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African black velvet tamarind (*Dialium guineense*) as a green adsorbent for groundwater remediation

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ABSTRACT: Process shells of black velvet tamarind fruits was investigated as an adsorbent for the potential removal of the pollutant heavy metal ions of Fe, Cr, Pb, Mn and Zn in well water samples collected from six different locations in Enugu Metropolis, Eastern Nigeria. Fourier Transform Infra-Red Spectroscopy absorption bands characteristic of stretching, scissoring and bending vibrations of - O-H groups of alcohols (R-OH) and phenols (Ph-OH), Sp^3 (–C-H) of an oxygen bonded carbon atom, –C-O-C- bond and Sp^2 (---C==C—H) carbons in a ring structure were seen between 3400 to 3200 cm^{-1}, 1438 to 1371 cm^{-1}, 2922/2851 cm^{-1} and 1155/1028 cm^{-1} as well as the 1509 cm^{-1} in the infra-red region of the electromagnetic spectrum in the infra-red absorption spectrum of processed black velvet tamarind fruit shells. Metal ion analysis was carried out by Flame Atomic Absorption Spectroscopy. The results showed that the adsorbent removed 40.50 to 63.30, 33.33 to 52.63, 26.23 to 48.05 and 15.00 to 37.84% respectively of Fe, Zn, Cr and Mn in the well water samples but was ineffective in removing Pb. This demonstrates the usefulness of black velvet tamarind fruit shells in decontaminating heavy metal burdened water and suggests that the technique has potential to reclaim polluted water of such nature, ameliorate the problems of insufficient potable water and waste disposal as well as protect the environment from heavy metal pollution.

Keywords: Black velvet tamarind, decontamination, Flame Atomic Absorption Spectroscopy, Fourier Transform Infra-red Spectroscopy, heavy metals.

INTRODUCTION

Water, a naturally occurring substance attracts the interest of professionals in diverse fields because it is essential to all living things and makes up about 50% of the body weights of all living organisms (Uchechukwu, 2017). As a universal solvent (Patil et al*.*, 2013) that exists in the three phases of solid, liquid and gas, water serves as a medium for the transportation of nutrients and waste matter in these organisms (Tansel, 2008). Its quality therefore is imperative to man not only for his welfare but because scientific data corroborate important links between illnesses and the amounts/types of fluids consumed, the health-promoting properties of nutrients taken with water as well as its best intake levels and consumption patterns (Balaji et al., 2014; UNEP Joint report, 2010; Tansel, 2008).

Unfortunately, water sometimes contains heavy metals; generically referred to as harmful metallic elements characterized by an atomic weight that is greater than 40.04 (Maigari et al., 2016; Duruibe et al., 2007) and other life threatening contaminants (UNEPGEMS/Water Programme, 2006). Galadima et al. (2011) linked the outbreak of infant blood poisoning (Adie et al*.*, 2012) and many other diseases reported among Kaduna State residents to Pb contamination of drinking water sources in Nigeria. Circumstances of waterborne diseases allegedly encountered by 64,212 Indian households were traced to high Fe levels detected in their domestic water supplies (Goyal, 2015). While investigating the fundamental issues associated with bouts of heavy metal pollution in Nigeria, Galadima and Garba (2012) described humans, animals and garden crops as eventual recipients of the Pb contaminant in the drinking water, foods and soil samples in the cosmopolitan cities across Nigeria. Uchechukwu et al. (2016) listed offensive taste, odour and appearance among other features as indicators of chemical contaminant in water. They reported studies which indicated the habitual heavy metals contamination of natural water sources through human and environmental activities.

Maigari et al. (2016) traced rising ecosystem related maladies and metabolic disorders to heavy metals migration along the “soil - plant - animal - man” path in the food chain. With Sardar et al. (2013), Sharma et al. (2009), Jadia and Fulekar (2009) and Järup (2003), they explained the inability of heavy metals to decompose into harmless products and listed diminutive growth and fitness, reproductive anomalies, carcinoma and death among the toxic effects of their accumulation in humans.

