Banana Peduncle Biochar: Characteristics and Adsorption of Hexavalent Chromium from Aqueous Solution

Adnan Asad Karim¹*, Manish Kumar¹, Sanghamitra Mohapatra¹, C. R. Panda¹ and Ankit Singh²

¹Department of Environment and Sustainability, CSIR-Institute of Minerals and Materials Technology, Bhubaneswar-751013, India.
²School of Biotechnology, KIIT University, Bhubaneswar, Odisha, India.

Authors’ contributions
This work was carried out in collaboration between all authors. Authors AAK and MK designed the study, performed adsorption experiment and wrote the protocol. Authors SM, CRP and AS help in the characterization of biochar. All authors read and approved the final manuscript.

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ABSTRACT

Aims: Biochar produced from different waste biomass has been documented as an adsorbent for heavy metal removal from contaminated water. In present study, Banana peduncle which considered as waste, but abundantly available and have high biomass was selected for production of biochar to investigate its adsorption capacity for hexavalent chromium.

Place and Duration of Study: Environment and Sustainability Department, CSIR-Institute of Minerals and Materials Technology (CSIR-IMMT), Bhubaneswar, India, between April 2014 and October 2014.

Methodology: Banana peduncle pyrolysed at 300°C and 500°C temperature and characterized by proximate, total carbon, FE-SEM, FTIR and XPS analysis. The adsorption of Cr (VI) studied through the batch experiment at different pH, adsorbent dose, and initial concentration.

Results: The banana peduncle biochar yield was 66 and 40% at 300°C and 500°C respectively.
1. INTRODUCTION

Chromium is a heavy metal and relatively more toxic pollutant in soil and water. It mainly exists in the hexavalent and the trivalent form. The compounds of Cr(VI) needs more attention because they are highly water soluble, mobile, non-biodegradable, toxic, mutagenic and carcinogenic while Cr³⁺ compounds have very low solubility and reactivity resulting in low mobility in the environment and hence low toxicity in living organisms. The hexavalent chromium mainly exists as chromate anions (HCrO₄⁻, Cr₂O₇²⁻) in acidic medium and dichromate anions (CrO₄²⁻) in basic medium [1,2]. Adsorption recognized as an efficient method for removal of Cr (VI) from wastewater. Globally activated carbon and ion-exchange resins are widely used for adsorption but found to be less utilized in developing countries due to their high cost and operational complexities [3]. This has encouraged research for the production of less costly and efficient adsorbents.

Low cost adsorbent mostly requires less processing, abundant in nature, by-product or waste material from industry [4-9]. Biochars produced from various biomass waste has emerged as an important low cost adsorbent for heavy metal removal from water and soil. Biochar from pine wood, pine bark, switch grass, softwood, buffalo weed, corn straw hardwood, canola, peanut and soybean straw etc., has been found to have significant adsorption capacity for heavy metals [10,11]. Biochar prepared from sugar beet tailings, oak wood and oak bark were explored for adsorption of Cr (VI) and they showed the monolayer adsorption capacity of 123 mg/g, 4.93 mg/g and 7.51 mg/g respectively. The main mechanism for removal of Cr (VI) was reported to be adsorption coupled reduction and complexation with carboxyl and hydroxyl group of biochar [12-15].

Biochars are mainly produced through pyrolysis i.e. heating in the absence of oxygen. This process adds to the cost of biochar production by demanding the use of inert gas or totally vacuum condition. Therefore, cost-effective production of biochar and its application as an adsorbent needs more research. This can be achieved by the selection of right waste biomass and production method. Banana being an annual crop, its cultivation generates nearly 220 t/hectare abundantly available plant residual waste which mainly consists of lignocellulose material [16]. Previous researchers had explored banana peel [17,18], banana pith [19] and banana stem [20] for removal of heavy metals from contaminated waters. The present study focused on preparation of biochar from banana peduncle (the stalk holding banana bunches) in limited amount of oxygen and without utilizing inert gas to study its adsorption capacity for hexavalent chromium. Banana peduncle contains a considerable amount of cellulose (48.31-60.41%), hemicellulose (10.20-15.75%) and lignin (17.56-20.66%) [21]. The objectives of the present investigation were to (1) study the effects of various parameters including pH, initial Cr (VI) concentration and adsorbent dose, (2) determine the adsorption isotherm and the mechanisms governing Cr (VI) removal by banana peduncle biochar.

