

## Original Research

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# Removal of arsenic from contaminated water: Phytoaccumulation and adsorbent-based removal by activated carbon prepared from *Typha tripholia*

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## Abstract

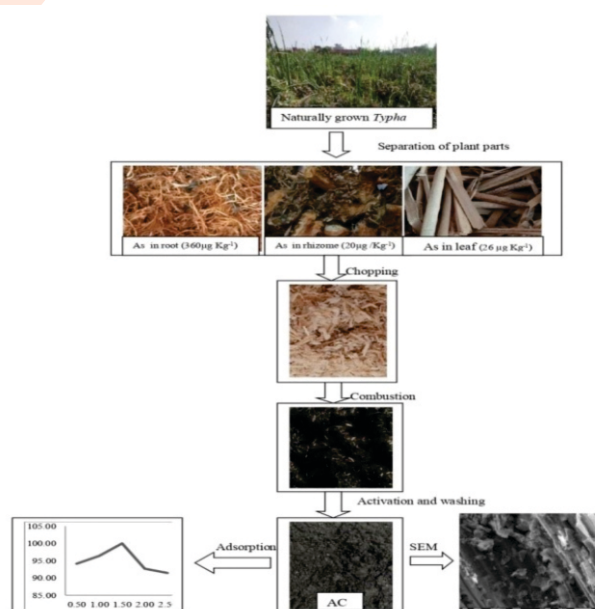
**Aim:** To assess the phytoaccumulation efficiency of arsenic in different parts of *Typha tripholia* (cattail) grown in the natural habitat of a pond located in Rada village of Bijua block, district Lakhimpur Kheri (U.P.) and to develop an adsorbent based removal technology by activated carbon prepared from *Typha tripholia* (ACTT) using physical activation.

**Methodology:** Phytoaccumulation of arsenic was studied in cattail plants. Rhizomes and leaves of plants were used to prepare activated carbon. The morphological characterization and surface structure were studied by using different modern techniques like Scanning electron microscopy (SEM), Energy-dispersive X-ray (EDX), and Brunauer-Emmett-Teller (BET). The adsorption efficiency and removal of arsenic were studied by using different doses of ACTT, i.e., 0.5, 1.0, 1.5, 2.0, 2.5 g 100 ml<sup>-1</sup> in the known solution of arsenate (30 ppb).

**Results:** The arsenic concentration in pond water was 19 µg l<sup>-1</sup>, which was higher than the permissible limit (10 µg l<sup>-1</sup>) of WHO, while in the soil it was 123 µg kg<sup>-1</sup>. The maximum As concentration in the root was 360 µg kg<sup>-1</sup>, while in the stem and leaf, it was 20 µg kg<sup>-1</sup> and 26 µg kg<sup>-1</sup>, respectively. Characteristics properties of ACTT showed amorphous nature and it contained 81.71 % carbon while other elements like oxygen (10.65 %), chloride (2.12 %) and potassium (5.52%) were also detected. The porosity of ACTT was 1.271 × 10<sup>-3</sup> cc g<sup>-1</sup> with pore volume 3.7 × 10<sup>-3</sup> cc g<sup>-1</sup>, while the pore size, pore width, and pore diameter were 4.498 nm, 2.0208 nm and 4.498 nm, respectively. Removal of arsenic increased on increasing the dose of activated carbon, and the maximum adsorption (99.6%) was observed at 1.5 g 100<sup>-1</sup> ml further it decreased.

**Interpretation:** Thus, *Typha tripholia* may be used as phytoremediation, a plant-based green technology for the removal of As from contaminated water while ACTT acts as a good adsorbent for As, due to its large surface area and pore space and a high degree of surface reactivity.