For example, at 100μgL^{-1}, Mn manifests as stunted growth and weight loss, anaemia, ulcer, neurological disorders as well as elevated plasma cholesterol and triglycerides levels in animals (Reis et al., 2010). Heedless of health status, Abetz et al. (2006) explained that beyond organ damages and risk to life, excess Fe accumulates in vital organs and create non–transferrin-bound Fe when at the body’s capacity to store the metal is exceeded (Darbari et al., 2006; Porter, 2005). Encumbering protein metabolism, oxidative stress as well as failing growth and reproductive functions (Reis et al., 2010) are among the *domino* effects of the toxicity of Zn, the masculine element (Duruibe et al., 2007) in mammalian males. Partial blindness and disturbed hormonal activities are mild effects of Pb toxicity (Saka et al., 2012; Stein, 1989) while impeded brain cells development and low acumen are signs of its overload (Lead Action News, 2009). Excessive Cr (VI) is responsible for intestinal tract disorders and haemorrhage, liver necrosis and nephrities climaxing in duodenal and lung cancer (Pandey and Madhuri, 2014; Food Safety Authority of Ireland, 1999). In addition to oxidative stress, reduced nutrient absorption, chlorosis, stunted growth, low fruits yield, metabolic disorders and reduced nitrogen fixing ability are the effects of heavy metals toxicity in plants (Ogali et al., 2008).

For this reason, there is high sensitivity, interest and effort towards heavy metals removal from polluted water sources (Ademiluyi and Ujile, 2013). Fogden (2009) underscored the exigency of effective decontamination of metal ions laden domestic water supplies. Consequently, various regulatory bodies have recommended safe and permissible limits for the presence of heavy metals in drinking water, soil and sediments (Uchechukwu, 2017). The WHO permissible limits for Zn, Fe, Mn, Cr and Pb in drinking water are: 3.00, 0.20, 0.1, 0.05 and 0.01 ppm respectively (O’Connel et al., 2008; Cobbina et al., 2015; Gimba et al**.**, 2015). Galadima et al. (2011) recommended sufficient potable water provision in Nigeria for the development and advancement of the human and economic potential of the citizenry. While Shirkhanloo et al. (2015) recommended the use of decontaminated water for food cultivation, Jadia and Fulekar (2009) solicited for assent and development of effective ways of remediating heavy metal polluted water sources.

As ubiquitous as there are methods for purifying heavy metals polluted water, Tansel (2008) and Kumar (2006) described the development of sustainable means of generating environmentally friendly and practicable techniques from agricultural wastes as a current area of research. Peanut hulls and testa, sphagnum peat, orange and banana peels, rubber fruit pericarp, cotton seed, sugarcane hulls and bagasse are some agricultural by-products which have been processed and tested as heavy metal ions adsorbents (Uchechukwu et al., 2017; Jorgetto et al., 2014; Zvinowanda et al*.*, 2010; Yurtsever and Sengil, 2009). Cheap and abundantly available agricultural by-products have little or no value, are easy to process and require little or no processing time (Uchechukwu, 2017; Chen et al., 2011). They are industry wastes (Crini, 2005) containing tannins, lignins and multiple –OH groups and COOH rich polyphenolic compounds (Guo et al., 2015; Şengil and Özacar, 2009). The groups possess the capacity to remove metal ions in aqueous solutions at favourable conditions (Ewansiha et al., 2013). Argun and Dursun (2006), Babarinde and Onyiaocha (2016), Haftu et al. (2014) listed good results, high metal binding capabilities, competitive performance, no sludge generation as well as specificity and selectivity reportedly obtained using them as their inspiration to appraise bark as an adsorbent.

