Experimental Scrutinization on Treatment of Organic and Inorganic Effluents using Adsorption Process

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Abstract: Wastewater treatment has become one of the most significant sources for irrigation and other activities in arid and semi-arid countries. Many conventional methods deal with different organic and inorganic compounds of wastewater. One of the most common is using activated carbon through an adsorption process to treat wastewater. Adsorption is used when the contaminants present in wastewater are removed using activated carbon as an adsorbent. This paper examines the results of experiments on removing organic and inorganic compounds using activated carbon prepared from waste tyres, banana trunks, tea leaves and date seeds. Moreover, studies state that activated carbon has a strong affinity for binding organic substances, even at low concentrations. Thus, it has become the premier method for treating organic-laden wastewater. This paper reveals a method to deal with contaminants in wastewater which is very effective. Various experimental tests were carried out to determine the adsorption capacity of the different activated carbon, expressed as percentage removal of Chemical Oxygen Demand, Total Suspended Solid, and Total Dissolved Solid, and the effects of pH, contact time and dosage. The surface area of the adsorbent, pore volume and an isothermal graph of the adsorbent were determined using BET (Brunauer-Emmett-Teller) Surface Area. Waste tyres (WT), date seeds (DS), tea leaves (TL) and banana trunk (BT) have 1260, 1144.52, 163.8 and 115.4 m2/g surface area with 1.62, 0.656, 0.066088 and 0.4566 cm^3 of total pore volume respectively indicating the use of waste tyres as an adsorbent. The moisture and ash content percentages were also determined and it was found that the above adsorbents have 2.6%, 6.4%, 7.4% and 9% moisture content, with 3.6%, 6.2%, 6.9% and 12% ash content respectively.

Keywords: Activated carbon, Adsorption, Banana trunk, Date seeds, Tea leaves, Tyres, Wastewater.

1. INTRODUCTION

Water is the most common and huge spread among all living things on earth. Moreover, water is used for many purposes, such as domestic consumption, industrial production, irrigation, transportation and energy production, and cleaning [1]. However, although the Earth's surface is covered by more than 70% of water, only 0.5% of this is appropriate for human use. Furthermore, this small percentage decreases as agriculture, industry and domestic requirements consume more than this fraction. At the same time, waste generates pollutants that increase water quality degradation, rendering it unfit. That is because wastewater has a considerable amount of organic compounds. If discharged in large quantities to receiving water bodies, it will cause exhaustion of the dissolved oxygen and cause environmental problems. This will make the environment uninhabitable for higher life forms such as fish. Surveys released by Public Health Departments in various countries have noted that significant numbers of people have been exposed to the hazards of heavy metals and other pollutants. So, to protect the environment and sustain life, wastewater has to be treated.

Even though several different techniques exist, such as chemical precipitation, membrane filtration, alum coagulation and iron coagulation, these techniques possess inevitable, unavoidable and often detrimental side effects (Yadav *et al*. 2021) [3]. There are different methods of wastewater treatment, the most common being an activated carbon-based treatment system, as it is quite effective in removing trace quantities of pollutants [4]. Unfortunately, using activated carbon for wastewater treatment is not feasible for developing countries as it entails high costs [3]. In light of the above situation, several studies evaluated the preparation of activated carbon from low-cost materials. These include olive cakes, date stones, cotton stalks, almond shells, corn cobs, waste tea, sawdust, cherrystones, rice bran, coffee husks, spent grains and giant reeds. As adsorption can remove soluble and particulate carbon, nitrogen, and phosphorus from domestic sewage by using chemical treatment methods, it is vital to study the effectiveness of using activated carbon prepared from low-cost precursor materials. Authors investigated various water treatment techniques for the waste water generated from decanter during ethyl acetate reactive distillation process [17]. Saravanan *et al* investigated the

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simulator model based on renewable energy groundwater purification systems to treat subterranean water in Oman [18]. Authors investigated the various types of energy sources that can be used for water treatment in oil and gas industries [19]. Authors investigated the role of an entrainer to break the water-ethanol azeotrope and extract water from the reactive part more efficiently in reactive distillation process [20].