**Key words:** Arsenic removal, Activated carbon, Adsorption, Cattail, Phytoaccumulation



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## Introduction

Anthropogenic sources like mining, the use of arsenic-containing pesticides and herbicides as well as geogenic sources are responsible for arsenic (As) contamination in soil and water. Arsenic is an extremely toxic metalloid and causes major health issues in human beings. It is one of the toxic and carcinogenic compounds which pose a high risk to large human populations even at low levels (Mandal and Suzuki, 2002). Wetlands are susceptible to As pollution from wastewater and residue discharges from industry and agriculture, making the sediments into As sinks (Bonanno and Cirelli, 2017). It is more dangerous in wetlands than on land because it may move more easily and enter food chains (Zhao et al., 2010). So, the removal of As from aquatic ecosystems is an ecological problem that requires investigation and remediation. Phytoremediation is a plant-based technology to remove toxic chemicals and elements from contaminated water due to the accumulation, translocation, and restriction in different plant parts. Many aquatic plants, such as *Typha angustifolia* L., *Canna glauca* L., *Colocasia esculenta* L. Schott, and *Cyperus papyrus* L., are frequently found in wetland ponds. Some of these plants show the potential to remove contaminants from water through phytoremediation.

*C. esculenta* is a wetland herbaceous perennial plant used in the manmade wetland which removes xenobiotics from polluted water tanks (Cheng et al., 2002). *Typha* is an aquatic or semi-aquatic perennial plant distributed in the Northern Hemisphere that is widely used for water treatment, and treatment of soil contamination (Hansen et al., 1998; Bankston et al., 2002) due to its nature to grow in marshy land. Several common processes like filtration, electrolysis, and adsorption as well as biological methods used to control water pollution. Among these, adsorption is one of the most effective methods to remove heavy metals using a suitable adsorbent (Singh et al., 2017). Although, activated carbon is a quite expensive adsorbent depending on its high-cost sources such as wood, coal, and coconut shell (Attia et al., 2008) but cheap sources of plant parts for preparing activated carbon can minimize the cost of preparation. Singh et al. (2017) used low-cost adsorbent for preparing activated for the removal of cadmium from aqueous solutions using water hyacinth (*Eichhornia crassipes*).

Due to the development of dense floating colonies, which result in a reduction in the amount of light and dissolved oxygen, its strong reproductive and adaptive ability harms native flora and wildlife (Gao and Li, 2004; Mahamadi, 2011). This macrophyte is being researched as a means of effectively cleansing polluted effluents. The networks of lignin and carbohydrates that make up its stems and leaves provide active spots on the material's porous surface that have a high affinity for capturing poisonous metals like arsenic (Sanmuga Priya and Selvan, 2017). Several studies have also focused on the preparation of activated carbon from coir pith, almond shells, olive and peach stones, oil palm stones, and plum kernels (Wu et al., 1999; Lua and Guo, 2000; Kadirvelu et al., 2001) for the removal of different heavy metals, however,

there is a scarcity of literature concerned with the removal of As by AC prepared from *Typha tripholia* (ACTT) plants. Activated carbons are extensively utilized adsorbent due to their high adsorption capacity, high surface area and microporous structure. Carbon is extremely valuable due to its large specific surface area and high chemical and mechanical stability. The sorption activity is typically determined by the chemical composition and pore structure (Arivoli et al., 2008). Thus, an integrated approach is for required selecting plants which may be used for phytoremediation and to develop good adsorbent from such plants as cheap and suitable adsorbent sources e.g., activated carbon prepared by *Typha tripholia* (ACTT). Hence, this study was divided into two phases: the first to assess the phytoaccumulation of As by *Typha tripholia* grown in natural habitats of ponds located in Rada village of Bijua block, district Lakhimpur Kheri (U.P.), India, and second to develop a adsorbent-based removal technology of As by ACTT from contaminated water.