While Xu and Zhuang (2014) and Abasi et al. (2011) advanced that the use of agricultural wastes in polluted water decontamination would satisfy the desire of using waste materials to recover other wastes, Jaishankar et al*.* (2014) and Şengil and Özacar (2009) listed adsorption, electrostatic interaction, chelation, micro-precipitation, ion exchange or their combinations as the processes through which substrates from these wastes eliminate metal ions. Jorgetto et al*.* (2014) and Şengil and Özacar (2009) included adaptability to the processes as reasons for the usefulness and effectiveness of plant constituents as alternative adsorbents for the removal and recovery of heavy metal in polluted aqueous media.

Across the globe, communities are challenged with water shortages (Bello et al., 2014). Despite Tansel (2008)’s report that three quarters of the earth’s surface is covered by water (Oliveira et al., 2013), the rhyme of the ancient mariner ‘water, water everywhere but not a drop to drink’ dominates since only a fraction of the water is available to life and usable by humans and animals while most of it sit in the oceans (Earthlearningidea; Oliveira et al., 2013). UNEP (2010), Fogden (2009), UNDP (2006) acknowledged the privation of potable water by over a billion people in 2008 and optimised a larger number if adequate measures are not initiated to reverse it. UNDP

(2006) listed access to potable water supply as a human right but Akinola (2014) reported the unavailability of potable water supply to residents of Ikeji and many Nigeria communities. In most parts of the Eastern Nigerian town of Enugu, designated as Coal City, hand dug wells are a common source of domestic water supply. The presence of heavy metals in coal and hard rock beneath the earth in many parts of the town results to pollution of the groundwater by the heavy metals. Ibeto and Okoye (2010) reported the result of a survey which showed that the Ni, Mn and Cr above WHO permissible limits detected in the blood samples of Enugu residents came through water sources.

Native to Southern Thailand and Malaysia (Ogungbenle, 2014), *D. guineense* known variously as: tumble tree, black velvet, (English); tamarinier noir, dialium de Guinée, afambeau (French); meko, kedebe, mako, mekohi (Fula); icheku (Igbo); awin (Yoruba); Tsamiyar kurm (Hausa); Yoyi (Ghana); kosito (Mandinka) and solam, solom (Wolof) is a dehisce belonging to the family of *leguminosae* (Ewédjè and Tandjiékpon, 2011). It is a densely leafy crown shrub that grows to a height of 30 m and widely spread in the savannah regions in Nigeria and many West African countries (Ogungbenle and Ebadan, 2014). The usually whitish flowers have short stout stalks and brownish hairs while the generally abundant dense velvet, black fruits appear spherical, flat and sometimes globose, with a brittle shell enclosing a shiny (Anusi et al., 2018) seed (or exceptionally two), embedded in a dry, brownish, sweetly/sourly acidic, edible pulp. While the tree flowers from September to October and fruits from October to January in Nigeria, it is covered with smallish white flowers, ripening between March and May (or earlier and later) in Ghana. The minerals, sugars and organic acids rich fruits are used in the treatment and management of bacterial diseases and fever, diarrhea and palpitations (Ewédjè and Tandjiékpon, 2011). The seeds are dispersed either by animals or water transport (Asoiro et al., 2017; Orwa et al., 2009).

Every year, large amounts of flavonoid rich (Afolabi et al*.,* 2018) black velvet tamarind (D*. guineense*) fruits are harvested. The brownish pulps are separated from the black pod before consumption (Abiodun and Ogugua 2012), utilization in beverage (Okudu et al., 2017; Obasi et al., 2013) and medical (Afolabi et al., 2018; Besong et al., 2016) preparations while the latter is discarded haphazardly as a reject in the environment where they litter and cause many pollution problems. There is need to garner and identify uses for this abundant waste. Its application in decontaminating metal ions polluted water would ensure economic utilization and serve the dual role of wealth creation and environmental clean-up. Ahalya et al*.* (2008) extolled the advantages of environmentally friendly tamarind pod shells and recommended its use in water purification. In addition, the use black velvet tamarind (D. *guineense*) shells for this purpose will lead to attitudinal change, reduce the challenges of wastes disposal and put into practice; the principle of using wastes to treat wastes.

