



Evaluation of *Luffa Cylindrica* Fibers in A Biomass Packed Bed for The Treatment of Paint Industry Effluent Before Environmental Release

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ABSTRACT

The aim of this study was to investigate the potential of *Luffa Cylindrica* fibers in a biomass packed bed for the treatment of paint industry effluent before releasing into the environment. The fibers were modified by mercerisation in 0.5M NaOH for 24 h. Both modified and unmodified fibers were characterised using Scanning Electron Microscopy with Energy-Dispersive X-ray Spectroscopy (SEM-EDS). A biomass packed bed was prepared for the study with a packing factor of 0.0617 and 0.0550 for the modified and unmodified fibers respectively. Measured parameters were the pH, colour, total dissolved solids (TDS), total suspended solids (TSS), dissolved oxygen (DO), chemical oxygen demand (COD) and biochemical oxygen demand (BOD). Negative findings were achieved for TSS, DO and COD whilst positive findings were achieved for pH, colour, TDS and BOD. The extended residence time of 48 h was only of advantage for colour and BOD removal. In the domain of the positive results, the untreated fibers achieved 35% colour reduction, 5% TDS reduction and 77% BOD reduction all in 2 h. The treated fibers achieved 35% colour reduction in 30 minutes, 32.5% TDS reduction in 2 h and 82% BOD reduction in 30 minutes. The fibers treated with NaOH performed better in all indices where positive results were achieved except for pH. *Luffa cylindrica* fibres can be used as effective packing material in a biomass filter for the treatment of paint industry effluent before releasing into the environment based on WHO limits.

Keywords: biomass, industrial effluent, *Luffa Cylindrica*, mercerisation, packed bed

INTRODUCTION

Wastewater has been explained as water that has been used and contains dissolved or suspended waste materials or water that has dwindling quality due to the adverse effect of anthropogenic activities (Ighalo and Adeniyi 2020c; Singh and Gupta 2017). Razak (2010) reported that about 60% - 85% of water usage becomes wastewater. In paint manufacturing industries, water finds application as a solvent and also for rinsing after which it is discharged (Ntwampe *et al.*, 2014). The wastewater contains hazardous substances that contribute to the high level of chlorinated aromatic compounds (Payan *et al.*, 2018a; Shojaie *et al.*, 2018), suspended solids, heavy metals (Babatunde *et al.* 2019), biological oxygen demands, chemical oxygen demand and low level of dissolved oxygen in the aquatic bodies (Darajeh *et al.*, 2016; Devi *et al.*, 2008; Hami *et al.*, 2007). Majorly, this wastewater is discharged without treatment due to the high cost and complexity associated with conventional treatment of wastewater (UNESCO, 2017). The use of biomass is typically simple, low energy, cheap and effective method of increasing the quality of wastewater before discharge by reducing impurities and absorbing heavy metals (Eletta *et al.*, 2020; Ighalo and Adeniyi, 2020a). Biomass can be used as precursors for developing other advanced materials for water treatment (Ashouri *et al.*, 2019; Ghasemipour *et al.*, 2020). However, there are simpler water treatment applications of biomass such as in packing materials for packed beds.

Dizge *et al.* (2011) utilised microporous polyurethane sponge in their packed bed in a hybrid system coupled with membrane ultrafiltration at a detention time of 16 minutes. They observed that the removal efficiency decreased with decreasing loading and membrane ultrafiltration improved the performance of the process. Ghasemi *et al.* (2011) obtained a removal efficiency of 78% for the biosorption of uranium after 3 h using a packed bed filled with *Cystoseira indica* biomass. Rahmah and Abdullah (2011) achieved 99% COD reduction and 87% turbidity reduction using a packed bed filled with *Ceiba pentandra* (L). Gaertn. (kapok) for the filtration of oily water. A similar study on oily water was conducted by Srinivasan *et al.* (2012) to coalesce the emulsion in a packed bed using *Mucor rouxii* Biomass. Simate and Ndlovu (2015) utilised a packed bed filled with immobilized cassava peel waste to investigate heavy metal removal in aqueous media by continuous flow experiment. They achieved biosorption capacities of

Table 1. Properties of the effluent

Parameter	Value
pH	8.15
TDS (mg/l)	585
TSS (mg/l)	2.85
DO (mg/l)	6.20
COD (mg/l)	20.0
BOD (mg/l)	4.60
Colour (TCU)	5.04

99.6, 116.2 and 132.8 mg/L for Co^{2+} , Cr^{3+} and V^{3+} respectively. Sooksawat *et al.* (2017) investigated the biosorption of Pb^{2+} and Cd^{2+} in a packed bed filled with *Chara aculeolata* biomass under continuous flow configurations. The system was still able to achieve 98% for Pb^{2+} and 100% for Cd^{2+} in the third cycle of reuse.

From the findings of some recent studies, *Luffa Cylindrica* is an effective biomass material for the removal of pollutants from aqueous media (Obboh and Aluyor, 2009; Obboh *et al.*, 2009). It is cheap and readily available within the context of this study. Furthermore, it does not have a competitive use except as a local sponge for bathing. Based on this review, *Luffa Cylindrica* has not been used as a packing material in biomass packed bed filters. The fibrous/spongy nature of the filter ensures that it is an excellent packing material for a biofilter. *Luffa* fiber was chosen as the filter media due to its fibrous vascular internal structure which can absorb these impurities thereby improving water quality. The understanding of the effectiveness of this media would be beneficial in water treatment systems.