2 MATERIALS AND METHODS

2.1. Methodology

1 kg of tea dust is boiled with distilled water at 100 ℃ for 15 minutes till the pH is neutral. After that, it is dried in a dryer at 120 ℃ for 6 hours. For preparing activated carbon from the banana trunk, 1 kg of banana skin is washed with distilled water to remove its impurities. It is then solar dried for 24 hr and then further dried at 50 ℃ to remove any drop of water. It is then soaked in ortho-phosphoric acid for a 24-hour (1:1) ratio [5]. Then the sample is heated in the furnace at 450 ℃ for one hour. After the thermal chemical activation, it is cooled at room temperature and then dehydrated with silica gel to absorb the Moisture, affecting the efficiency of the prepared activated carbon. It is then ground to mesh size 75μm and stored in an airtight container for analyzing the effectiveness. The same procedure is repeated for other materials but at a different activation temperature. The waste tyres must be dried at 350 ℃ for 3 hours and burned at 550 ℃ for 3 hours, whereas the date seeds must be dried at 100 ℃ for 24 hours and at 450 ℃ for 30 minutes [6].

On the other hand, the tea leaves must be first boiled with distilled water at 60 ℃ for 15 minutes. After checking for pH, this is followed by subjecting it to drying at 100 ℃ for 6 hours [7]. The sample will then be burned at 600 ℃ for 30 minutes.

2.2. Experimental Analysis

After preparing activated carbon, the following analysis was done to check its effectiveness.

2.2.1. Surface Area and Total Pore Volume Analysis

The surface area and total pore volume of the adsorbents by measuring the BET Surface Area method. The equipment consists of two primary components: a circular vibrating bowl that is amplitude variable and controls the initial sample powder delivery rate. The second component is a sample collector, a rotating disc holding eight test tubes. The instrument is connected to a monitor for the display of results.

The analysis device was an Agilent Cary 630 FTIR spectrum, where the spectra were obtained from vibration or frequency changes in the bond that are naturally present in the sample [8]. The moisture content was found by weighing 5 g of adsorbent in a petri dish and then drying it at 105 ℃ for 20 minutes. After 2 hours, weigh the petri dish again and find the moisture content% by weight difference reported in percentage. The ash content of activated carbon can be determined by taking 5g adsorbent in the crucible and then burning it in the furnace at 750 ℃ for 1 hour and measuring the weight difference [9].

2.2.2. Physiochemical Properties

This project conducted all the experiments at room temperature and atmospheric pressure in batch mode. The batch mode was selected because it is simpler compared to continuous study. The investigation is run in a different flask of 250 ml capacity filled with 50ml of wastewater. The required pH in the 2 to 10 was adjusted using 0.1N NaOH and 0.1N HCl solution. 0.2 g of adsorbent was added to each bottle. The sample was then kept in a rotary orbital shaker at 100 pm. The bottles were withdrawn from the shaker after 1 hour [7]. The samples were then filtered to analyze their effectiveness.

The amount of adsorbent, the volume of wastewater and speed of shaking were maintained, and the time intervals varied from 30, 60, 90, 120 and 150 minutes. The samples were then filtered to analyze their effectiveness. To understand the effect of stirring time, the pH of the sample was adjusted to the optimum pH obtained in the previous experiment [10].

To study the effect of dosage, the pH of the sample and the contact time was adjusted at the optimum value obtained from previous experiments. The volume of wastewater and stirring speed had to be the same as above while varying the dosage concentration from 0.2 to 1 g [6].

2.2.3. Chemical Oxygen Demand (COD)

COD measurement was done by pipetting 2ml of sample into a prepared vial, mixing it, and transferring it to the digester block at 150 ℃ for 120 minutes. The vials are cooled at room temperature and placed in the photometer to determine the COD readings (mg/l).