## Materials and Methods

**Experimental procedure:** The cattail (*Typha tripholia*) plant was used to assess the phytoaccumulation of arsenic in different plant parts. The plant was uprooted from a natural habitat of a pond located in Rada village of Bijua block, district Lakhimpur Kheri (U.P.), India. The plant was grown in arsenic-contaminated water and soil (Fig. 1). Soil, water, and plant samples were collected to analyze them for assessing arsenic contamination. The intact plant with root was washed two to three times thoroughly with tap water to remove dust from leaves and mud from the root. Thereafter, plant parts i.e., root, rhizome, and leaves were thoroughly separated. After washing with glass-distilled water, plant parts were packed in leveled paper envelopes and dried in an oven at 70°C for 24h. Arsenic concentration was assessed after wet digestion ( $\text{HNO}_3$ :  $\text{HClO}_4$  in 10:1 v/v mixture) of the oven-dried plant material (Piper, 1967). Arsenic content was determined with atomic absorption spectrophotometer (Perkin Elmer Analyst, 300).

**Preparation of activated carbon:** Plants were separated into roots, rhizomes, and leaves. Each plant part was chopped into small pieces ( $\pm 0.5$  cm length) and dried under the sun for 5 days. Different pieces were washed with glass distilled water several times and dried in an oven at 110°C overnight. These dried plant materials were packed in a sealed container for experimentation. In different batches, ACTT was prepared in the absence of oxygen. Sealed plant materials were kept in a muffle furnace at 250°C for 4hr. Materials were then carbonized by adjusting the furnace temperature at desired activation temperature (500°C) for 45 min. After carbonization, the ACTT was refluxed with distilled water for 3 hr which was repeated several times until it achieved a constant pH. For physical activation, ACTT was refluxed with 0.1 M  $\text{HNO}_3$  to remove heavy metal ions, tar, and ash, followed by the second step refluxed with distilled water several times to remove the acid. The ACTT was dried in an oven for 4 hr at 105°C. Dried samples were sieved to pass through a 100-mesh sieve and stored in desiccators for further use.

**Morphological characterization of ACTT:** For characterization of elemental composition of ACTT, Energy-dispersive X-ray (EDX) 6510, LA, at 20.00 kV was used. Scanning electron microscopy (SEM) images were obtained with a field emission scanning electron microscope (Carl Zeiss NTS GmbH, Oberkochen (Germany) Model: SUPRA 40 V P operated at an accelerating voltage of 10 kV. Characteristics properties of ACTT were studied by using XRD. For Brunauer-Emmett-Teller (BET), Quanta chrome Autosorb 1C was used to analyze the surface area and the pore size of AC. Atomic absorption spectrophotometer (Perkin Elmer Analyst 300) was used for estimating arsenic concentration in the solution after removal.

**Adsorption experiment:** To assess the arsenic removal efficiency by ACTT from contaminated water, a known solution of arsenic ( $30 \mu\text{g l}^{-1}$ ) was prepared by using an arsenate stock solution of  $1000 \text{ mg l}^{-1}$  ( $\text{As}_2\text{O}_5$ ) in glass distilled water. Different doses of activated carbon, i.e., 0.5, 1.0, 1.5, 2.0, and  $2.5 \text{ g } 100^{-1} \text{ ml}$  were used in the known solution of arsenic ( $30 \mu\text{g l}^{-1}$ ) and shaken at constant speed at 60 rpm for 180 minutes. The equilibrium data were obtained by As (V) onto prepared ACTT. Thereafter, the performance of the best dose of activated carbon was selected based on the maximum removal percentage. A control sample was taken before getting in contact with the activated carbon to determine the exact concentration of adsorbate. The filtrates were used to analyze As concentration by AAS after filtration using Whatman no. 42 filter paper.