With the scope of the authors' search, *Luffa Cylindrica* fibers have not been utilised as a packed bed for water treatment. This is an important novelty of the current study. The aim of this study was to investigate the potential of *Luffa Cylindrica* fibers in a packed bed for the treatment of paint industry effluent before releasing into the environment. The fibers were modified by mercerisation. Both modified and unmodified fibers were characterised using Scanning Electron Microscopy with Energy-Dispersive X-ray Spectroscopy (SEM-EDS). Parameters measured were the pH, colour, total dissolved solids (TDS), total suspended solids (TSS), dissolved oxygen (DO), chemical oxygen demand (COD) and biochemical oxygen demand (BOD). Besides the advantage of water pollution remediation, the study also recycled waste PET bottles for the development and design of the packed bed. This is an added advantage as it reduces the cost of design and offers an alternative use for the non-biodegradable environmental waste.

MATERIALS AND METHODS

Materials

Luffa Cylindrica fruits were obtained from the communities in Ilorin, in Kwara state. Industrial paint wastewater sample was obtained from a paint production facility in Ikeja, Lagos State. The properties of the effluent in terms of the indices of interest in this study are reported in **Table 1**. Distilled water and the Sodium Hydroxide (NaOH) of analytical grade were also used for the study.

Preparation of *Luffa Cylindrica* Fibres

The dried *luffa* was peeled from the dried fruit pod manually and cut into smaller pieces. The seeds were removed completely from the smaller pieces. A batch of the fibre was set aside in a bowl for mercerization (pre-treatment) and another batch for washing with distilled water. Mercerization was done with sodium hydroxide solution (NaOH) which was prepared by dissolving 200 g of NaOH in 10 L of distilled water to obtain a 0.5 M solution. The *luffa* fibres were immersed in this solution for 24 h at room temperature after which it was removed and soaked in distilled water for another 24 h and then sundried till constant weight. Similar treatment concentration and residence time have been employed in other studies for biomass modification (Azwa and Yousif, 2019; Mittal and Chaudhary, 2018; Yahaya *et al.*, 2015). The other batch of *luffa* fibres were soaked in distilled water for 24 h and sun-dried till constant weight. The two different batches of *luffa* fibres were then cut into smaller, tiny pieces and stored separately in well-labelled containers.

Characterisation of the *Luffa* Fibres

The biomass fibers were characterised by Scanning Electron Microscopy with Energy-Dispersive X-ray Spectroscopy (SEM-EDS) were used to determine the surface morphology and the composition of the fibers. SEM-EDS (SEM, Phenom ProX, Phenom-World BV, Netherlands) was used at an acceleration voltage of 15 kV and a magnification of 1000× (Adeniyi *et al.*, 2020).

Design and Development of Filter Column

A PET (Polyethylene terephthalate) plastic vessel measuring 750 cm³ volume was used in the construction of the filter bed, a ball valve was used at one end as the sampling point and the other end was designed to be an openable cap for the inlet passage of the substrate feed (paint industry effluent). A schematic diagram of the filter column is shown in **Figure 1**. The tiny pieces of the *luffa* fibres were weighed. A packing factor of 0.06 was used for developing the packed bed in the filter column for the mercerised (treated) *luffa* fibres and the packed bed with non-mercerized (untreated) fibres was 0.055. The weighed *luffa* fibres, 46.3 g for treated fibre was carefully stuffed and packed in the filter column and 41.3 g for untreated fibre in the second column. The

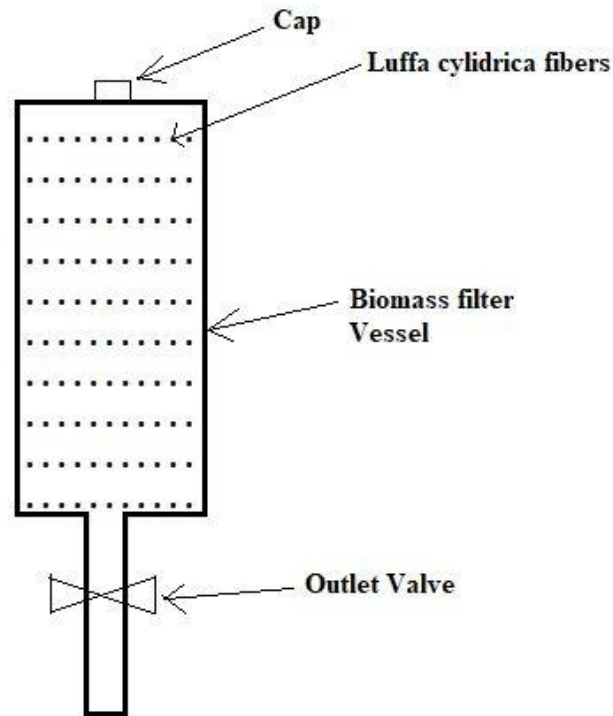


Figure 1. Schematic representation of the packed bed filter column

maximum possible packing achieved by human compression of the fibers was utilised while loading the packing material. The differences in the packing factors were due to the stiffness of the fibers.

Experimental Procedures

Two batch experiments were carried out to investigate the reduction of Total Suspended Solids (TSS), Total Dissolved Solids (TDS), Biological Oxygen Demand (BOD), Chemical Oxygen Demand (COD), pH and Colour of the wastewater sample using the treated and untreated *luffa* fibres. The packing factor of the packed bed with treated fibres was 0.06 and that of the packed bed with untreated fibres was 0.055. The two columns were mounted vertically using a retort stand and the feed (paint wastewater) was loaded from the top flowing downwards. The ball valve at the bottom of the column remained in a closed position. Samples were collected at 30 minutes' interval for 2 h by opening the ball valves and collecting in a properly labelled sample bottle for the two setups. A final sample was collected after 48 h of trial experimenting. All experiments were conducted twice and the reported results in the discussion section are average values of these runs. Standard error bars are included in the data plots in the results section.

