighting the presence of bacterial load, which may be involved in the accoumpanying biodegradation processes which could be occurring along with chemical/adsorption treatment processes.

The variation of sorption levels with time, using PAC and PAMS, respectively, are shown in Fig. 2 and Fig. 3.

Optimum removal of contaminants was observed to be at 10 min fluidization for PAC adsorption. The rapid rise in uptake level between 5 and 10 min typically depicts a physical process, which also suggests saturation of adsorption surface beyond 10 min.

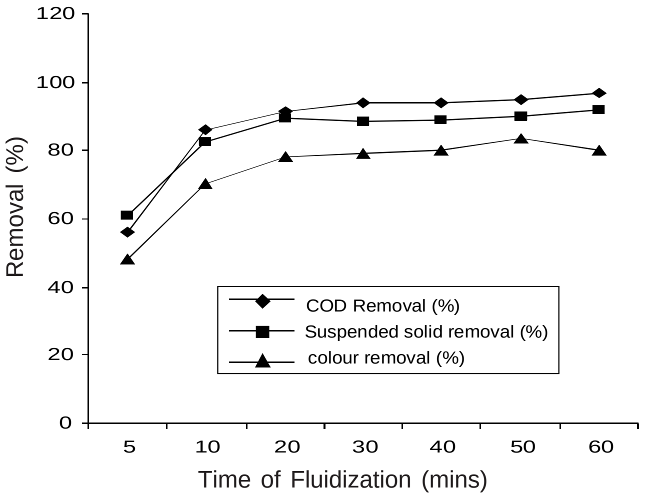

Fig. 2. Effect of variation of time of contact between 2.0 g powdered activated carbon (PAC) and the secondary palm oil mill affluent per liter during fluidization at 80 kNm^{-2} pressure.

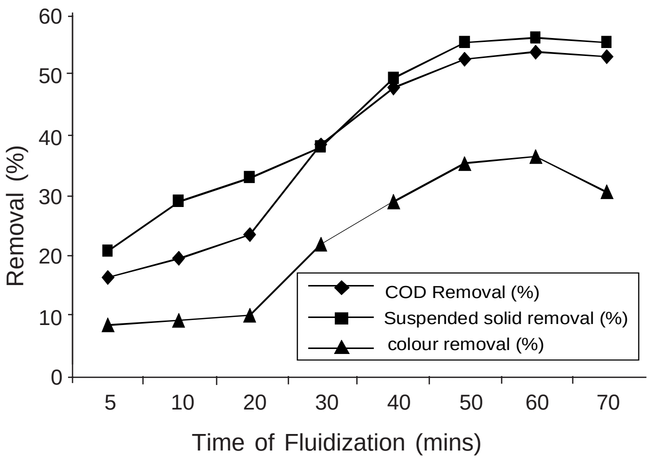

Fig. 3. Effect of variation of time of contact between 2.0 g propenoic acid modified sawdust (PAMS) and the secondary palm oil mill effluent per litre during fluidization at 80 kNm^{-2} pressure.

The uptake of materials from solution by PAMS was more gradual, and the curves rose steadily from 5 min through 10 min and attained peaks at 50 min (Fig. 4). Beyond 60 min, no significant rise in the curves was noticed even up to 80 min. The differences in the uptake pattern (Fig. 2, Fig. 3), suggest different uptake mechanisms for the two adsorbents. Chemisorption is more probable with PAMS. Fig. 4 and 5 illustrate, respectively, the optimum amount of PAC and PAMS required for the sorption clarification of the secondary effluent.

These show narrower range of variation for the amount of PAC required than is needed for PAMS. Preliminary studies indicate an optimum range for PAC between 2 and 3 g; 2.5 g PAC yielded optimum removal for COD, SS and colour from the effluent. But with PAMS, 4.0 g was required for even lower level percentage removal.