2.2.4. Total Suspended Solid (TSS)

It was determined by weighing the dry filter paper and then filtering 50ml of the sample using filter paper. It is then weighed again to find the TSS in mg/l [11]. The filter paper is dried 50 ℃ for 24 hours and then cooled at room temperature.

2.2.5. Total Dissolved Solid (TDS)

The measurement of TDS in this project was carried out using a Water analysis kit device. The TDS value can be determined by simply dipping the electrode of the sample after the filtration process.

3. RESULTS AND DISCUSSIONS

3.1. Moisture Content

Table 3.1: Moisture Content

Adsorbents	Initial Weight (g)	Final Weight 2(g)	Moisture Content %
W.T	5	4.87	2.6
D.S	5	4.68	6.4
T.L	5	4.63	7.4
B.T	5	4.55	9

The moisture content is the quantity of water physically bound to A.C under normal conditions. According to Table **3.1**, the W.T, D.S, T.L and B.T, respectively, have less than 10% moisture content %, and the B.T and T.L have heights of moisture content of 9% and 7.4%, respectively. The D.S and W.T have the lowest moisture content of 6.4% and 2.6%, respectively. Studies have shown that the A.C, which has a lower moisture content %, has more contaminants' adsorption efficiency.

Thus, the lower moisture contents in W.T indicate that the carbon structure has extensive porosity through the activation process.

Since the adsorption efficiency increased as the moisture content% decreased, the W.T give a high adsorption efficiency because it has a low moisture content%. This means it has fewer pores that contain water (more surface area for adsorption) which may increase the capability of W.T to adsorb more contaminants particles.

3.2. Ash Content%

According to Table **3.2**, W.T, D.S, T.L and B.T respectively, have less than 15% ash content%. The B.T and T.L have the highest ash content% of 12 % and 6.9 %, respectively. The W.T and D.S have the lowest ash content% of 3.6% and 6.2%, respectively.

Table 3.2: Ash Content%

Adsorbent	Initial weight (g)	Final weight (g)	Ash Content %
W.T	5	4.82	3.6
D.S	5	4.69	6.2
T.L	5	4.65	6.9
B.T	5	4.4	12

The low ash content% indicates that the A.C have low inorganic content and high fixed carbon. This is because ash content can reduce the efficiency of reactivation. Therefore, the lower the ash content, the better A.C. Hence, the W.T has a high adsorptive capacity [12].

3.3. Surface Area and Total Pore Volume

The adsorptive capacity of A.C is associated with its internal surface area and pore volume. The size of the molecules that may be adsorbed is limited by pore volume. Usually, the adsorbent with a large surface area is better in its adsorption process.

Table 3.3: Surface Area and Total Pore Volume of Adsorbents

Adsorbents	Initial Weight (g)	Final Weight 2(g)	Moisture Content %
W.T	5	4.87	2.6
D.S	5	4.68	6.4
T.L	5	4.63	7.4
B.T	5	4.55	9

The surface area for four samples was determined using BET Surface Area. The result is given in Table **3.3.** The result shows that the surface area of W.T was high, and the B.T was low. These low surface areas may be due to the large size of nitrogen gas that enters with difficulty into the smaller diameter of the microspore or due to the characteristics of A.C itself.

Anyway, the W.T and D.S have high surfaces compared to the result sure researchers [13], respectively, which means the activation process was high and successful.

3.4. Fourier Transform Infrared Spectra (FTIR) Spectrum Analysis

The FT-IR analysis was carried out to determine the adsorbents' wavelength and strength at each peak.

The frequency change indicates the peaks where the functional group plays a role in adsorption. The spectra were collected between 675.426 and 3755.774 cm-1. The graph obtained from the equipment is a plot between adsorbance and wavelength according to the figure (Figure **3.1-3.4**) represented below. The W.T chart shows a more complex diagram indicating the high adsorption capacity.

