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Performance of *Prosopis africana* peel powder (PAPP) as a novel sorbent for remediating malachite green contaminated aqua system

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This study was designed to investigate *Prosopis africana* peel powder (PAPP) as a potential, local, cost-effective, readily-available coagulant for remediating dye-contaminated water and compare it with activated charcoal (a standard coagulant). PAPP was prepared and characterized using standard procedures. Preliminary characterization of the malachite green (MG) contaminated water was carried out before and post-treatment with PAPP and activated charcoal, respectively, in terms of turbidity, pH, salinity, temperature, electrical conductivity, total solid (TS), total suspended solid (TSS) and total dissolved solid (TDS). The concentration of the MG adsorbed by both coagulants were determined and compared. Results showed that the percentage of MG removed increased proportionally with the concentration of the coagulants. With PAPP, the pH of the solution was 4.67, the salinity and TSS were zero while TS and turbidity were only pronounced at higher concentrations; whereas with activated charcoal, the pH was 6.37, the turbidity, TSS, TS and salinity were zero but TDS and conductivity were concentration dependent. This study has shown that PAPP could be employed as a potential adsorbent in the treatment of MG contaminated water. A study is currently in progress to activate PAPP and compare its dye removal efficiency with activated charcoal.

Key words: Malachite green, coagulant property, contaminated water, activated charcoal, *Prosopis africana* peel powder (PAPP).

INTRODUCTION

The discharge of industrial effluents such as dye contaminated wastewater and other environmental pollutants into water bodies (natural streams and rivers) poses serious public health problems, especially as most of the dyes are not biodegradable (Vahl et al., 2011; Aksu, 2005; Arami et al., 2005; Artioli, 2008; Srivastava

et al., 2004). Consequent upon this, several strategies have been embarked upon to decolorize effluents (Gong et al., 2009; Cheng et al., 2008; Mittal et al., 2005) but despite all sophistications, adsorption remains the simplest and most cost-effective (Malik et al., 2007; Zhang et al., 2008). Yet the high cost of adsorbents makes the process uninteresting in addition to recent snags associated with most conventional coagulants. This has caused the paradigm shift to bio-coagulants and bio-flocculants as safer, cheaper and greener alternatives in the remediation of dye contaminated water (Kumar et al., 2005; Kumar, 2007; Hamdaoui et al., 2008).

Prosopis refers to pod-bearing trees or shrubs that occur in arid and semi-arid zones of America, Africa and Asia, with 44 species already reported (World Agroforestry Centre, 2008). The adsorptive properties of

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Abbreviations: PAPP, *Prosopis africana* peel powder; TSS, total suspended solids; TDS, total dissolved solids; TS, total solids; MG, malachite green; PZC, point of zero charge; SVI, Sludge volume index; DREM, direct reading engineering method.

Prosopis cineraria has been investigated (Garg et al., 2004). *Prosopis africana* (Fam. Mimosaceae), a perennial leguminous woody tree of about 70 ft high mostly found growing in the savanna regions of West Africa (Kolapo et al., 2009), has very hard stem that is used in different parts of Nigeria for making boats, pestle and wooden gong; its fermented seeds are used as food condiment/flavouring agent (Aremu et al., 2006); its young leaves and shoots are folders highly sought after towards the end of the dry season (Agboola, 2004) and the gum from the seed mesocarp possesses drug delivery properties (Attama and Adikwu, 2000). Various parts of the tree have been used for the treatment of various ailments such as bronchitis, dermatitis, gonorrhoea, dysentery, malaria, rheumatism, sore throat, fevers, skin diseases, headache, toothache, stomach cramps, skin diseases, and as a dressing for wounds or cuts (Kolapo et al., 2009; Adikwu et al., 2003). To date, there is no literature report on the sorbent property of *P. africana* peel powder (PAPP). In Nigeria, *P. africana* peel is a waste material found among the rural dwellers that process the seed as condiment for cooking (Aremu et al., 2006). After removal of the seed, a little fodder is normally reserved for domestic animals while the remaining portion is often discarded into farmlands (Agboola, 2004). We therefore reasoned that this material could serve as a low-cost material to remediate dye-contaminated water which is a common occurrence in most coastal regions of Nigeria. Meanwhile, progress in the development of modern materials, including so-called nanomaterial, is enhanced by advances in powder technologies and improvement in powder characterization methods, since the efficiency of ultrafine grained powders are controlled by their chemistry, size and shape. Thus, in this study, the sorbent property of PAPP for malachite green (MG) contaminated water was investigated and compared with activated charcoal, a standard coagulant.