The 48 h experiment trial experiment was conducted to determine if any performance advantage can be gained by longer residence time. After the tests, the parameters measured were the pH, colour, total dissolved solids (TDS), total suspended solids (TSS), dissolved oxygen (DO), chemical oxygen demand (COD) and biochemical oxygen demand (BOD).

RESULTS AND DISCUSSION

Surface Morphology of *Luffa Cylindrica* Fibres

SEM analysis was used to investigate the morphological structure of the fibres when subjected to mercerisation. The SEM micrographs of untreated *luffa* fibre shown in **Figure 2a** show a smooth amorphous waxy/gummy layer on the surface and packed fibre structure indicating the possible presence of lignin (Mohanta and Acharya, 2016). Though heterogeneous in appearance, symmetrical formations are observed on the surface due to the inherent structure of the biomass material. After modification by alkali (NaOH), the surface of the *luffa* cylindrical fibre became clean and smoother as shown in the SEM micrograph in **Figure 2b** which indicates the possible absence of gummy/waxy substance and lignin as compared to the untreated fibre (Adeniyi *et al.*, 2019b). The packed structure is of the untreated fiber is collapsed and parallel sheets are observed in the surface morphology. Mercerisation helps to remove the solubles and improve the wettability of the fibers (Adeniyi *et al.*, 2019a). The roughened appearance of the fibres suggests that it would give a better performance in water treatment as the surface area would have likely been increased raising its capacity for different pollutants (Ighalo and Adeniyi, 2020b).

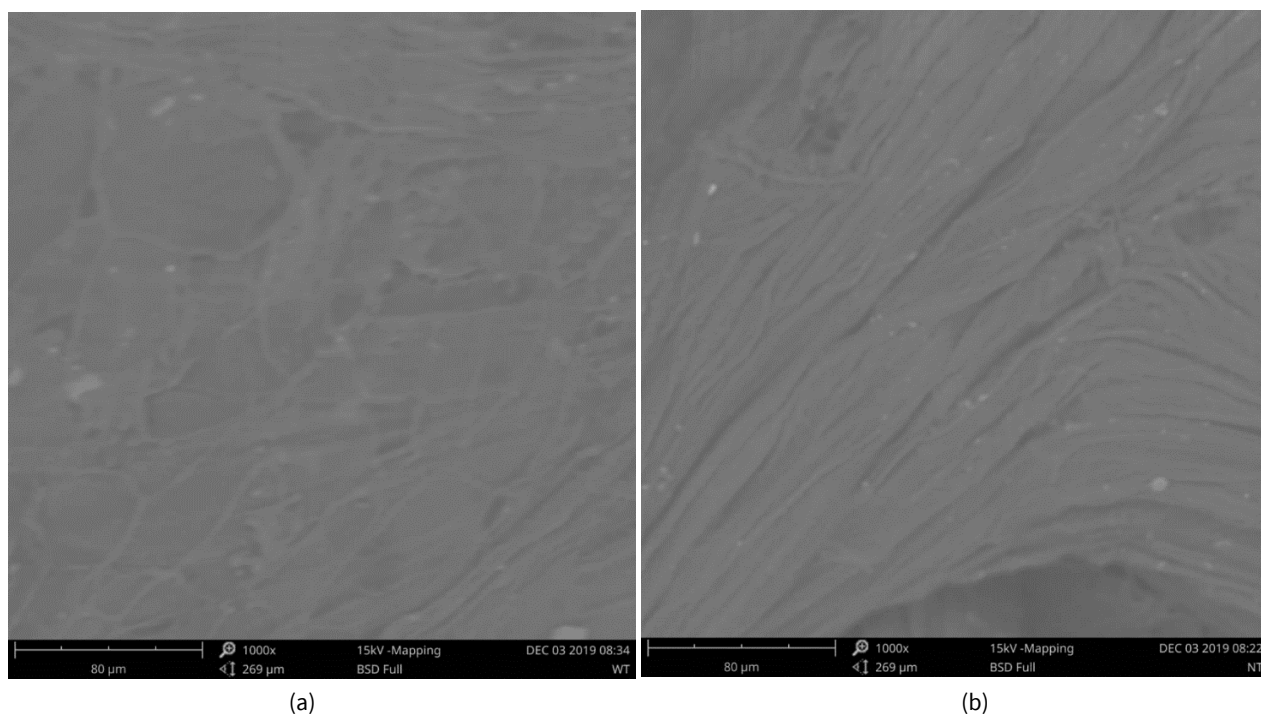


Figure 2. a. SEM micrograph for untreated *luffa cylindrica* fibre b. SEM micrograph for NaOH treated *luffa cylindrica* fibre

Table 2. EDS Analysis of untreated and treated *luffa cylindrica* fibre

Element Number	Element Symbol	Element Name	Untreated <i>luffa cylindrica</i> fibre		Treated <i>luffa cylindrica</i> fibre	
			Atomic Conc.	Weight Conc.	Atomic Conc.	Weight Conc.
6	C	Carbon	80.67	59.80	80.72	60.18
30	Zn	Zinc	1.35	5.45	1.57	6.36
19	K	Potassium	2.22	5.36	1.16	2.82
20	Ca	Calcium	1.96	4.86	2.72	6.77
47	Ag	Silver	0.67	4.44	0.79	5.30
17	Cl	Chlorine	1.58	3.45	0.78	1.72
8	O	Oxygen	3.18	3.14	5.06	5.03
16	S	Sulphur	1.08	2.13	0.95	1.89
7	N	Nitrogen	2.25	1.95	-	-
14	Si	Silicon	1.02	1.77	1.08	2.88
13	Al	Aluminium	1.00	1.66	0.74	1.24
26	Fe	Iron	0.41	1.42	0.00	0.00
12	Mg	Magnesium	0.90	1.36	0.58	0.87
15	P	Phosphorus	0.66	1.27	0.92	1.76
11	Na	Sodium	0.74	1.06	2.94	4.19
22	Ti	Titanium	0.29	0.87	0.00	0.00