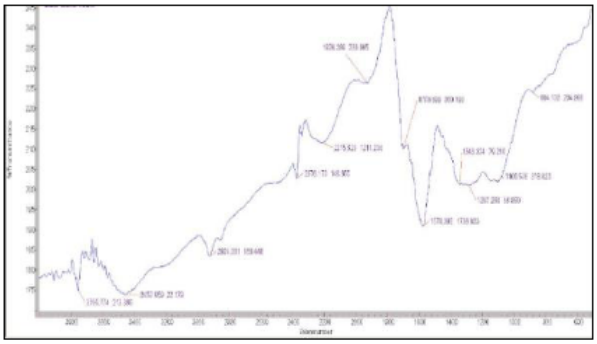

Figure 3.1: FT-IR analysis on W.T.

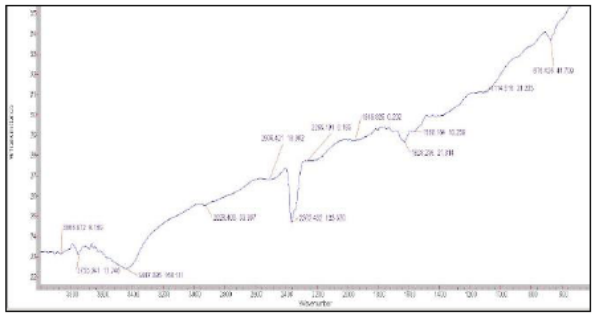

Figure 3.2: FT-IR analysis on D.S.

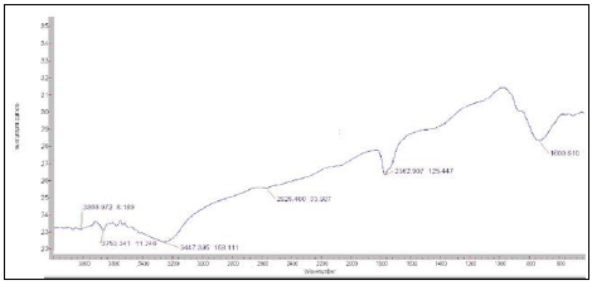

Figure 3.3: analysis of B.T.

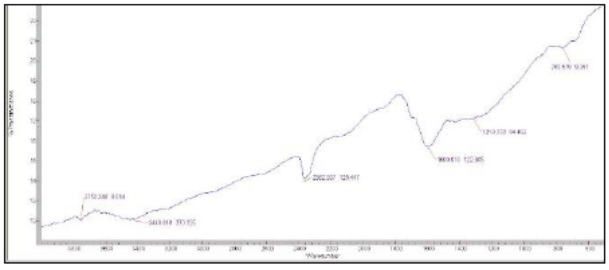

Figure 3.4: analysis of T.L.

3.5. Effect of pH on COD

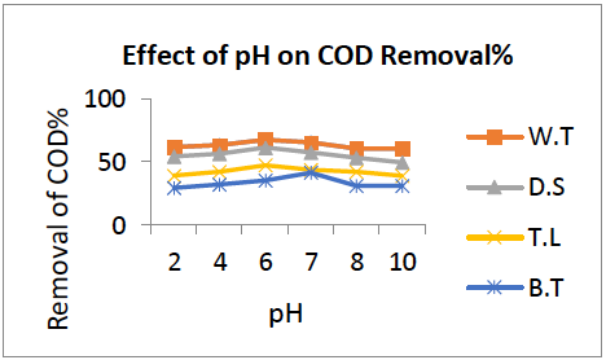

Figure 3.5: Effect of pH on COD removal%.

The ability of A.C to adsorb the contaminants in wastewater was also affected by the acidity condition of the adsorbent surface. The pH of the solution can affect the adsorption process. The effect of pH on COD removal from wastewater is illustrated in Figure **3.5**. It was found that the COD removal% is very sensitive to the pH of the wastewater. It was observed that, as pH increased, the COD removal% increased until it reached a certain point and then started to decrease as it got further. It decreased once it attained the base condition, and this was because the reaction mechanism changed. The formation of OH- radicals will interfere with the oxidation of the organic matter of wastewater. Anyway, the constant zone between pH 8 and pH 10 in W.T and B.T curve is due to the attainment of equilibrium between the adsorbent and adsorbance in which the adsorbate species usually form a surface layer, which is only a molecule thick, on the surface of the adsorbent, which prevents the organic substances from sticking to the adsorbate [11].