MATERIALS AND METHODS

The following materials were used without further purification: HCl, NaOH, ether, nitric acid (BDH, England), malachite green (MG) [CI = 42,000B, chemical formula = $C_{23}H_{26}ON_2$, nature = basic green), *P. africana* peel powder (PAPP), activated charcoal.

Preparation and characterization of *Prosopis* peel powder

The *prosopis* peel was obtained after boiling and separating the edible fermentable seed from the coat which is of two parts, the fleshy white mesocarp (the gum containing portion) and the hard pericarp (the outermost part), which is always thrown away as waste product among the Nigeria local people who process this plant seed as food condiment. It was washed with deionized water, air-dried and ground into powder using laboratory grinding machine. The powder was sieved with a laboratory sieve of known mesh size to obtain micro powders.

The pH of a solution of the *prosopis* powder was determined using a laboratory pH meter (Hanna, pH 21 microprocessor). The

micromeritics of the powder was also determined.

The point of zero charge (PZC) was determined using the solid addition method (Oladoja and Aliu, 2009) namely: 45 ml of 0.1 M KNO_3 solution was transferred to a series of 100 ml conical flask. The pH_0 values of the solution were adjusted from 2 to 12 by adding either 0.1 N HNO_3 or NaOH. The total volume of the solution in each flask was made up to 50 ml by adding the KNO_3 solution. The pH_0 of the solutions were accurately noted. Then, *prosopis* powder (1 g) was added to each flask and securely capped, immediately. The suspension was manually agitated for 0.5 min after which the pH values of the resultant supernatant liquids were noted. The difference between the initial and final pH values ($\Delta pH = pH_0 - pH_f$) was plotted against the pH_0 . The point of intersection of the resulting curve at which pH_0 gave the PZC was noted.

The stability of the *prosopis* powder was assessed by dispersing 2 g in different aqueous solutions [acidic (0.1 N HCl), neutral (distilled water) and basic solutions (0.1 M NaOH)]. The mixture was agitated intermittently, for a period of 120 min. Thereafter, the mixture was filtered and the residue was dried to a constant weight. The magnitude of the difference in weight of the powder before and after soaking in different aqua media was used as a measure of its stability in the different solutions.

Preparation of malachite green

Malachite green is a basic dye with chemical formula ($C_{23}H_{26}ON_2$). An accurately weighed quantity of the dye was dissolved in double distilled water to prepare the stock solution (500 mg/L). Different working solutions were prepared by successive dilution with double distilled water. The concentrations of the residue dye solutions were determined by determining the absorbance characteristics wavelength using UV/visible spectrophotometer. A standard solution of the dye was taken and the absorbance was determined at different wavelengths to obtain a plot of absorbance versus wavelength. The wavelength corresponding to maximum absorbance (λ_{max}) was determined from this plot. The λ_{max} of malachite green was found to be 620 nm. Calibration curve was plotted between absorbance and concentration of the dye solution.

Coagulation experiment

The conventional jar test procedure with a six-unit multiple stirrer system was used. *Ab initio*, the absorbance readings of the various concentrations of the coagulants were taken before coagulation. Activated charcoal (100 to 1000 mg/L), as the standard coagulant was added to the samples of the synthetic dye-contaminated water at different concentrations (5 to 120 mg/L) and rapid mixing was performed at 200 rpm for 2 min. A flocculation time of 20 min and then 30 min of settling time were allowed. PAPP was also employed as the test coagulant at the same concentration as the standard.