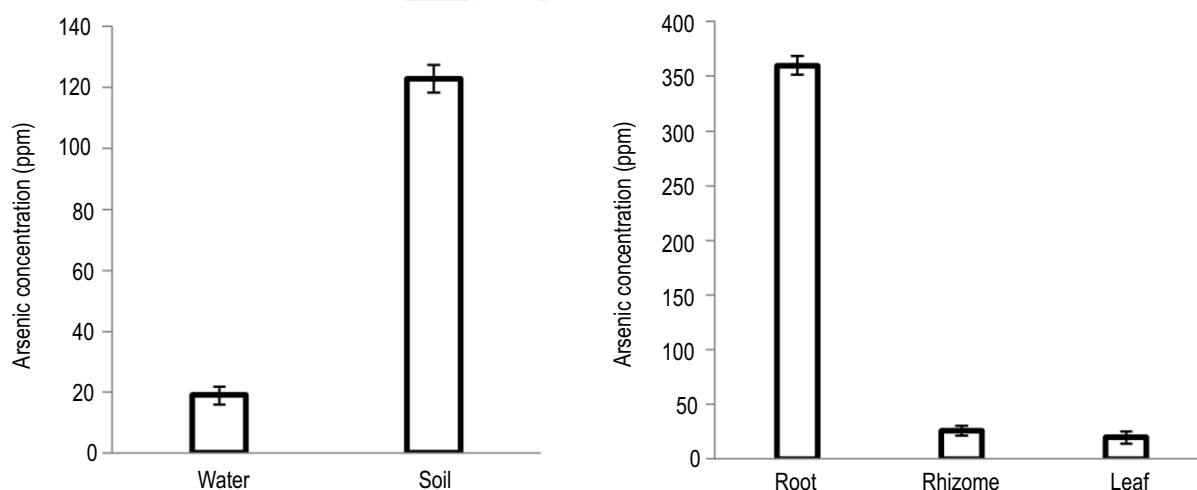
**Determination of arsenic concentration:** The final concentration of arsenate (As [V]) was determined using AAS. The percentage removal and the amount of arsenate adsorbed onto ACTT were calculated by difference of initial and final concentration (Anisuzzaman et al., 2015).

**Statistical analysis:** The results were analyzed using Sigma stat

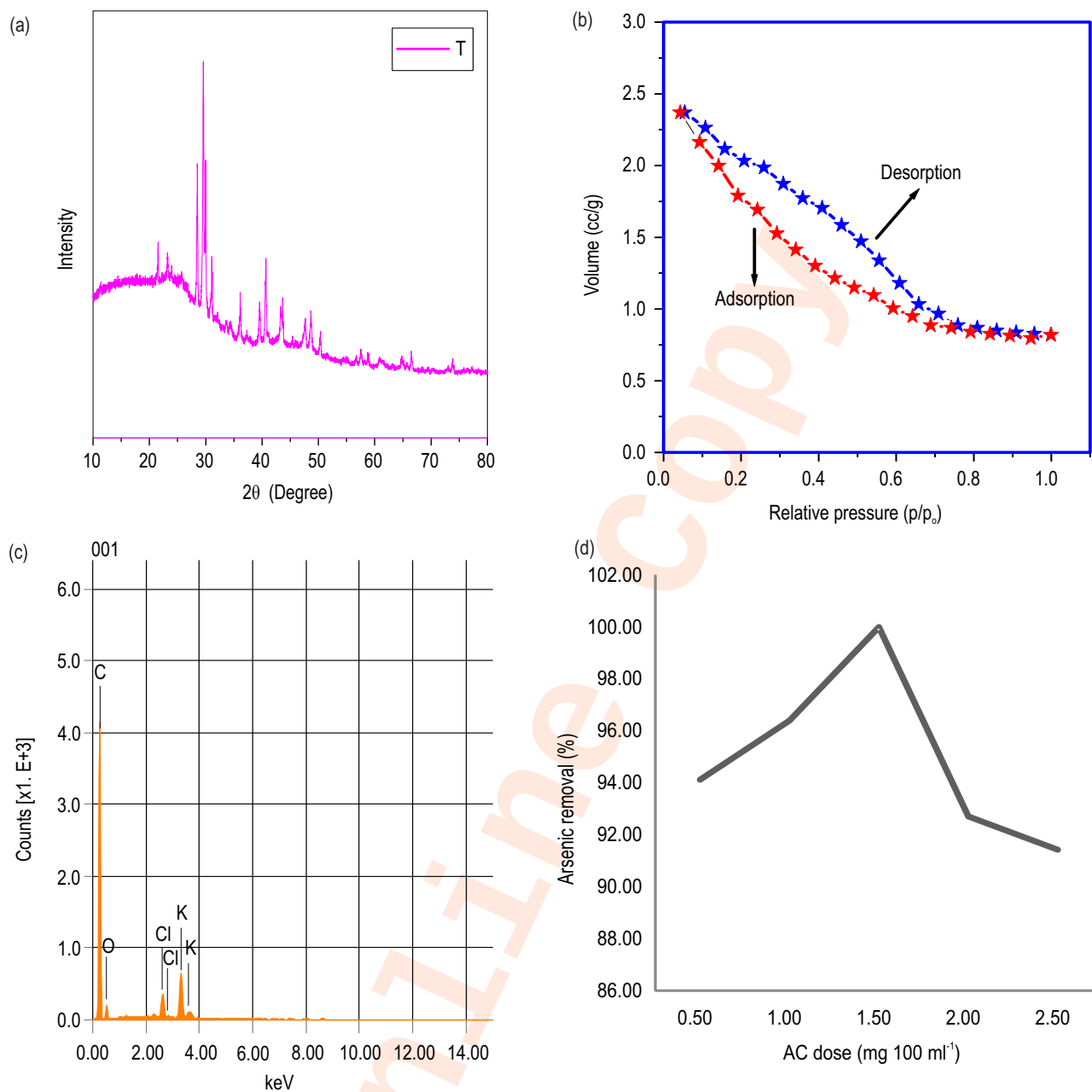
4 software. As a comparative analysis, the unpaired 't'-test was performed. The probability value was found less than 0.05 ( $P < 0.0001$ ) and was considered statistically significant.