3.6. Effect of pH on TSS Removal%

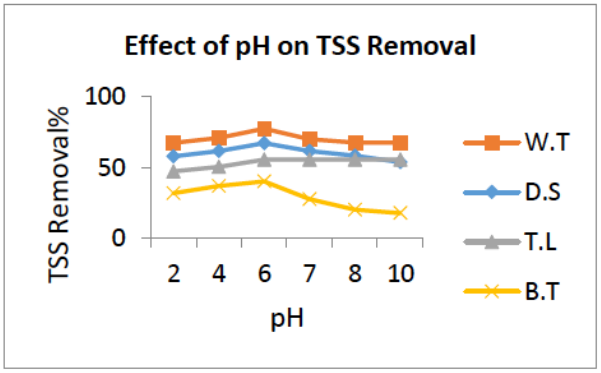

Figure 3.6: Effect of pH on TSS removal %.

From the graphical representation (Figure **3.6**), it was seen that, as the pH increased, the TSS removal%

increased. However, it decreased once it reached the base condition because H+ will enhance the pore size of the adsorbent, causing many contaminants to stick in it, which will tend to decrease the number of suspended solids in the sample. However, once it is in base condition, the OH- will compete with organic substances, and more OH- will stick to the surface of the adsorbents. Therefore, the amount of suspended solid removed will be high at pH 6 (base condition) since the amount of organic that sticks on the surface of the adsorbents is less. The optimum pH was found at pH 6.

3.7. Effect of pH on TDS Removal%

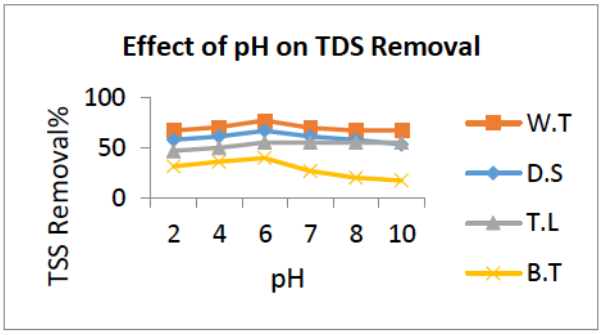

Figure 3.7: Effect of pH on TDS removal%.

From the graphical representation (Figure **3.7**), it was seen that, as the pH increased, the percentage removal of TDS also increased [14].

3.8. Effect of Contact Time on COD Removal%

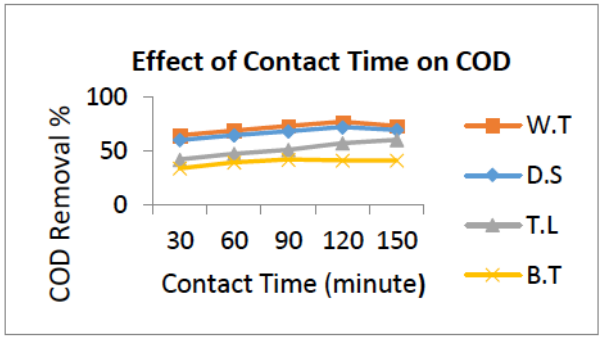

Figure 3.8: Effect of Contact Time on COD.

COD removal% from the graphical representation (Figure **3.8**) shows that as contact time increases, the COD removal increases. This is because the more time available, the more organic substances get to stick on the surface of the adsorbent. Therefore, the amount of chemicals that oxidize will increase as time increases. The optimum time is shown at 120 minutes.