Characterization of the treated water

Determination of total solids

After flocculation, 50 ml volume of each water sample was collected from the same depth, added to pre-tared porcelain dish of known weight (W_1) and evaporated to determine the total residue on dryness. This was done by placing the dish with its content on a steam bath and subsequently in an oven (Galenkemp No. 335, England) at 100°C. The samples were intermittently cooled in desiccators and weighed until constant weights (W_2) were obtained. The difference in weights ($W_2 - W_1$) was calculated and the total solids determined using the equation:

Table 1. Chemical composition of *prosopis* peel powder.

| Physicochemical characteristics | Values |
|-------------------------------------|--------|
| pH solution | 7.01 |
| Bulk density (g/cm ³) | 0.633 |
| Tapped density (g/cm ³) | 0.840 |
| Carr's compressibility index (%) | 20.7 |
| Angle of repose (θ°) | 30 36 |
| Flow rate (g/cm) | |
| <5 | 1.47 |
| 5-10 | 1.73 |
| >10 | 2.25 |
| Particle size (μm) % | |
| >90 | 40.32 |
| 90-63 | 47.12 |
| <63 | 12.56 |
| Ash (%) | 35.6 |
| Moisture (%) | 12.5 |
| Protein (%) | 38.65 |
| Carbohydrate (%) | 0.537 |
| Fat (%) | 3.45 |
| Organic matter (%) [100-ash] | 64.4 |

$$\text{Total solids (\%)} = \frac{\text{constant weight of residue}}{\text{volume of water sample}} \times 100 \quad (1)$$

$$= \frac{W_2 - W_1}{50} \times 100 \quad (2)$$

Where W_2 is the weight of residue + dish, and volume of water sample evaporated is 10 ml while W_1 is the weight of empty dish.

Turbidity test

Turbidity was measured as a function of total suspended solids (TSS) using the gravimetric method. A pre-tared glass fibre was dried in the oven (Galenkamp No. 335, England) at 100°C and weighed (W_1). A 50-ml volume of each water sample taken at the same depth was filtered through the fibre bed until two consecutive constant weights were obtained. The TSS was calculated using the equation:

$$\text{TSS (\%)} = \frac{W_2 - W_1}{50} \times 100 \quad (3)$$

Where W_2 is the constant weight of the solids on the filter bed, W_1 is the weight of the empty tarred glass fibre.

Total dissolved solids (TDS)

TDS was determined by the direct reading engineering method (DREM), using multimeter TDS monitor (Hack Model C 150, USA). Briefly, 50 ml of each sample was collected from the same depth

and added into a 50 ml glass beaker. The probe of the meter already calibrated prior to use was dipped into the sample and the measurement of TDS was read off the liquid crystal display (LCD) on the screen of the equipment. The test was done in triplicates for validity of statistical data.

Conductivity and salinity

Conductivity of the water samples was done by the DREM method using the same multimeter at different modes (salinity, conductivity, TDS modes). The pre-calibrated probe was dipped into 50 ml of each water sample in a 50 ml glass beaker. Pressing the conductivity mode button displays the measured conductivity on the LCD panel of the meter.

Salinity was also determined using the same procedure. This involved dipping the pre-calibrated probe into each water sample and switching on the salinity mode button of the meter. The measurements were read off the LCD panel of the meter in triplicates for statistical significance.

pH

The pH of the sample was determined by the same DREM, using a pH meter (Suntex, Soughai). The electrode of the pre-calibrated pH meter was dipped into the sample and the value displayed was recorded.

Characterization of the sludge

In order to observe the settling characteristics of the sludges produced from the use of activated charcoal and PAPP, the sludge volume index (SVI) measurement was carried out over time, on the sludges produced.