A number of researchers have characterized black velvet tamarind (D. *guineense*) (Asoiro et al*.,* 2017; Utubaku et al*.,* 2017; Ogungbenle, 2014; Adamu et al., 2015; Osanaiye et al*.,* 2013), evaluated its potential in chemicals (Anusi et al., 2018; Ajiboye et al., 2015), beverages (Abiodun et al., 2017; Onwuka and Nwokorie, 2006; Nguyen, 2015) and medical preparations **(**Eze et al., 2018) as well as a corrosion inhibitor (Osarolube and James, 2016). There are also reports about the use of Indian tamarind (*T*. *indica*) kernel powder (TKP) to remove heavy metal ions from a variety of polluted water samples. For example, using it, Singh and Kumawat (2012) removed Pb(II), Cd(II), Cu(II), Zn(II) and Fe(II) from industry wastewater; Singh et al. (2010) carried out metal and waste water purification as well as selective binary separation of Cu^{2+} Zn^{2+}, Cd^{2+} and Pb^{2+} ; Sharma and Singh (2005) removed Fe^{2+}, Cu^{2+}, Zn^{2+}, Cd^{2+} and Pb^{2+} from the Agucha open cast mine water, Bhilwara (Rajasthan), India while Gupta and Babu (2009) carried out the equilibrium, kinetics and regeneration studies involved in its removal of Cr (VI) in aqueous solutions. After carbonization, Pandharipande and Kalnake (2013) used Indian tamarind fruit shells to purify Cr(VI) and NI(II) ions synthetic feed solutions and even though, Ahalya et al. (2008) and Popuri et al. (2007) respectively had separately utilized *T*. *indica* fruit shells to remove Cr(VI) in a simulated aqueous mixture and in the comparative study of the biosorption of Cr^{6+} from aqueous solutions, scientific literature is deficient of information about the use of any part of black velvet tamarind (D*. guineense*) in polluted water remediation. The aim of this study is to determine the extent of the removal of contaminant Zn, Pb, Mn, Fe and Cr in ground water by fruit shells of Nigerian black velvet tamarind (*D. guineense*). In addition to the desire to guarantee sustainable potable water supply, interest in the investigation of black velvet tamarind (*D. guineense*) fruit shells for water purification was motivated by the successful results achieved and published using the fruit shells of *T. indica* and other polyphenol-rich agricultural wastes in wastewater purification.

MATERIALS AND METHODS

New Brunswick G10 gyratory, Merck Millipore pH 0 – 14 Universal indicator paper, Agilent Technologies Cary 630 Fourier Transform Infra-Red Spectrophotometer (4000 - 650 cm^{-1}) pycnometers, whatman filter paper, Varian Spectra *AA* 220 FS model atomic absorption spectrophotometer were apparatuses used in this study while the reagents: deionized water (DI H_2O), hydrochloric acid (HCl), nitric acid (HNO_3), sodium hydroxide (NaOH), lead (II) nitrate ($(PbNO_3)_2$), zinc sulphate heptahydrate ($ZnSO_4.7H_2O$), iron (II) sulphate heptahydrate ($FeSO_4.$

$7H_2O$), manganese (IV) oxide (MnO_2), chromium (III) hexahydrate ($CrCl_3.6H_2O$) and any other chemical used were of analytical grade, obtained from the Department of Industrial Chemistry, Enugu State University of Technology, Enugu, (ESUT) Nigeria and were used without further purification.

Collection of water samples

Water samples were collected from six different hand dug wells located at different parts of Enugu Metropolis, Eastern Nigeria. Housekeeping around the wells which serve as the only sources of domestic water supply to families resident in the areas was impressive. Each sample was collected by filling a 2 litres sterilized and labelled plastic bottle with water from a particular well (WS1, WV1, WW1, WX1, WY1 and WZ1) using a stationed rubber fetcher dedicated for the purpose. The bottle was rinsed twice with a particular well water before filling. After withdrawing 50 mL of water from each bottle using a measuring cylinder, 50 mL of dilute HNO_3 was added to prevent the precipitation of the constituent metal ions before covering and transporting the bottles to the laboratory.