Composition of *Luffa Cylindrica* Fibres

EDS can show the elemental mapping of a material (Payan *et al.*, 2018b) on a hydrogen-free basis. The carbon content and inorganic elements present in the treated and untreated *luffa cylindrica* fibre samples are presented in **Table 2** as analysed by EDS. The corresponding spectra are shown in **Figures 3a-b**. The EDS reveals that the surface of the fiber contains some chemical elements with carbon having the highest proportion of about 60 wt% in both the treated and untreated *luffa* fibre as shown by the EDS spectrums. The modification of the *luffa cylindrica* fibre by NaOH removed elements such as iron (Fe) and titanium (Ti) completely and reduced the atomic concentration of potassium (K), chlorine (Cl), sulphur (S), aluminium (Al) and magnesium (Mg). However, this analysis has established that there is not much compositional difference and chemical transformation between the treated and untreated fibers.

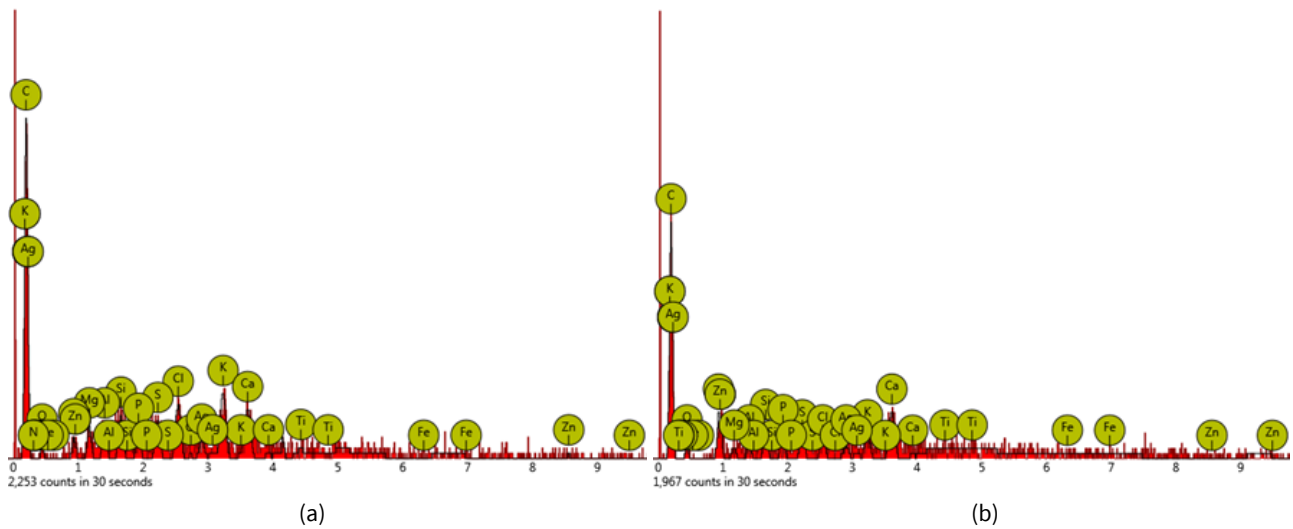


Figure 3. a. EDS spectrum for untreated *luffa cylindrica* fibre **b.** EDS spectrum for NaOH treated *luffa cylindrica* fibre