Statistical analysis

All experiments were performed in replicates for validity of statistical analysis. Results were expressed as mean ± SD. ANOVA and student's t-test was performed on the data sets generated using Predictive Analytics Software. Differences were considered significant for P values < 0.05.

RESULTS

The results of the physicochemical characterization of the *prosopis* powder are presented in Table 1. The pH of the *prosopis* powder solution was 7.01 while the bulk density was 0.633 g/cm³. The results of the chemical composition revealed an average inorganic fraction of the *prosopis* peel (ash content = 35.6%) and the predominance of protein (38.65%) amongst carbohydrate (0.5337%) and fat content (3.45%). The results of the zero point of charge of the *prosopis* peel powder (Ph_{PZC} 5.6) is presented in Figure 1.

This shows that at a pH less than 4.8 the surface of the PAPP is predominated by negative charges. The flow rate (g/s) of the *P. africana* peel powder varied directly with the height of suspension of the funnel such that at 5, 10 and 15 cm the flow rates were 1.47, 1.73 and 2.25 g/s respectively. The angle of repose was between 30 to

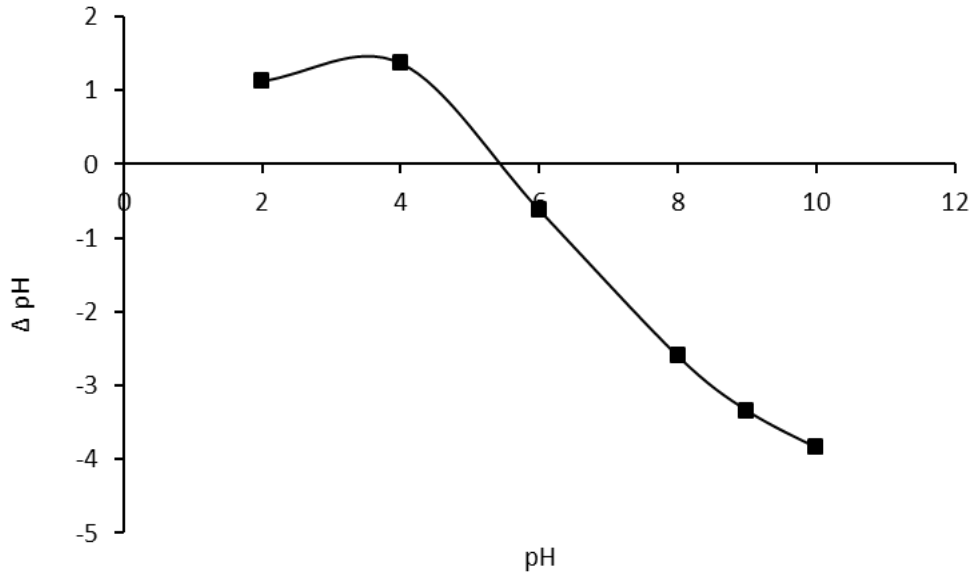


Figure 1. Determination of the point of zero charge (PZC) of *Prosopis africana* peel powder.