2. MATERIALS AND METHODS

2.1 Preparation of Biochar

The banana peduncle (Musa species) was used to prepare biochar through slow pyrolysis. It was reduced into small pieces, washed and oven dried at 60°C for three days. The dried peduncles placed in a lid covered crucible pyrolysed in muffle furnace at two different temperatures, i.e., 300 and 500°C with heating rate of 50°C for 1 hour, followed by cooling to
room temperature inside the furnace. Subjected temperatures for pyrolysis were based on the results of earlier reports [11]. Prepared biochars were sieved using an aperture sieve to obtain particle size in the range of ≤100 microns and stored in the air tight container. Biochars prepared were referred as BC-300 (Banana peduncle biochar prepared at 300°C), BC-300 Cr (BC-300 after chromium adsorption), BC-500 (Banana peduncle biochar prepared at 500°C), and BC-500 Cr (BC-500 after chromium adsorption).

2.2 Characterization of Biochars

The biochar yield (percentage of biochar obtained after pyrolysis) was calculated using the formula, i.e., Yield (%) = Ma/Mb×100; Where, Ma and Mb are the mass of sample after and before pyrolysis respectively. The pH of biochar was measured with digital pH meter (Thermo Scientific Orion STAR A215) after adding it to distilled water at a ratio of 1:20 and shaken for 10min. The proximate analysis (moisture, ash, volatile matter and fixed carbon content) was carried out in a Thermo gravimetric analyser (LECO TGA 601) following ASTM method D5142. The total carbon content of biochar samples were determined by using an Elemental analyser (VARIO TOC Cube). The morphology of biochar samples was analyzed in Field Emission Scanning Electron Microscopy (FE-SEM) with Carl Zeiss SUPRA 55VP. Elemental detection of material done with an energy dispersive X-ray microanalyser (OXFORD ISI 3000 EDAX) attached to the scanning electron microscopy. Fourier transform infrared (FTIR) analysis was utilized to verify the presence of surface functional groups and changes in the biochar before and after adsorption. FTIR analysis was made by using IR Prestige-21, FTIR-8400S, SHIMADZU Corporation (Kyoto, Japan). For this, 0.1 g biochar mixed with 1 g of KBr, spectroscopy grade (Merck, Darmstadt, Germany) and spectra obtained in the range from 400 to 4000 cm⁻¹. In addition, X-ray photoelectron spectroscopy (XPS) measurements were carried out to determine the valence state of chromium on the biochar surface after adsorption. For XPS analysis, pellets of biochar were made by a pelletizer after mixing it with a binder (Poly Vinyl alcohol). The X-ray photoelectron spectra (XPS) of the biochar measured on a Prevac photoelectron spectrometer equipped with a hemispherical VG SCIENTA R3000 analyser. The biochar samples loaded through a load lock into an ultrahigh vacuum analytical chamber. XPS measurements were taken with a monochromatized aluminum source Al Kα (E=1486.6 eV) and a low energy electron flood gun (FS40A-PS) to compensate the charge on the surface of nonconductive samples. The peaks recorded with constant pass energy of 100 eV for the survey and high resolution spectra. The binding energies referenced to the C 1s core level (284.6 eV) for hydrocarbon contaminations.

2.3 Adsorption Experiments

For the Cr (VI) adsorption study, the stock solution of Cr (VI) was prepared using analytical grade K₂Cr₂O₇ (MERCK) in distilled water. All working solutions were prepared by diluting the stock solution with distilled water, and the pH adjusted to the desired values with 1 M HCl or 1 M NaOH solution. The effect of pH on removal of Cr (VI) by biochar was investigated by varying solution pH from 2.0 to 10.0 while keeping Cr (VI) at 100 mg/L and biochar dose at 2 g/L. The effect of biochar dose studied in the range of 0.2–8 g/L while keeping 100 mg/L Cr (VI) concentration and pH at 2. Equilibrium experiments were conducted with Cr (VI) concentrations range of 50 - 800 mg/L. Solution were agitated in a thermostated mechanical shaker at 140 rpm at room temperature (27±0.5°C) for 24 hours. After each adsorption experiment, known volume of the solution was filtered by Whatman filter paper-41 for Cr (VI) analysis. The Cr (VI) concentration was measured by UV-visible spectrophotometer (Vario) after development of a purple violet color with 1, 5diphenylcarbazide in acidic solution measured at 540 nm. The chromium concentration retained in the adsorbed phase (adsorption capacity) was calculated by following equation-1:

\[ q_e = \frac{(C_0 - C_e) \times V}{W} \quad (1) \]