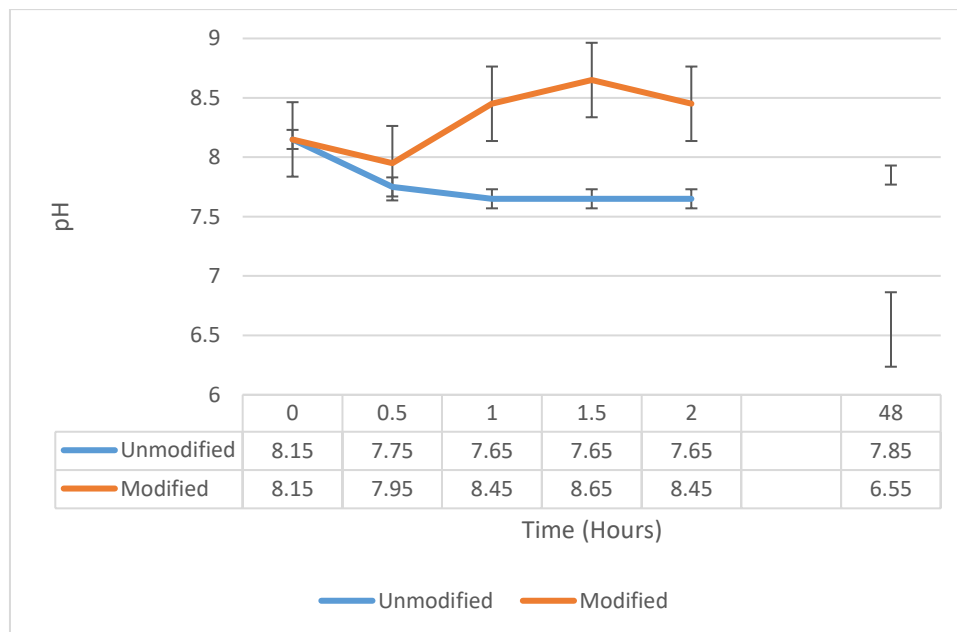


Figure 4. Effect of *luffa cylindrica* packed bed treatment on effluent pH

Effect on pH

The pH is the degree of acidity or alkalinity of the solution (Boyd *et al.*, 2011). The effect of the treatment of paint industry effluent by untreated and treated *luffa cylindrica* fibres on pH is shown in **Figure 4**. It can be observed that the untreated fiber is more efficient in regulating the effluent pH back to 7. Within the 2 h of experiments, the pH was reduced from 8.15 to 7.65. However, the NaOH modified fiber was observed to slightly raise the pH of the effluent. This could be due to the alkaline modification of the fibers affecting the solution chemistry of the effluent (despite the rinsing of the fibers in distilled water for 24 h after mercerisation). The interaction and removal of H^+ in solution with the negatively charged surface of biomass filtration media could lead to pH rise (Tang *et al.*, 2010). The 48 h trial experiment showed that this effect is mitigated in the long terms as the solution gradually drops to allowable pH levels for effluent. It can be surmised that both fibers do not present a pH disadvantage when used as packing material for the treatment of paint industry effluent in a biomass packed bed.

Effect on Colour

The effect of the treatment of paint industry effluent by untreated and treated *luffa cylindrica* fibres on colour is shown in **Figure 5**. From the results, the treated fibers reduced the colour of the effluent within 30 minutes of the experiment (reduction from 5.04 to 3.30 TCU) and did not give an advantage after this. However, the best performance of the modified fibers was achieved in the 48 h trial experiment. The untreated fiber was less suitable for the mitigation of colour in the effluent achieving a reduction from 5.04 to 3.30 TCU in 2 h. Colour rise observed beyond the optimum time threshold could be due to the desorption of the pigments from the fibers back to the solution. It can be surmised that the NaOH treated fibers was more suitable in removing colour from the effluent than the untreated fibers. This is because the mercerisation exposes more short length crystallites and