3.9. Effect of Contact Time on TSS Removal

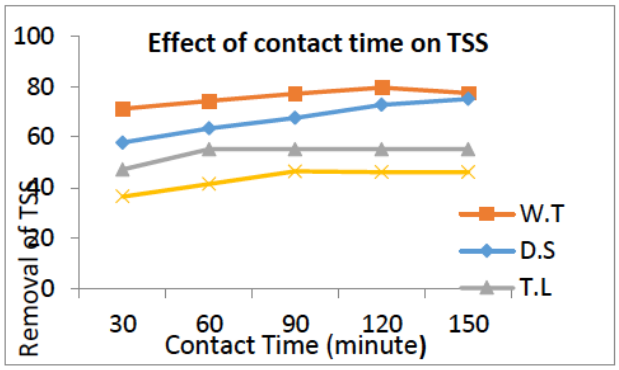

Figure 3.9: Effect of contact time on TSS Removal%.

From the graphical representation (Figure **3.9**), it was seen that, as the time contact increased, the TDS removal% increased. This is because, as time contact increased, more organic substances got stuck on the surface of the adsorbent. Moreover, surface adsorption increased. Therefore, the amount of suspended solid decreased. The optimum time was 120 minutes, as was hypothecated by (Al Khusaibi *et al*., 2015).

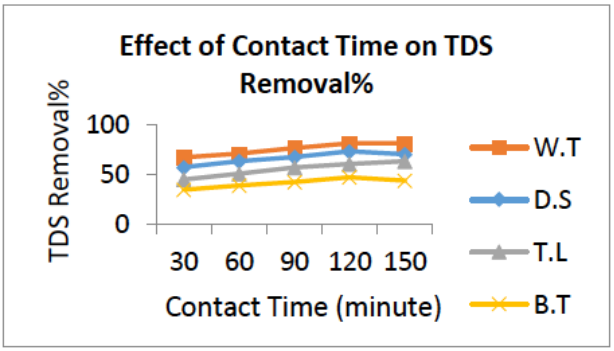

Figure 3.10: Effect of Contact Time on TDS Removal%.

3.10. Effect of Contact Time on TDS Removal%

From the graphical representation (Figure **3.10**), it was seen that, as the time contact increased, the TDS removal% increased. This is because, as time contact increased, more organic substances got stuck on the surface of the adsorbent. Moreover, surface adsorption increased. Therefore, the amount of suspended solid decreased. The optimum time was at 120 minutes.

3.11. Effect of Dosage on COD Removal%

The COD removal effect is sensitive to dosage. The effect of dosage on COD removal from wastewater is illustrated in Figure **3.11**

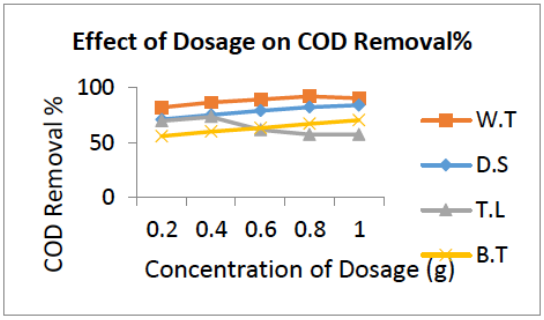

Figure 3.11: Effect of dosage on COD Removal%.

Figure **3.11** describes the effect of dosage in COD removal%. It shows that, as the dosage concentration increased, the COD removal% increased. The increase in COD removal as dosage concentration increased was because the higher the concentration dosage, the greater the surface area would be. Hence, more organic substances get to stick to the surface of the adsorbent. Therefore, the amount of chemicals that oxidize will increase as time increases. The optimum dosage showed at 0.8g.