Where, \( q_e \) is adsorption capacity, \( C_0 \) and \( C_e \) are the initial and equilibrium concentrations (mg/L), of Cr(VI) in solution respectively, \( V \) is the volume (l), and \( W \) is the weight (g) of the biochar as adsorbent. The removal of Cr (VI) ions was calculated by following equation-2:

\[ \text{Removal (\%)} = \frac{C_0 - C_e}{C_0} \times 100 \quad (2) \]
3. RESULTS AND DISCUSSION

3.1 Characteristics of Biochar

The characteristics of banana peduncle biochar derived at two pyrolysis temperatures presented in Table 1. The biochar yield at 300°C and 500°C was 66% and 40% respectively. Biochar yield varies according to the feedstock type, pyrolysis temperature, and heating rate. The high biochar yield also related to higher inorganic constituents (indicated by high ash content) and high lignin content in biomass [11,22]. With the rise in the pyrolysis temperature from 300°C to 500°C, the biochar yield decreases due to decomposition of lignin and cellulose in the feedstock [23]. Higher volatile matter content observed in BC-300 (33.69%) in comparison to BC-500 (24.37%) and a higher moisture content in BC-300 (26.28%) than BC-500 (21.26%). The fixed matter content is more in BC-500 (18.89%) in comparison to BC-300 (15.19%) which makes BC-500 more stable. Ash contents also increased from 24.84% in BC-300 to 35.48% in BC-500. Increase in ash content with rise in temperature was may be due to the concentrations of minerals and organic matter combustion residues [24]. The pH of BC-300 was 8.1 and 10.1 for BC-500, which may attributed to the alkali salts separation from the organic matrix in the feedstock [25]. The total carbon content was found to be 37.79% in BC-300, and 41.86% in BC-500 indicating increased carbonization as pyrolysis temperature increases. Similar observations in biochar with increasing pyrolysis temperature have reported by other researchers [26-28].

### Table 1. Characteristics of Banana peduncle biochar

| Parameters             | BC300   | BC500   |
|------------------------|---------|---------|
| Biochar Yield (%)      | 66      | 40      |
| pH                     | 8.1     | 10.2    |
| Moisture (%)           | 26.28   | 21.26   |
| Volatile matter (%)    | 33.69   | 24.37   |
| Ash content (%)        | 24.84   | 35.48   |
| Fixed matter (%)       | 15.19   | 18.89   |
| Total carbon (%)       | 37.79   | 41.86   |

3.2 Adsorption Experiments

3.2.1 Effect of pH

Removal of Cr (VI) by BC-300 and BC-500 at different pH shown in Table 2. The result illustrated that as the pH decreases from 10 to 2, the removal percentage of Cr (VI) increases from 6.1 to 91.1% in BC-300 and 1.2 to 54% in BC-500. At lower pH, the surface of the biochar becomes positively charged due to availability of more H⁺ ions and therefore had a stronger attraction for negatively charged Cr (VI) ions in the solution. Hence, the adsorption increases with decrease in pH due to electrostatic attraction between H⁺ and Cr (VI) ions. At higher pH, the concentration of OH⁻ increases and overall charge on the biochar surface becomes negative, which cause hindrance in the adsorption of negatively charged Cr (VI) ions. This results in the decreased adsorption of Cr (VI) at higher pH [29]. Similar effect of pH on adsorption of Cr (VI) was previously confirmed by many research studies [30, 31].