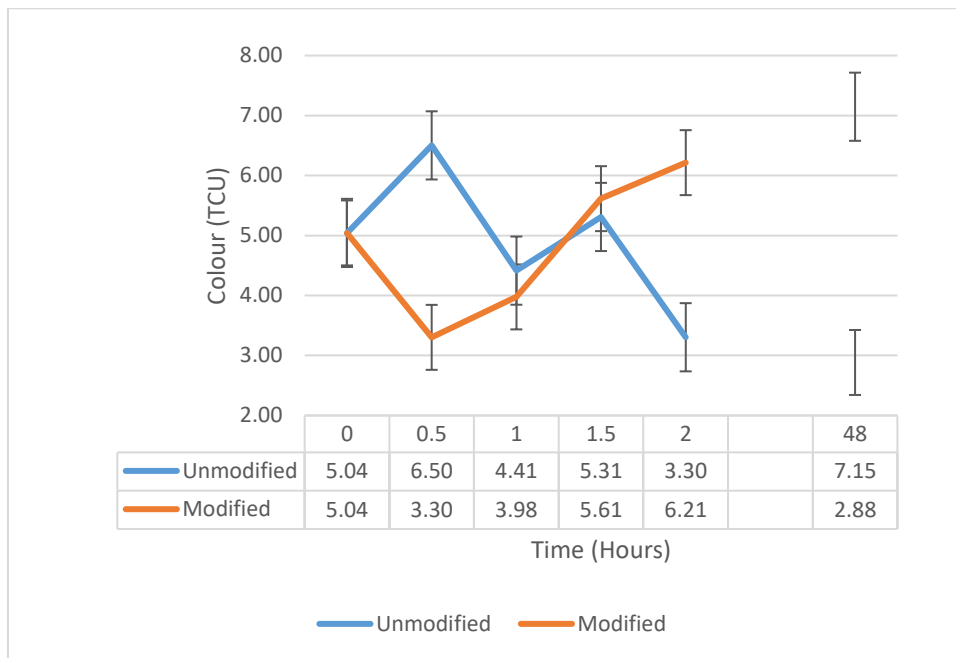


Figure 5. Effect of *luffa cylindrica* packed bed treatment on effluent colour

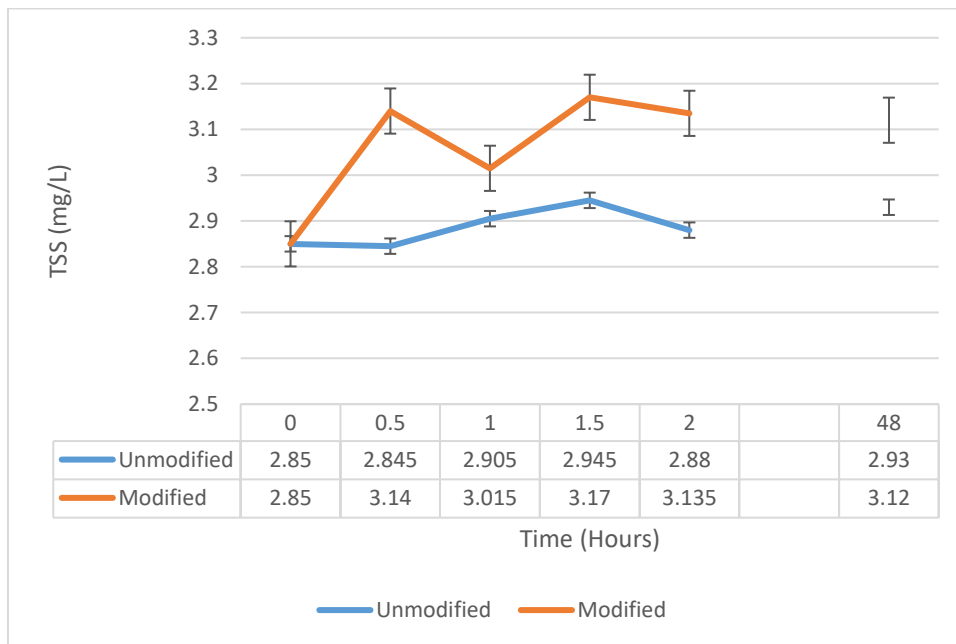


Figure 6. Effect of *luffa cylindrica* packed bed treatment on effluent TSS

improves the wettability of the fibers (Adeniyi *et al.*, 2019b). These improved properties ensure a better interaction of the fibers with the pigments in the effluents. In summary, an optimum 35% colour reduction was achieved after 30 minutes for the modified fibers and 2 h for the unmodified fibers. The minimum value of 3.3 TCU obtained in the study is below the 5 TCU permissible limit by WHO (2003). This suggests that in the domain of colour, the effluent is safe for environmental release without any significant detrimental effect in the ecosystem.

Effect on Total Suspended Solids (TSS)

Suspended solids are particles large enough to not pass through the filter used to separate them from the water (Bowers and Binding, 2006). The effect of the treatment of paint industry effluent by untreated and treated *luffa cylindrica* fibres on TSS is shown in **Figure 6**. In this section, a negative result was achieved for both fibers for the 2 h experiment and the 48 h trial experiment. It is observed that the fibers are unsuitable for the removal of suspended solids from the paint industry effluent. This is unsurprising as the fine particles of pigment and other suspended solids cannot be separated by the larger packing material. These materials are generally removed by physical techniques such as ultrafiltration membranes (Teodosiu *et al.*, 1999) and flocculation (Irfan *et al.*, 2017). Though the porous structure of the packing material in this study generates tortuous convolutions, these are still too large to handle suspended solids.

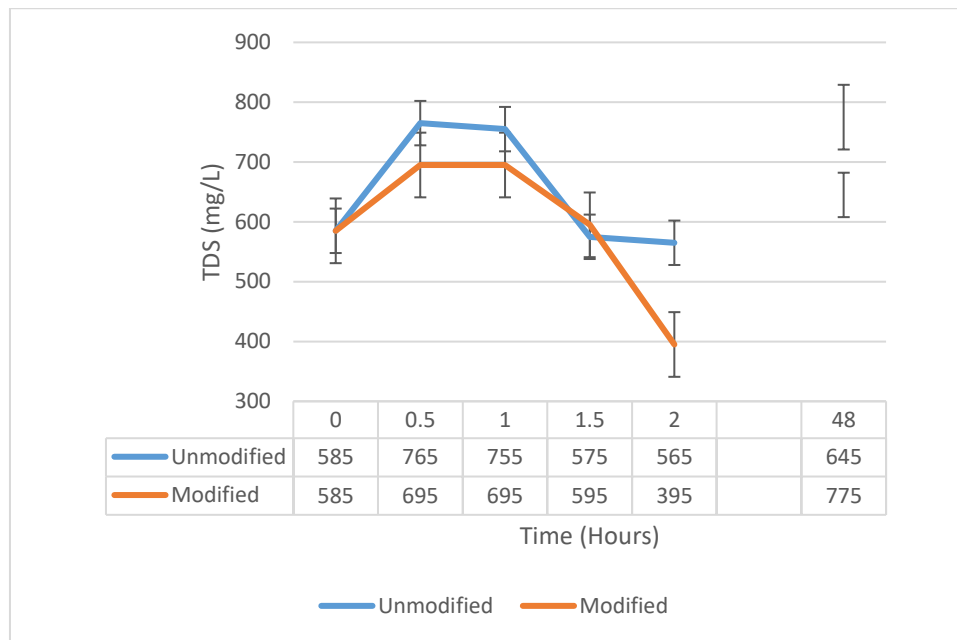


Figure 7. Effect of *luffa cylindrica* packed bed treatment on effluent TDS

Effect on Total Dissolved Solids (TDS)

Dissolved solids are the combined content of all dissolved inorganic and organic substances present in a liquid in the molecular or ionized form (Saravanakumar and Kumar, 2011). The effect of the treatment of paint industry effluent by untreated and treated *luffa cylindrica* fibres on TDS is shown in **Figure 7**. There was an initial rise in TDS in the first 30 minutes of residence of the effluent in the packed bed before reduction commenced. This could be due to secondary leaching from the fibers due to new interactions with the various ionic species in solution. With time, mass transfer dynamics controlled the process to impose a concentration driving force of dissolved solids from the solution to the interstices in the fibres. It was observed that at the end of the 2 h experiment, the untreated fibers reduced the TDS from 585 to 565 mg/l which is relatively poor. However, the treated fibers reduced the TDS to 395 mg/l. It was also observed the 48 h trial experiment was not favourable for the process. The superiority of the treated fiber can be ascribed to the positive effect of mercerisation on the morphology and functional properties. In summary, an optimum 5% TDS reduction was achieved after 2 h for the unmodified fibers and 32.5% TDS reduction after 2 h for the modified fibers. The minimum TDS value of 395 mg/l obtained in the study is below the 500 mg/l permissible limit by WHO (2003). This suggests that in the domain of TDS, the effluent is safe for environmental release without any significant detrimental effect in the ecosystem.