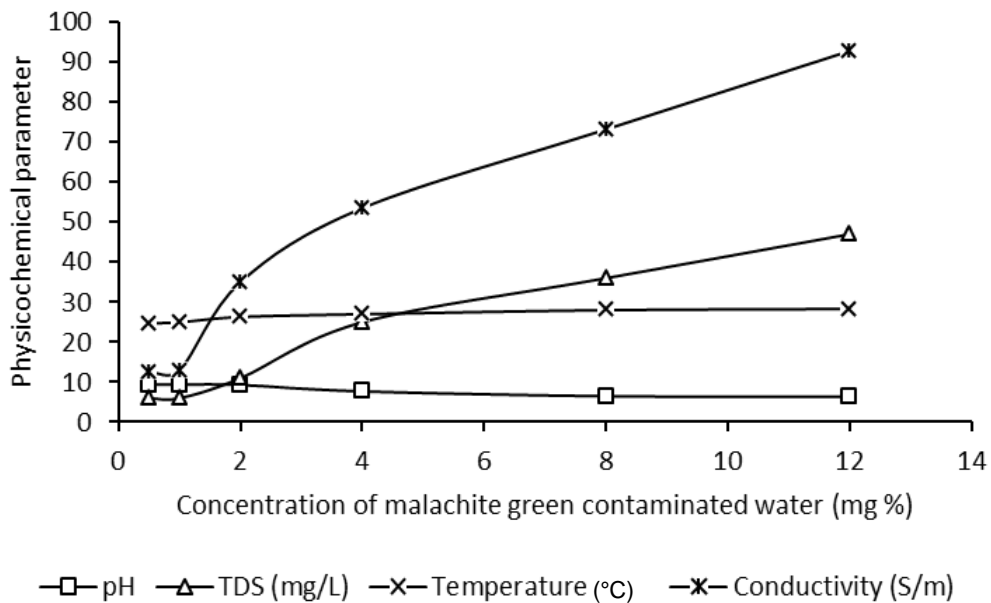


Figure 2. Physicochemical properties of malachite green contaminated water before coagulation experiment.

36°. The Carr's index of *prosopis* powder was 20.7%. In the determination of the stability of *prosopis* peel powder in different aqua media, the magnitude of the difference in weight was highest in the sample dispersed in acidic solution (23.5%) while the magnitude of the difference in weight of the samples dispersed in neutral and basic solutions were 10 and 0% respectively.

Before commencing the coagulation experiment, the malachite green contaminated water was tested for

turbidity, pH, total dissolved solids (TDS), total suspended solids (TSS), total solids (TS), temperature, conductivity and salinity. The turbidity, salinity (%), TSS and TS (mg/L) were infinitesimal (0) while all other parameters varied in a concentration dependent manner except the pH (6.4 to 9.4), as shown in Figure 2. The result of the coagulation experiment shows that the percentage of the dye removed increased proportionally with the concentration of PAPP (Figure 3). The pH of the

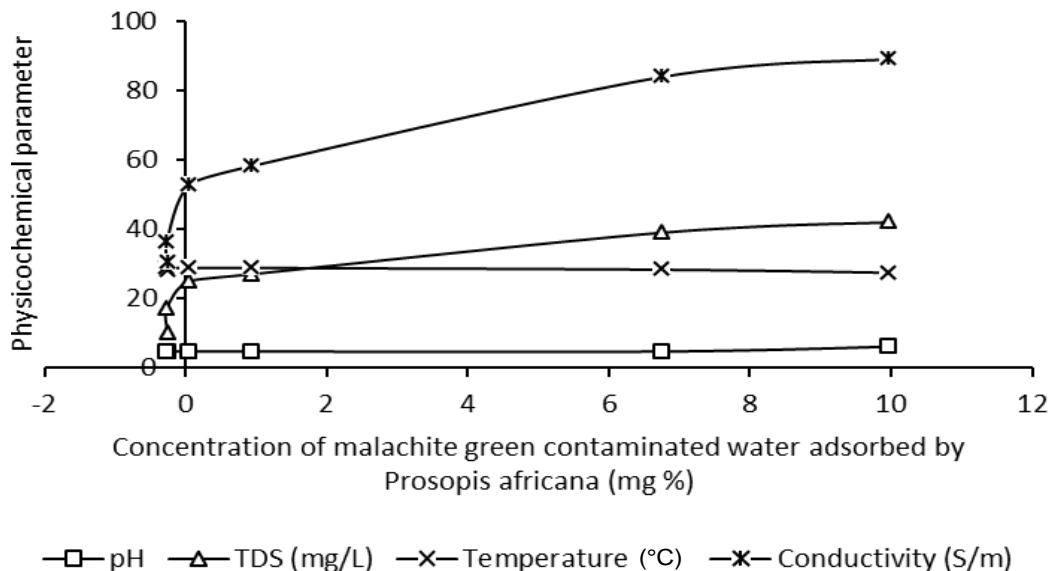


Figure 3. Post PAPP coagulation physicochemistry of malachite green contaminated water.