### Table 2. Effect of pH on Cr (VI) adsorption by biochar (BC-300 & BC-500)

| pH | Removal % | Adsorption capacity (q_e), (mg/g) |
|----|-----------|----------------------------------|
|    |           |                                  |
| BC-300 |          |                                  |
| 2    | 91.18     | 45.59                            |
| 4    | 46.02     | 23.01                            |
| 6    | 6.86      | 3.43                             |
| 8    | 6.16      | 3.08                             |
| BC-500 |          |                                  |
| 2    | 54.06     | 27.03                            |
| 4    | 28.27     | 14.13                            |
| 6    | 17.28     | 8.64                             |
| 8    | 1.28      | 0.64                             |

3.2.2 Effect of adsorbent dose

The effect of adsorbent (biochar) dose on the chromium removal efficiency by biochar is illustrated in Table 3. In BC-300, with the increase in adsorbent dose from 0.4 g/l to 8 g/l, the removal capacity increases from 13 to 95.6%. In BC-500, the removal capacity increases from 7.7 at 0.4 g/l to 69.7% at 8 g/l. The Cr (VI) removal increases may be because of more availability of surface and pore volume at higher adsorbent doses. As the adsorbent dose increases, the availability of the potential binding sites also increases, leading to higher adsorption of Cr (VI).

3.2.3 Effect of initial chromium concentration

The effect of initial chromium concentration on the chromium removal efficiency by biochar is illustrated in Table 4. As the initial concentration increases, the removal capacity of BC-300 and BC-500 decreases. In BC-300, the removal percentage decreases from 99.7% at 50mg/l to
32.9% at 800 mg/l. In BC-500, the removal capacity decreases from 44.3% at 50 mg/l to 10.4% at 800mg/l. The Cr (VI) initial concentration in the solution effects the equilibrium uptake of Cr (VI). As the initial concentration increases, the removal of Cr (VI) decreases because the capacity of the adsorbent materials gets exhausted sharply with the increase in initial chromium concentration. The total available adsorption sites are restricted to a fixed adsorbent dose, which became saturated at a higher concentration leading to decreased removal capacity [12].

3.3 Adsorption Isotherm

The adsorption isotherm was evaluated using linear Langmuir and Freundlich model as given in equations 3 and 5. The Langmuir adsorption isotherm predicts the formation of an adsorbed solute monolayer, with no site interactions between the adsorbed ions. It also proposed interaction takes place by adsorption of one ion per binding site and the sorbent surface is homogeneous and contains only one type of binding site. Because, the Freundlich model does not predict surface saturation. It considers the existence of a multi-layered structure [32].

3.3.1 Langmuir Isotherm

The linear form of Langmuir isotherm is expressed in equation-3.

$$\frac{C_e}{q_e} = \frac{1}{Q_{max}b} + \frac{C_e}{Q_{max}}$$

Where, $q_e$ is the amount of solute adsorbed per unit weight of adsorbent (mg/g), $C_e$ is the equilibrium concentration of solute in the bulk solution (mg/L), $Q_{max}$ is the monolayer adsorption capacity (mg/g), and $b$ is a constant related to the free energy of adsorption ($b \propto e^{-\Delta G/RT}$). Its value is the reciprocal of the concentration at which half-saturation of the adsorption reached. The essential characteristic of a Langmuir isotherm can express in terms of a dimensionless constant separation factor ($R_L$) which is calculated by following equation-4 [32].

$$R_L = \frac{1}{1+bc_0}$$

Where, $b$ is the Langmuir constant; $C_0$ (mg/L) is the initial concentration; $q_e$ the amount of chromium adsorbed (mg/g) on the prepared adsorbents and $C_e$ the equilibrium concentration of chromium in solution.

3.3.2 Freundlich Isotherm

The linear form of Freundlich isotherm is given by equation as:

$$\log q_e = \log K_f + \frac{1}{n}\log C_e$$

Where, $q_e$ is the amount of solute adsorbed per unit weight of adsorbent (mg/g), $C_e$ is the equilibrium concentration of solute in the bulk solution (mg/l), $K_f$ is a constant indicative of the

### Table 3. Effect of adsorbent dose on Cr (VI) adsorption by Biochar

| Adsorbent dose (g/l) | Removal (%) | Adsorption capacity ($q_e$), mg/g |
|---------------------|-------------|-----------------------------------|
| BC-300              |             |                                   |
| 0.4                 | 13.52       | 33.82                             |
| 1.2                 | 41.33       | 34.44                             |
| 1.6                 | 61.59       | 38.49                             |
| 2                   | 77.04       | 38.52                             |
| 8                   | 95.63       | 11.95                             |
| BC-500              |             |                                   |
| 0.4                 | 7.73        | 19.34                             |
| 1.2                 | 26.31       | 21.88                             |
| 1.6                 | 35.94       | 22.46                             |
| 2                   | 44.40       | 22.20                             |
| 8                   | 69.70       | 8.71                              |