Sampling

Shells of fresh black velvet tamarind (D. *guineense*) fruits were obtained from a seller at Eke Ugbor market, Akama Oghe, Enugu State, Nigeria. It was identified by the Department of Applied Biology and Biotechnology at ESUT. After sorting and removal of dirt, black velvet tamarind fruit shells was thoroughly washed, air-dried for a week and further dried in the oven at 110°C for 2 hours. It was subsequently weighed and ground using a hand mill to obtain 250 µm particle size. 1 kg of unprocessed shells yielded 500 g of black velvet tamarind fruit shells (*D. guineense*) powder (BVTP) was stored in a polyethylene bag at laboratory conditions until it was ready for use.

Preparation of calibration solutions

A multi-metal primary stock solution containing 100 ppm each of Zn, Fe, Cr, Pb and Mn ions was prepared by dissolving 0.1100, 0.1245, 0.1281, 0.04 and 0. 0395 g respectively of $ZnSO_4.7H_2O$, $FeSO_4.7H_2O$, $CrCl_3.6H_2O$, $Pb(NO_3)_2$ and MnO_2 in a little DI H_2O contained in a 250 ml standard flask and diluting to volume with DI H_2O. 1, 2, 5 ppm calibration solutions as well as a 4 ppm quality control (QC) standard respectively were then prepared by pipetting 5, 10, 25 and 20 ml aliquots from the stock solution, transferring into separate 500 ml standard flasks and diluting to the mark with DI H_2O.

Characterization

Processed BVTP was characterized according to specific gravity (Sp. g $_{(g/L)}$) and native functional groups which were respectively determined by means of a pycnometer and FTIR Spectrometry at room temperature (RT). The specimen was thoroughly mixed with nujol before FTIR analysis.

Adsorption studies

BVTP was first activated by soaking overnight in dilute HNO_3 and washing to a neutral pH with DI H_2O before using for adsorption experiments. For this purpose, 10 g of the acid activated BVTP was suspended in 100 mL of the six different well water samples held in separate 250 mL Erlenmeyer flasks. The procedure was repeated in another set up which served as duplicate samples. The pH of each water sample was adjusted to 6 ± 0.05 using Millipore pH 0 – 14 universal indicator paper before adding the adsorbent. The flasks were screwed on to the G10 gyrator and agitated at 150 rpm (revolutions per minute) for 2 hours at room temperature (RT). At the end of the process, they were disengaged and the suspensions rapidly filtered on a whatman No. 2^V folded filter paper. The filtrates were labelled as follows: WS2, WV2, WW2, WX2, WY2 and WZ2 and WS2', WV2', WW2', WX2', WY2' and WZ2'. The total concentration (ppm) of each of the metals expressed as: Fe^{2+}, Zn^{2+}, Cr^{3+}, Pb^{2+} and Mn^{4+} in the filtrates was determined by Atomic Absorption Spectrophotometry using air: acetylene flame system. The amount of each metal ion removed from the untreated water samples (WS1, WV1, WW1, WX1, WY1 and WZ1) of predetermined metal ion concentration by the adsorbent at equilibrium *Qe,* was computed using the model**:**

$$Qe = \left[\left(\frac{C_o - C_e}{M}\right)\right] x\ L \qquad (1)$$

Where: C_o and C_e are the initial concentration and final concentration (ppm) of the metal ion remaining in the water at equilibrium while L and M respectively are the volume of the water and the weight of the adsorbent (g) used in the experiment.

The average of two values was reported in their percentages. A quality control standard containing 4 ppm of each metal ion was tested along with the samples before and at the end of the determinations in order to verify the performance of the spectrophotometer.