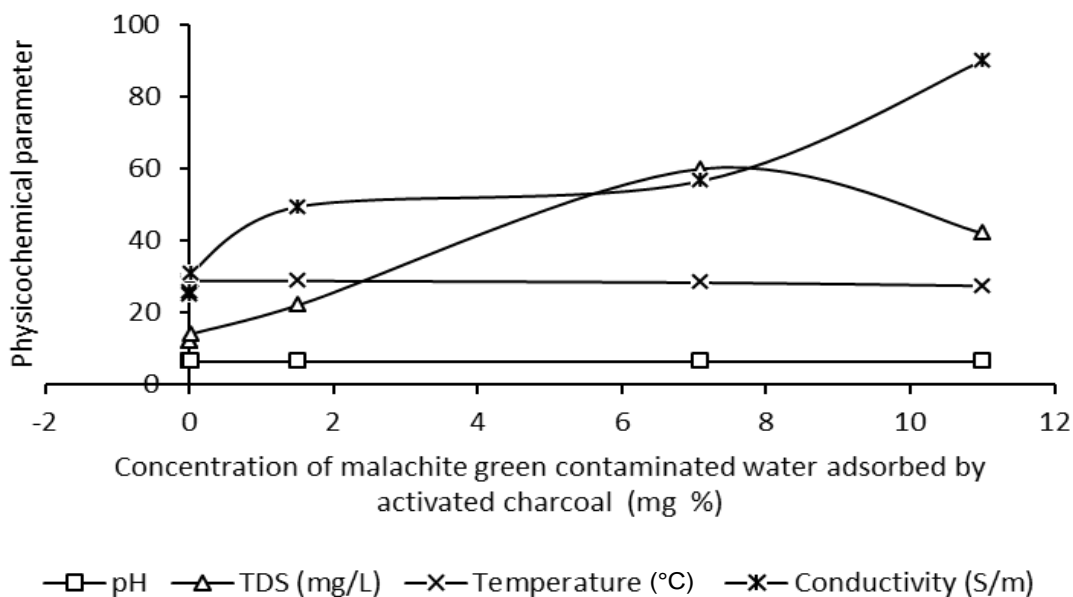


Figure 4. Post activated charcoal coagulation physico chemistry of malachite green contaminated water.

solution was 4.67. The salinity was zero but TS and turbidity were only pronounced at higher concentrations while the TSS was infinitesimal. Similarly, the result obtained when activated charcoal was used as the primary coagulant shows that the percentage of the dye removed increased proportionally with the adsorbent dosage for each dye concentration (Figure 4). The pH was 6.37. The turbidity, TSS, TS and salinity were zero but TDS and conductivity (S/m) were concentration dependent. Comparative adsorptive capacity of *P.*

africana and activated charcoal in malachite green contaminated water are depicted in Figure 5.

DISCUSSION

The nutritional content of *prosopis* more especially the protein content explains why the local people of Nigeria that process *prosopis* as a food condiment usually use *prosopis* peel as fodder for domestic animals (Aremu et