Effect on Dissolved Oxygen (DO)

DO (measured in mg/l) is the amount of oxygen dissolved in the effluent (Bahadori and Smith, 2016). The effect of the treatment of paint industry effluent by untreated and treated *luffa cylindrica* fibres on DO is shown in **Figure 8**. The quality of the water is better when dissolved oxygen is higher (WHO, 2003). In this section, negative results were obtained. The current process does not give an advantage in terms of the dissolved oxygen both for the 2 h experiment and the 48 h trial experiment. This could be due to the fibre's inability to sufficiently handle to ionic species in the effluent which still leads to a high chemically mediated demand for DO. DO is expected to be above 7.5 mg/l and the results of the study show that this was not achieved. DO is considered as one of the most important water quality parameters (Gorde and Jadhav, 2013) as it determines the extent to which the body can support organics. Furthermore, it is an indirect index of oxygen demand from the water sample.

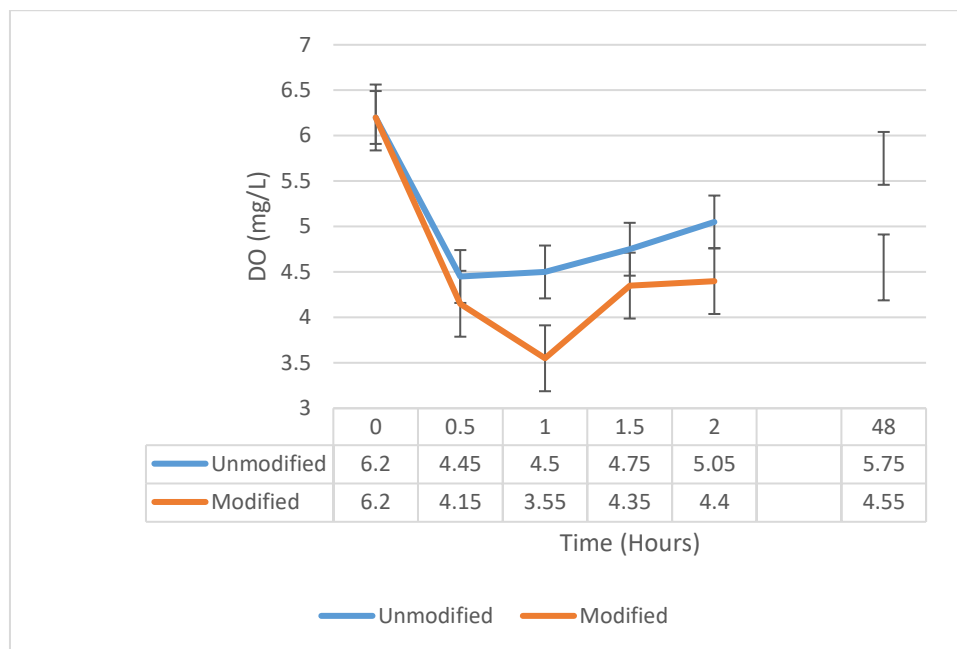


Figure 8. Effect of *luffa cylindrica* packed bed treatment on effluent DO

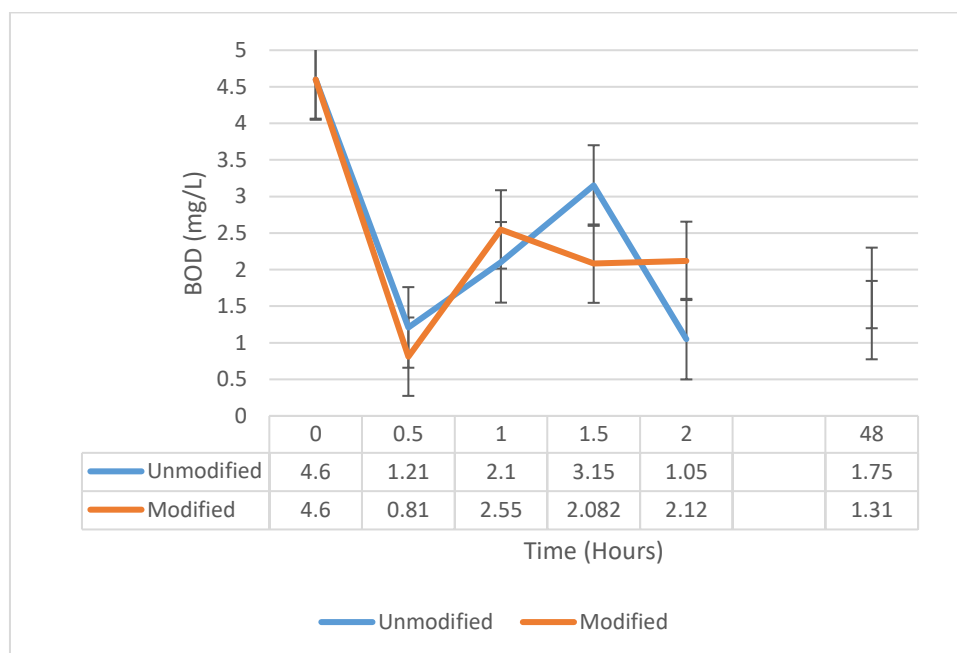


Figure 9. Effect of *luffa cylindrica* packed bed treatment on effluent BOD

Effect on Biochemical Oxygen Demand (BOD)