RESULTS AND DISCUSSION

The Sp. g of BVTP was determined to be 0.32 g/mL. The FT-IR spectrum of BVTP is presented in Figure 1. Strong and broad absorption bands characteristic of -O-H groups stretching of alcohols (R-OH) and phenols (Ph-OH) were seen between 3400 to 3200 cm^{-1}. Medium peaks found in the 1438 to 1371 cm^{-1} regions are probably due to in-plane

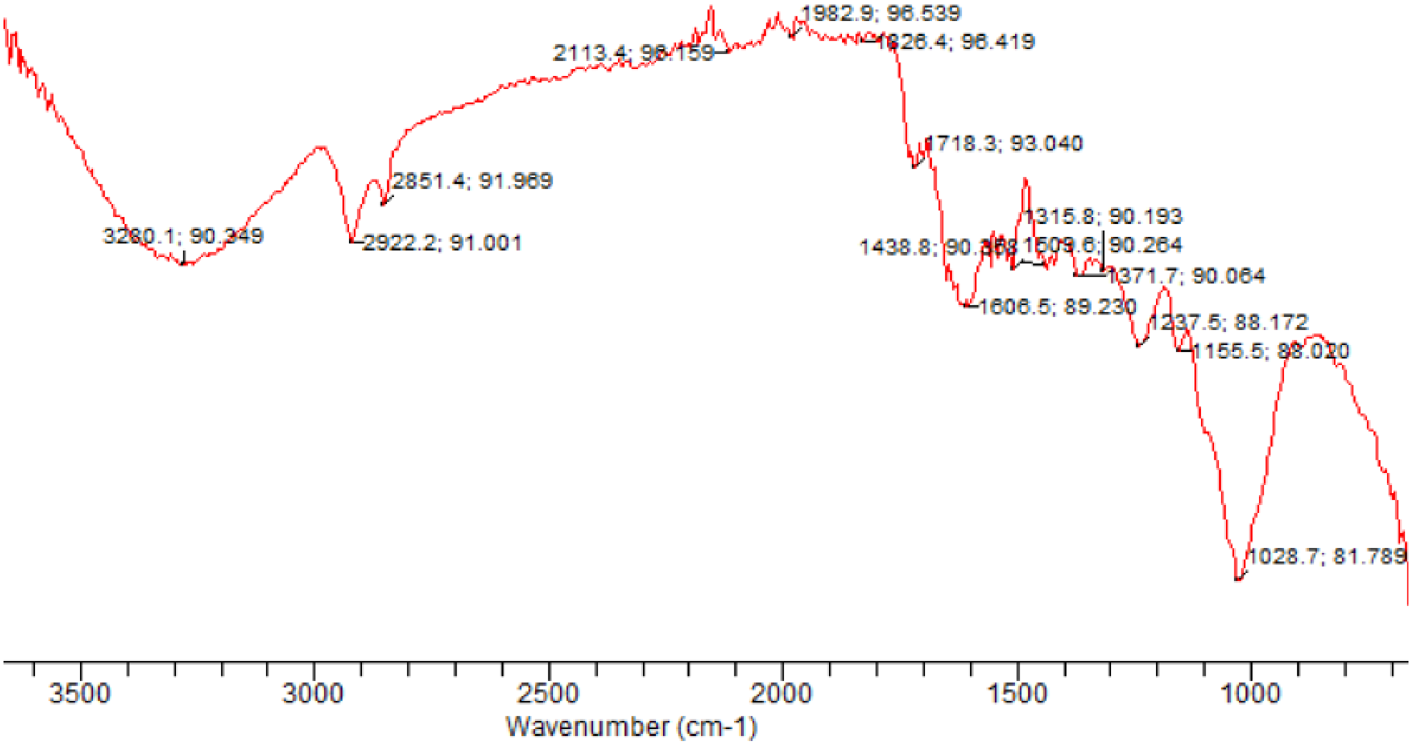

Figure 1. FTIR spectrum of BVTP.

and out-of-plane bending vibration of secondary -OH groups in the chemical structure of black velvet tamarind (*D. guineense*). A twin singlet and sharp peaks respectively found at 2922/2851 cm^{-1} and 1155/1028 cm^{-1} are thought to occur from stretching and bending vibrations of Sp^3 (–C-H) of an oxygen bonded carbon atom in the tamarind molecule. These peaks at 1155/1028 cm^{-1} may also be as a result of stretching/scissoring movements indicative of the presence of –C-O-C- bond in the molecule. The doublet at 1606 and the singlet at 1509 cm^{-1} suggest stretching movements by Sp^2 (---C==C—H) carbons in a ring (Meyer, 2000).