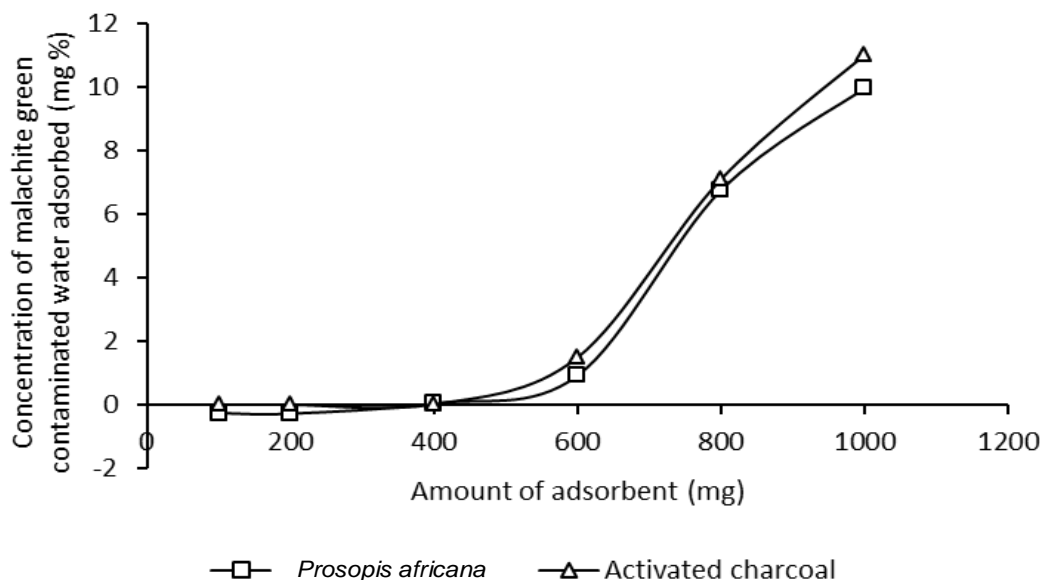


Figure 5. Comparative adsorptive capacity of PAPP and activated charcoal on malachite green contaminated water.

al., 2006; Agboola, 2004). The point of zero charge of the *prosopis* peel powder surface was determined to understand its role in the removal of the dye molecule from the aqua system and to understand and optimize the influence of pH on the dye removal (Oladoja and Aliu, 2009). Interaction of cations with the surface of a material is favoured at $\text{pH} > \text{Ph}_{\text{PZC}}$ while the interaction of anions is favoured at $\text{pH} < \text{Ph}_{\text{PZC}}$. This shows that at a pH less than 4.8 the surface of the *prosopis* peel is predominated by negative charges. Since malachite green (chloride or oxalate salts of triarylmethane dye) refers to the coloured cation and not the chloride or oxalate anions of the salt which have no effect on the colour, it implies that the PAPP which adsorbed the coloured cation of the dye molecule at a pH of 4.67 just below the Ph_{PZC} 5.6 had a surface that was negatively charged. The flow rate result suggests a fairly flowing powder. The Carr's compressibility describes inter-particulate interaction and is an indication of the flowability of a powder. Carr's index of greater than 25 is considered to be an indication of poor flowability and below 15 is of good flowability (Well, 2003). The Carr's index of *prosopis* powder invariably credits it as a fairly flowing powder, consistent with previous studies (Attama and Adikwu, 2000; Adikwu et al., 2003). The appreciable loss in weight observed in the sample dispersed in acidic solution suggests some reaction due to inorganic components, probably CaCO_3 in acidic solution, which may lead to the evolution of CO_2 . It also gives an idea of why there was a change from colourless to light pink while sedimenting in distilled water in the coagulation experiment of malachite green with *prosopis* alone. The colour change may suggest a hydrolytic degradation process. It is believed that by the

time we properly investigate the chemical constituents of PAPP (which is currently in progress) in terms of atomic absorption spectrophotometry and x-ray diffraction, that the highest percentage of the metals to be leached would be found in acidic solution while the least percentage would be found in the basic solution.