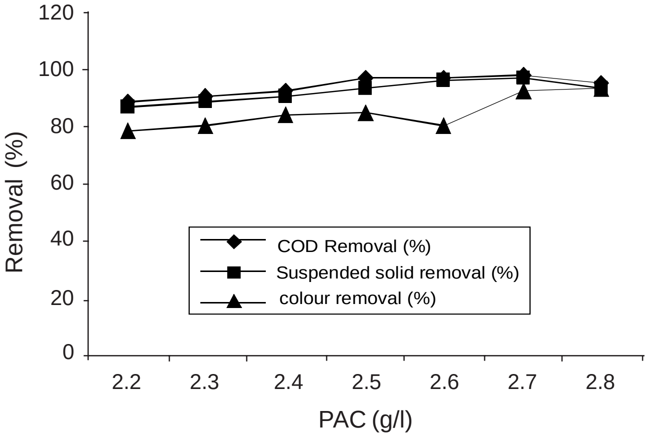

Fig. 4. Optimum amount of powdered activated carbon (PAC) required for the fluidization of secondary palm oil mill effluent at 10 min.

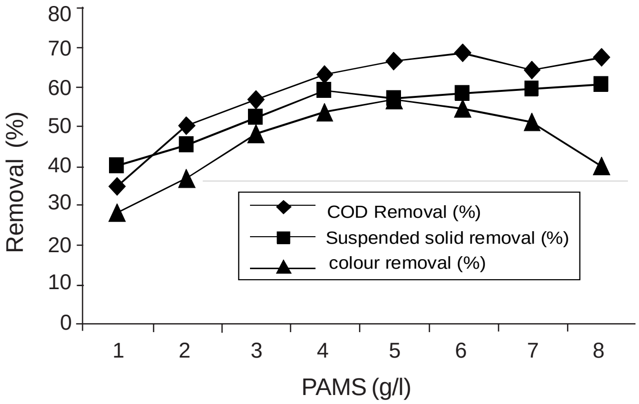

Fig. 5. Optimum amount of propenoic acid modified sawdust (PAMS) required for the fluidization of secondary palm oil mill effluent at 50 min.

Tables 2 and 3 show the level of clarification by PAC and PAMS, respectively, against the initial values as shown in Table 1.

It has been shown that during the sorption process, the non-biodegradable substances, which could inhibit biodegrading processes (enzyme inhibition), are more favourably removed by adsorption from the effluent medium, leaving the effluent medium more favourable for microbial breakdown of the contaminant materials (Suidan *et al.*, 1996) This dual clarification process leaves the effluent clearer than only the adsorption process would have achieved.

The sorption patterns obtained in the present studies suggest different sorption mechanisms for PAC and PAMS. The high

Table 2. Analysis of the powdered activated carbon (PAC) fluidized secondary palm oil mill effluent at 10 minutes

Parameters	Range of values	Mean	SD	SE	Reduction (%)
Colour (nm)	0.016-0.022	0.01925	0.002754	0.001377	92.5
Odour	slightly rancid oily odour				-
Temperature (°C)	23-27	24.75	2.061553	1.030776	-
pH	6.1-6.5	6.325,7.0^a	0.170783	0.085391	-
Turbidity (NTU)	20-28	24.5	3.696846	1.848423	53.5
Conductivity (µS/cm)	(0.13-0.39) x 10^3	0.28	0.119164	0.059582	63.3
Total solids (mg/l)	105.3-162.4	135.925	23.70758	11.85379	81.1
Suspended solids (mg/l)	45.3-95.3	70.45	21.48309	10.74155	70.9
Dissolved solids (mg/l)	60.0 67.1	63.3	2.947315	1.473658	84
Dissolved oxygen (mg/l)	5.2-6.0	5.605	0.408289	0.204145	75.8*
BOD$_5$ (mg/l)	6.0-7.8	6.8975	0.805828	0.402914	92.7
COD (mg/l)	28-33	30.525	2.214159	1.107079	89
Nitrate-N (mg/l)	0.1-0.6	0.4375	0.203695	0.101848	65.8
Sulphates (mg/l)	0.01-0.04	0.025	0.01291	0.006455	85.7
Chlorides (mg/l)	0.21-0.29	0.2525	0.038622	0.019311	78.5
Phosphates (mg/l)	0.61-0.73	0.675	0.051962	0.025981	46.6
Oil and grease (mg/l)	86-90	88.25	1.666333	0.833167	85.5
TBC (per 100 ml)	(0.09-0.15)x10^6	1.250 x 10^5	26457.51	2.646 x 10^4	99.1