3.12. Effect of Dosage on TSS Removal%

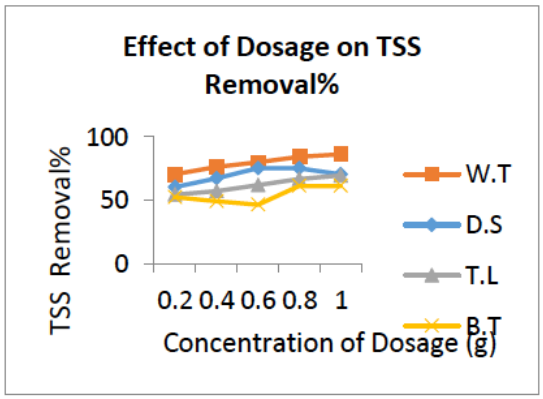

Figure 3.12: Effect of dosage on COD Removal%.

According to Figure **3.12**, the TSS removal% is sensitively affected by the dosage concentration. The W.T curve shows a directly proportional between the TSS removal and dosage. From the graphical representation, the TSS removal% increased with dosage concentration. The reason was that, as the concentration of dosage increased, more surface area was available and, hence, more contaminants stuck to the adsorbent. Therefore, the amount of suspended solid was less.

3.13. Effect of Dosage on TDS Removal%

The TDS removal effect is sensitive to dosage. The dosage effect on TDS removal from wastewater is illustrated in (Figure **3.13**).

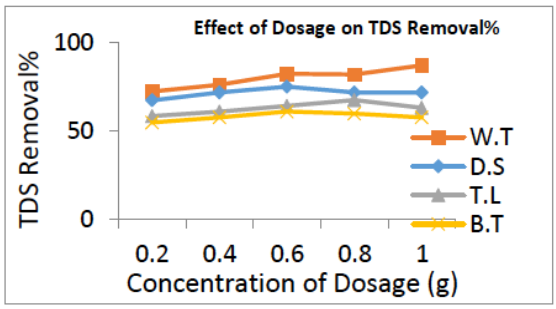

Figure 3.13: Effect of dosage on TDS Removal%.

From the graphical representation, the TDS removal% increased with dosage concentration. The reason was that, as the dosage concentration increased, more surface area was available and more contaminants stuck to the adsorbent. Therefore, the amount of dissolved solid was less. The authors group studied the water treatment methodologies using advanced oxidation techniques [15, 16].

4. CONCLUSIONS

Wastewater has a significant level of toxic, odorous and undesired organic contaminants. Hence, an adsorption process must be applied using A.C as an adsorbent. (1) The A.C was successfully prepared from different waste materials in this study, mainly W.T, D.S, T.L and BS. Further, to know the characteristics of adsorbents, the physical properties of adsorbents were determined by using BET Surface Area, Moisture Content, Ash Content and FTIR. (2) The influence of physicochemical properties, namely Chemical Oxygen Demand (COD), Total Suspended Solid (TSS) and the Total Dissolved Solids (TDS), were verified in the adsorption process. (3) Additionally, the performances of the adsorbents were compared together. (4) A.C prepared from W.T can be used successfully in wastewater treatment. The highest adsorbed contaminants were done by W.T % of COD removal, % of TSS removal and % of TDS removal. W.T and D.S proved to be the best in wastewater treatment. However, BS and T.L show low adsorption capacity, which may be based on their physical characteristics. (5) The adsorption capacity was found to vary with pH, contact time and adsorbent dose. (6) The increase in pH led to a rise in the adsorption of contaminants. However, it decreased once it reached the base condition. (7) Similarly, an increase in contact time and adsorbent dose will significantly lead to a rise in a specific area and, hence, an increase in adsorption capacities. (8) The isothermal graph was satisfactory in predicting adsorption capacities. Therefore, it can expect adsorption capacities under different pH, contact time, and adsorbent dose conditions. (9) The

design of the mixing tank was also attempted in this work.

LIST OF ABBREVIATION (S)

BET	Brunauer-Emmett-Teller
WT	Waste tyres
DS	Date seeds
TL	Tea leaves
BT	Banana trunk
COD	Chemical Oxygen Demand
TSS	Total Suspended Solid
FTIR	Fourier Transform Infrared Spectra
TDS	Total Dissolved Solid
TSS	Total Suspended solids
AC	Activated carbon

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