### Table 4. Effect of initial concentration on Cr (VI) adsorption by biochar

| Initial Concentration (mg/l) | Removal (%) | Adsorption capacity ($q_e$), mg/g |
|------------------------------|-------------|-----------------------------------|
| BC-300                       |             |                                   |
| 50                           | 99.71       | 24.92                             |
| 100                          | 70.22       | 35.11                             |
| 200                          | 51.60       | 51.60                             |
| 400                          | 26.13       | 52.26                             |
| 600                          | 29.04       | 87.12                             |
| 800                          | 32.98       | 131.94                            |
| BC-500                       |             |                                   |
| 50                           | 44.38       | 11.09                             |
| 100                          | 30.41       | 15.20                             |
| 200                          | 25.25       | 25.25                             |
| 400                          | 18.72       | 37.44                             |
| 600                          | 13.44       | 40.32                             |
| 800                          | 10.46       | 41.85                             |
relative adsorption capacity of the adsorbent (mg/g), and 1/n is a constant indicative of the intensity of the adsorption.

For the calculation of adsorption isotherms, $C_0$ and $q_e$ values were obtained from adsorption of hexavalent chromium at different initial concentrations with 2g/l adsorbent dose, pH-2, contact time-24 hours at room temperature. The plots of the Langmuir isotherm ($C_0/q_e$ versus $C_0$) and Freundlich isotherm ($\log C_e$ versus $\log q_e$) for BC-300 and BC-500 are shown in Fig. 1. The corresponding Freundlich and Langmuir parameters along with correlation coefficients are given in Table 5. It is clear from the result that the Langmuir model has better regression coefficients than Freundlich model. The monolayer adsorption capacity ($Q_{max}$) of BC-300 (114.94 mg/g) and BC-500 (49.75mg/g) for Cr (VI), was calculated from the linear Langmuir isotherm. In both BC-300 and BC-500, the value of $R$ was found to be greater than 0 and less than 1, indicating the favorable adsorption of Cr (VI) on banana peduncle biochar.

3.4 FE-SEM Analysis

Solid particle morphology and elemental composition can be described by FE-SEM, providing information about the structural variations in biochar particles after thermal treatment and presence of chromium on the biochar surface after adsorption. The biochar samples were employed for FE-SEM analysis after Cr (VI) adsorption with 100 mg/l concentration, adsorbent dose- 0.4 g/l, pH-2 and contact time-24 hours at room temperature. FE-SEM images of biochar (BC-300 and BC-500) before adsorption and elemental analysis after Cr (VI) adsorption showed in Fig. 2. These images were taken to compare the morphological changes in the pore structure of biochars after carbonization and to confirm the adsorption of chromium on its surface. A reduction in pore size of BC-500 in comparison to BC-300 and appearance of internal pores observed in biochars (BC-300 & BC-500) which may be due to the escape of volatiles during carbonization. The chromium peak observed in BC-300 and BC-500 after adsorption (BC-300 Cr & BC-500 Cr), confirms its adsorption on biochar.