Figure 2 is a graphical illustration of the amounts of Fe^{2+}, Zn^{2+}, Cr^{3+}, Pb^{2+} and Mn^{4+} in the different well water samples removed by BVTP. The results show that apart from well WS where 8.33% of Pb^{2+} showed to have been removed, BVTP removed zero amount of Pb^{2+} from the rest of the well water samples. Inability of BVTP to remove Pb^{2+} in the water samples may have been due to the comparatively large ionic radius of the Pb^{2+} with respect to the co-metal ions in the water. While Mostafa et al. (2012) explained the relatively smaller atomic size and ionic radius of Hg^{2+} as reasons for the higher removal of the heavy metal from the solution in comparison with Pb^{2+} by the synthesized 6-poly-dimethylaminoethyl-methacrylate-rice-straw (PDMAEMRS) grafted copolymer, Ogali et al. (2008) and Horsfall et al. (2003) respectively attributed the higher adsorption of Mg^{2+} over Cd^{2+}, Zn^{2+}, Cu^{2+} and Pb^{2+} and Cu^{2+} over Zn^{2+} by DPS-OME cation exchange resin and untreated and acid treated cassava (*Manihot esculenta cranz*) waste biomass to the smaller ionic radii of Mg^{2+} and Cu^{2+} with respect to the co-ions in their solutions. Moreover, Uchechukwu (2017) peanut testa extract (PTE) demonstrated zero adsorption of Pb^{2+} in laboratory simulated Pb water solutions.

The ionic radius of a chemical element is a measure of the size of its ion or the mean distance from the centre of the nucleus to the boundary of the surrounding cloud of electrons. The ionic radii of Fe^{2+}, Zn^{2+}, Cr^{3+}, Mn^{4+} and Pb^{2+} respectively are: 0.645Å, 0.74Å, 0.52 Å, 0.46 and 1.19Å (Barbalace, 2018).

The result also showed that BVTP demonstrated maximum Fe^{2+} removal efficiency in all the samples. In addition to relative ease of reactivity of Fe^{2+} in the presence of moisture, the observation may be attributable to the tendency of Fe^{2+} to chelate with phenolic groups bearing catechins and other tannins to produce metal complexes (Shen et al., 1997). Despite the report by Nada et al. (2005) which indicated a metal ion adsorption trend of $Fe^{2+} > Mn^{4+} > Zn^{2+} > Pb^{2+}$ by chemically modified cotton wastes and the obvious greater ionic radius of Zn^{2+} in comparison of Mn^{4+}, more Zn^{2+} than Mn^{4+} was removed from the water samples by BVTP.

Although Zn and Mn levels in all the well water samples tested (except for WS1) in this study showed to be within WHO permissible levels, treatment with BVTP showed to