* = increase %; ^{a} = adjusted pH; TBC = total bacterial count; NTU = nephelometric turbidity unit

Table 3. Analysis of the propenoic acid modified sawdust (PAMS) fluidized secondary palm oil mill effluent at 50 minutes

Parameters	Range of values	Mean	SD	SE	Reduction (%)
Colour (nm)	0.066-0.071	0.06875	0.002217	0.000573	70.8
Odour	slightly rancid oily odour	-	-	-	-
Temperature (°C)	25 -28	26.225	1.265899	0.326854	-
pH	5.99-6.3	6.1225;7.0^a	0.152834	0.039462	-
Turbidity (NTU)	38-46.4	42.025	3.720551	0.960642	15
Conductivity (µS/cm)	(0.38-0.44) x 10^3	0.41	0.02582	0.006667	33.3
Total solids (mg/l)	250-360	295.75	46.20516	11.93012	48.5
Suspended solids (mg/l)	61-106	91.25	20.83867	5.38052	37.2
Dissolved solids (mg/l)	189-254	220.5	31.39533	8.106239	49.4
Dissolved oxygen (mg/l)	3.80-4.0	3.915	0.086987	0.02246	18.5*
BOD$_5$ (mg/l)	80-112	95.175	13.97053	3.607176	9.6
COD (mg/l)	200-260	229.25	29.22756	7.546522	21.3
Nitrate-N (mg/l)	1.0-1.20	1.135	0.091469	0.023617	1.7
Sulphates (mg/l)	0.08-0.12	0.0975	0.017078	0.00441	47.6
Chlorides (mg/l)	0.51 0.72	0.6275	0.096047	0.024799	52.4
Phosphates (mg/l)	0.80-1.0	0.91	0.088694	0.022901	29
Oil and grease (mg/l)	99.5-268	184.475	71.69009	18.5103	57.5
TBC (per 100 ml)	(0.10-1.2)x10^7	5.03 x 10^6	3.229 x 10^5	3.33 x 10^6	91.5

* = increase %; ^{a} = adjusted pH; TBC = total bacterial count; NTU = nephelometric turbidity

amount of the required PAC might have been caused by the size of the colloidal and aqueous contaminants, most of which at this stage exist as contaminant coagulant complexes. These large molecular moieties and the oily macromolecular structures may have sizes that are too large for the micropores of the PAC. It follows that possibly only the surface adsorption occurs. Therefore, if surface unimolecular coverage is the uptake mechanism, then quite reasonable quantity of PAC would be required to achieve a clear solution. The initial rapid uptake at 0-10 min suggests a physical process that can be interpreted using the Freundlich isotherms model, however, the heterogenous nature of the effluent makes uniform interpretation difficult.

Cellulosic materials have been shown to be a repository of some reactive groups such a >C=O, -CHO, -HS, -OH, etc. Some of these might still remain after treatment of the cellulosic material to provide sites for contaminant uptake via a formal bond (Lopez-Delgodo *et al.*, 1998). Besides, the grafted moiety presents a hydrophobic portion and hydrophilic functional group: ($-CH_2-CH_2-COOH$). Hence, it is proposed here that H^+ ion likely destabilizes the electrical colloidal and aqueous states of the contaminants. These become more disposed to be chemisorbed on active sites and physiosorbed on hydrophobic matrices on the PAMS. At the optimum sorption time and amount of adsorbents, a comparison between the adsorptive capacities of PAC and PAMS shows a removal differential of 28.6%/g COD, 19.1%/g SS and 19.3%/g colour in favour of PAC. That less than 30% removal differential per gram adsorbent was observed between the adsorbents suggests that modified cellulosic materials could be used as substrates for tertiary clarification of secondary effluents. It is obvious that a follow-up disinfection step to eliminate the microbial community, will precipitate left-over contaminants and "shelf" the effluent. This will result in enhancive the effluent quality and increase its reuse applicability.

Conclusion

Modified cellulosic materials are capable of yielding sorbents with uptake capacities comparable with those of powdered activated carbon. With an uptake differential of less than 30% per gram, 2-propenoic acid modified sawdust portends a viable alternative to PAC.

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