3.5 FTIR Spectra

The biochar samples were employed for FTIR analysis after Cr (VI) adsorption with 100 mg/l concentration, adsorbent dose- 0.4 g/l, pH-2, contact time-24 hours at room temperature. The FTIR spectra (Fig. 3.) were interpreted by following the literature [33, 34]. The broad bands at 3268.05 (BC-300), 3308.30 (BC-300 Cr), 3198.86 cm$^{-1}$ (BC-500) and 3268.91 cm$^{-1}$ (BC-500 Cr) indicates the presence of H- bonded OH stretching of hydroxyl groups. The – CH$_2$ stretching of the aliphatic group are represented by bands at 2935.85 (BC-300), 2923.13 (BC-300C). The alkyne group (-C≡C stretching) can represent by bands at 2153.35 (BC-300) and 2179.63 cm$^{-1}$ (BC-300 Cr). The peak at 1595.51 (BC-300, BC-300Cr) and 1606.09 (BC-500, BC-500 Cr) can assigned to -C=C-C stretches of aromatic ring groups. The peaks observed in 1343.16 (BC-300), 1370 (BC-300Cr), 1378.32 (BC-500) and 1344.54 cm$^{-1}$ (BC-500Cr) could be assigned to hydroxyl (-OH) bending vibrations and C=O stretching of carboxylate ions. Peaks at 1118.21 (BC-300), 1091.93 (BC-300Cr) represents -CO stretching vibration of an alcoholic group and 1003.56 cm$^{-1}$ (BC-500) represents cyclohexane ring vibrations. The peaks at 832.25 (BC-500, BC-500Cr), 786.73 (BC-300) and 746.89 cm$^{-1}$ (BC-300Cr) specifies the presence of C-H out of the plane bending of an aromatic ring. The absorptions band between 3250 to 3650 cm$^{-1}$ and additional bands in the ranges 1600-1300, 1200-1000 and 800-600 cm$^{-1}$ confirmed that banana peduncle biochar contains unsaturated (contains C=C or aromatic) and hydrogen bonded hydroxyl compounds. The band between 850 and 670 cm$^{-1}$ and near 1600cm$^{-1}$ supports the presence of aromatic ring bands in biochar [33]. After adsorption of Cr (VI), changes in functional groups and band shifts were noticed in biochar. In BC-500, after adsorption the band shift from 3198.86 to 3268.91 and 1378.32 to 1344.54 cm$^{-1}$, whereas in BC-300 band shift from 3268.05 to 3308.30 and 1343.16 to 1370 cm$^{-1}$. Thus, the shift of the vibrational bands assigned to hydroxyl and carboxyl group indicates their involvement in the removal of Cr (VI) through the formation of surface complexes.

3.6 XPS Analysis

The biochar samples were employed for XPS analysis after Cr (VI) adsorption with 100 mg/l concentration, adsorbent dose-0.4g/l, pH-2 and contact time-24 hours at room temperature. X-ray Photoelectron spectroscopy was used to investigate the valence state of the chromium bound on banana peduncle biochar and presented in Fig. 4. In BC-300, significant bands observed at binding energies of 577.08 eV and
586.26 eV, which can be assigned to Cr 2p3/2 (Cr(OH)_3) and Cr 2p1/2 (CrO_3) respectively. In BC-500, significant bands of binding energies at 577.3 and 587.14 eV represents Cr 2p3/2 (Cr(OH)_3) and Cr2p3/2, sat (CrO_3) respectively [35]. Thus, the XPS spectra revealed that hexavalent chromium gets reduced to trivalent chromium on banana peduncle biochar.

Table 5. Adsorption isotherm parameters for Cr (VI) adsorption on BC-300 and BC-500

| Freundlich isotherm | Langmuir isotherm |
|---------------------|-------------------|
| K_f, m | R² | 1/n | Q_max, g/m² | b, L/g | R² | R_L |
| BC-300 | 28.767 | 6.21 | 0.694 | 0.161 | 114.94 | 0.00975 | 0.6978 | 0.50632 |
| BC-500 | 2.563 | 2.26 | 0.9725 | 0.4413 | 49.751 | 0.00785 | 0.9928 | 0.56022 |

Fig. 1. Langmuir and Freundlich isotherm plot for Cr (VI) adsorption on biochar (BC-300 and BC-500)

Fig. 2. FESEM image of BC-300 and BC-500 before adsorption of Cr (VI) and its elemental analysis after adsorption
4. CONCLUSION

India being largest producer of Banana in the world, produces huge amount of waste biomass like banana peduncle. The readily availability, high biomass and good biochar yield of banana peduncle justify its suitability for biochar production and cost-effective adsorbent for removal of Cr (VI). The present study highlights that removal efficiency of Cr (VI) by banana peduncle biochar mainly depends on the pH of the solution and initial concentration. The adsorption behavior of Cr (VI) on banana peduncle biochar followed both the Langmuir and Freundlich isotherm, but fitted well with Langmuir isotherm. The monolayer adsorption capacity for Cr (VI) was higher in biochar prepared at 300°C (114.9 mg/g) than 500°C (49.5 mg/g). The adsorption coupled reduction, and complexation of chromium with carboxyl and hydroxyl group of biochar in acidic medium are responsible for Cr (VI) removal. The present experimental results provide an insight to generate a low cost biochar from banana peduncle and support its utilization for better waste management.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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