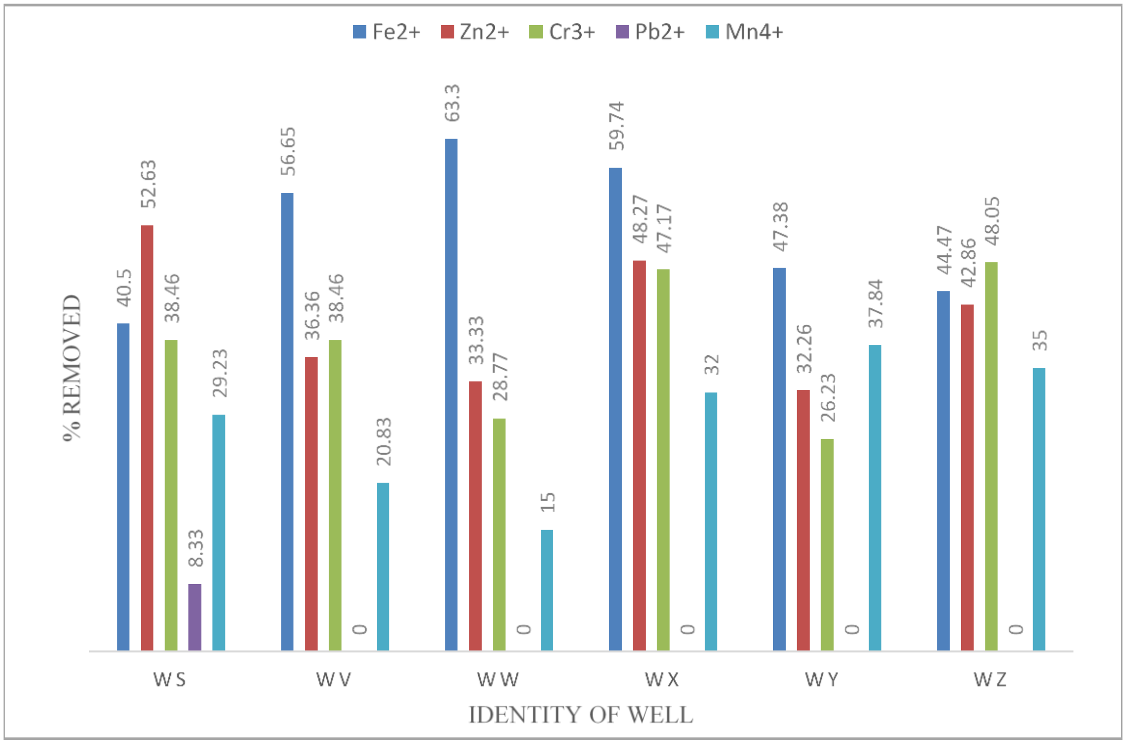

Figure 2. Percentage adsorption of Fe^{2+}, Zn^{2+}, Cr^{3+}, Pb^{2+} and Mn^{4+} in well water by BVTP.

reduce their concentrations to even safer levels. Total Cr concentration in all the samples which exceeded WHO permissible limits was reduced to permissible and much lower levels; thus, much safer levels respectively in sample WS1 and the other wells (WHO, 1993). The WHO permissible limit for total Cr in potable water is 0.05 mg/l out of which 38.46, 40.00, 28.77, 47.17, 26.23 and 48.05% respectively were removed from the well water samples: WS, WV, WW, WX, WY and WZ.

The ability of BVTP to remove the heavy metal pollutants in the well water samples studied in this research is as a result of its chemical constitution. Its FTIR spectra suggests an abundance of –OH and Ph-OH groups in its chemical structure like most agricultural wastes of plant origin. These characteristically high polarity –OH and Ph-OH groups possess natural ability to interact with, bind and remove metal ions from their aqueous solutions (Ewansiha et al., 2013; Elaigwu et al., 2009).

Conclusion

The findings from this study shows that large volume black velvet tamarind fruits shells which is usually thrown away can be harnessed and usefully employed to decontaminate heavy metals polluted water. The research supports the potential application of this simple and safe technique for the purposes of rehabilitating polluted water, mitigating the burden of water scarcity and waste disposal as well as protecting the environment from heavy metal pollution. It is therefore a significant way to utilize tamarind fruits shells utilization for sustainable development.

Recommendation for further study

The present study was undertaken during the rainy season when liberal precipitation could lower heavy metals concentration in groundwater. It is suggested to assess the extent of contamination of the water samples by these pollutants in the dry season and repeat the tests in order to evaluate how much of the contaminants BVTP would remove from polluted water samples of higher heavy metal levels as a further study. Secondly, it was observed that BVTP imparted an orange-red colouration to the water samples under investigation. It is therefore recommended to immobilize the soluble components of BVTP through chemical or structural modification to avoid the problem of secondary pollution.

CONFLICT OF INTEREST

The authors declare that they have no conflict of interest.

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