Within most solid materials, a current arises from the flow of electrons, which is called electronic conduction. In water and ionic materials or fluids a net motion of charged ions can occur. This phenomenon produces an electric current and is called ionic conduction. When malachite green was dissolved in distilled water, it dissociated into its coloured cations and non-coloured anions. The motion of these ions produced electrical current which was measured by the conductivity meter. The conductivity is directly proportional to the concentrations of malachite green contaminated water. This is because, as the concentration of malachite green increases, the total ions that produced the electric current also increases. Electrical conductivity (EC) estimates the amount of total dissolved salts (TDS), or the total amount of dissolved ions in the water. Total dissolved solids (TDS) is a measure of the total ions in solution. EC is actually a measure of the ionic activity of a solution in terms of its capacity to transmit current. In dilute solution, TDS and EC are reasonably comparable (Oladoja and Aliu, 2008). The TDS of a water sample based on the measured EC value can be calculated using the following equation: $\text{TDS (mg/L)} = 0.5 \times \text{EC (dS/m or mmho/cm)}$ (Oladoja and Aliu, 2009). This equation proves that as the total dissolved ions of malachite green increases, the electrical conductivity of the malachite green contaminated water increases.

In the coagulation experiment, the probable interactions between the peel powder, activated charcoal and the malachite green molecules are: a) the dye may either be precipitated or adsorbed on the surface of the peel powder or the activated charcoal; b) the interparticulate contact between the adsorbents and the dye molecules will enhance the colloidal load of the aqua system due to their surface areas. Our preliminary experience with alum as a primary coagulant showed that there was no variation in the dye removal even when we increased the concentration of alum, at constant concentration of malachite green. Therefore, alum had no precipitating ability on malachite green contaminated water. The mechanism of dye removal from aqua media involves a chemical interaction between the partially hydrolysed Iron (Fe) or Aluminum (Al) coagulant and some acidic group on the coloured molecules, forming an insoluble basic salt (Valh et al., 2011). The insolubility of this resultant basic salt causes precipitation, removing from the solution both the colour and the coagulant compounds. The slight solubility of certain salts of coloured compounds and coagulant is frequently responsible for some of the colour remaining even after optimization conditions are obtained for the removal (Kumar et al., 2005; Kumar, 2007). Since malachite green is a basic dye, the formation of an insoluble basic salt is unlikely hence the removal of colour by alum precipitation did not occur. The addition of the peel powder to the different dye solutions produced water with very low residual dye concentration. The dye removed could be ascribed to adsorption on the large surface area of the peel, consistent with earlier studies (Arami et al., 2005; Artioli, 2008). The value of the bulk density of *prosopis* peel powder (0.633 g/cm^3) showed that it is less dense than water (1.00 g/cm^3) hence the sedimentation of some of the dye-loaded peel powder occurred slowly for weeks. As a result, sedimentation of some of the very less-dense peel powder particles was done at the time of sampling for residual dye quantification and this made the treated water a little turbid (at a higher dye concentrations) and residual dye concentration high. Compared with powdered activated charcoal, a standard coagulant (Malik et al., 2007), *P. africana* peel powder produced less coagulant effects and thus is less efficient than activated charcoal in removing the dye from malachite green contaminated water. The adsorptive capacity of PAPP would be enhanced by activation.

Conclusions

Application of adsorption processes in wastewater treatment for the removal of colour from industrial wastewaters is highly desirable owing to severe problems the latter poses to human health and aquatic life. Naturally-occurring (plant-derived), eco-friendly, local, cost-effective, readily-available and efficient adsorbents could substitute current expensive adsorbents and

methods of removing dyes from wastewater in wastewater treatment. Assessment of the sorbing properties of malachite green contaminated water on *P. africana* peel powder (PAPP) and powdered activated charcoal demonstrated positive results. This study has shown that, although the coagulant effect of PAPP was less than that of powdered activated charcoal (a standard coagulant) on malachite green, PAPP could be employed as a potential sorbent in remediating malachite green contaminated water. A study is currently in progress to activate PAPP and compare its dye removal efficiency with activated charcoal. More so, further studies would seek to investigate the sorbent characteristics of PAPP on other basic dyes for possible industrial utilization in the treatment of dye contaminated wastewaters.

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