BOD (measured in mg/l) is the oxygen requirement of all the organic content in the effluent during the stabilisation of organic matter usually 5-day (sometimes 3-day) period (Kamaruddin *et al.*, 2019). The effect of the treatment of paint industry effluent by untreated and treated *luffa cylindrica* fibres on BOD is shown in **Figure 9**. It was observed that optimum performance for the reduction of BOD was achieved in 30 minutes where the BOD dropped for 4.6 mg/l to 0.81 mg/l for the treated fibers and 1.21 mg/l for the untreated fibers. This also indicates that NaOH treatment helped to improve the performance of the fiber. Furthermore, the 48 h trial experiments also gave positive results albeit poorer than those obtained in 30 minutes. Due to the disadvantage of time (which is of the essence in an industrial process), the optimal results can be said to be achieved in 30 minutes. This is a confirmation that the poor levels of DO obtained in the study is not due to biochemically mediated demand for DO. It can be surmised that the NaOH treated fibers was more suitable in reducing the BOD from the effluent than the untreated fibers. An optimum 77% BOD reduction was achieved after 2 h for the unmodified fibers and 82% BOD reduction after 30 minutes for the modified fibers. The minimum BOD value of 0.81 mg/l obtained in the study is considered good for a water sample. There are no specific permissible limit by WHO (2003) for BOD.

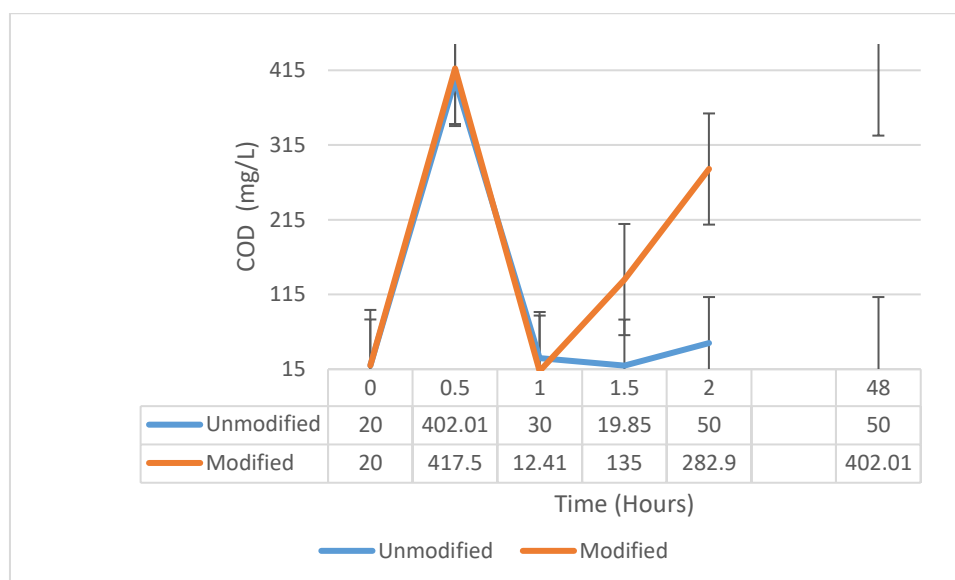


Figure 10. Effect of *luffa cylindrica* packed bed treatment on effluent COD

Effect on Chemical Oxygen Demand (COD)

COD (measured in mg/l) is the equivalent amount of oxygen consumed in the chemical oxidation of all organic and oxidisable inorganic matter present in the effluent (Shi *et al.*, 2020). The effect of the treatment of paint industry effluent by untreated and treated *luffa cylindrica* fibres on COD is shown in **Figure 10**. In this section, negative results were also obtained. The current process does not give an advantage in terms of the chemical oxygen demand both for the 2 h experiment and the 48 h trial experiment. This is due to the fibre's inability to sufficiently handle to ionic species in the effluent which still leads to a high COD. This is a confirmation that the poor levels of DO obtained in the study is due to chemically mediated demand for DO. There are no specific permissible limit by WHO (2003) for COD.

CONCLUSION

In this study, a packed bed utilising modified and unmodified *luffa cylindrica* fibre as packing material were used for the treatment of paint industry effluent. SEM revealed the absence of gummy/waxy substance and lignin in the treated fibers as compared to the untreated fibre. The EDS revealed that the surface of the fibres contained about 60 wt% Carbon and established that there is not much compositional difference between the treated and untreated fibers. Negative findings were achieved for TSS, DO and COD whilst positive findings were achieved for pH, colour, TDS and BOD. Both fibers did not present a pH disadvantage when used as packing material for the treatment of paint industry effluent in a biomass packed bed. NaOH treated fibers was more suitable in removing colour from the effluent than the untreated fibers. The treated fibers reduced the colour of the effluent within 30 minutes of the experiment from 5.04 to 3.30 TCU. The treated fibers reduced the TDS from 585 to 395 mg/l. It was observed that optimum performance for the reduction of BOD was achieved in 30 minutes where the BOD dropped for 4.6 mg/l to 0.81 mg/l for the treated fibers and 1.21 mg/l for the untreated fibers. The extended residence time of 48 h was only of advantage for colour and BOD removal. In the domain of the positive results, the untreated fibers achieved 35% colour reduction, 5% TDS reduction and 77% BOD reduction all in 2 h. The treated fibers achieved 35% colour reduction in 30 minutes, 32.5% TDS reduction in 2 h and 82% BOD reduction in 30 minutes. The fibers treated with NaOH performed better in all indices where positive results were achieved except for pH. It can be concluded that *Luffa cylindrica* fibres can be used as effective packing material in a biomass filter for the treatment of paint industry effluent before releasing into the environment based on WHO limits. This mean that the release would not lead to an observable environmental degradation and death of the flora and fauna in the ecosystem.

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