## Results and Discussion

Phytoaccumulation was studied on a naturally growing cattail plant (*Typha tripholia*) in a pond located at Rada village, Bijua block, district Lakhimpur Kheri (U.P.), India. Results indicated that arsenic concentration in pond water was  $19 \mu\text{g l}^{-1}$ , which was higher than the recommended limit ( $10 \mu\text{g l}^{-1}$ ) as per WHO guidelines while in the soil, the As content was  $123 \mu\text{g Kg}^{-1}$ , which showed that pond water and soil were contaminated with arsenic. The main reason for As contamination of pond water may be due to surface runoff in agricultural fields with pesticides and insecticides used by farmers collected in the pond. Although, As [v] and As [III] are bioavailable for aquatic plants while As [V] are dominant species in polluted water (Sizova et al., 2002). The dynamics of As[V] exchange between water and adsorbing colloids are analogous to the phosphate so there is a competition for exchange sites among phosphate and As [V] which replace phosphate and enter the plants (Mkandawire et al., 2004a). The concentration of arsenic varied in different plant parts. The maximum accumulation was observed in the roots ( $360 \mu\text{g kg}^{-1}$ ) while in the rhizome and leaf, it was  $20 \mu\text{g kg}^{-1}$  and  $26 \mu\text{g kg}^{-1}$ , respectively (Fig. 1 a,b) which showed that high accumulation of arsenic concentration in the roots further, restrict to transport in rhizome and leaf. Thus, *Typha* could be suitable for the remediation of arsenic-contaminated aquatic systems. It has been widely documented that cattail plants, which are wetland macrophytes that have huge biomass, high cellulosic and lignolytic materials, rapid growth, and strong adaptability, and an ideal candidate for phytoremediation (Bonanno and Cirelli, 2017). As a rhizomatous helophyte, *Typha angustifolia*, tolerated sediment As concentrations up to  $11120 \text{ mg kg}^{-1}$  (Bonanno et al., 2017) and accumulated As upto  $16017 \text{ mg kg}^{-1}$  in roots and  $1028 \text{ mg kg}^{-1}$  in



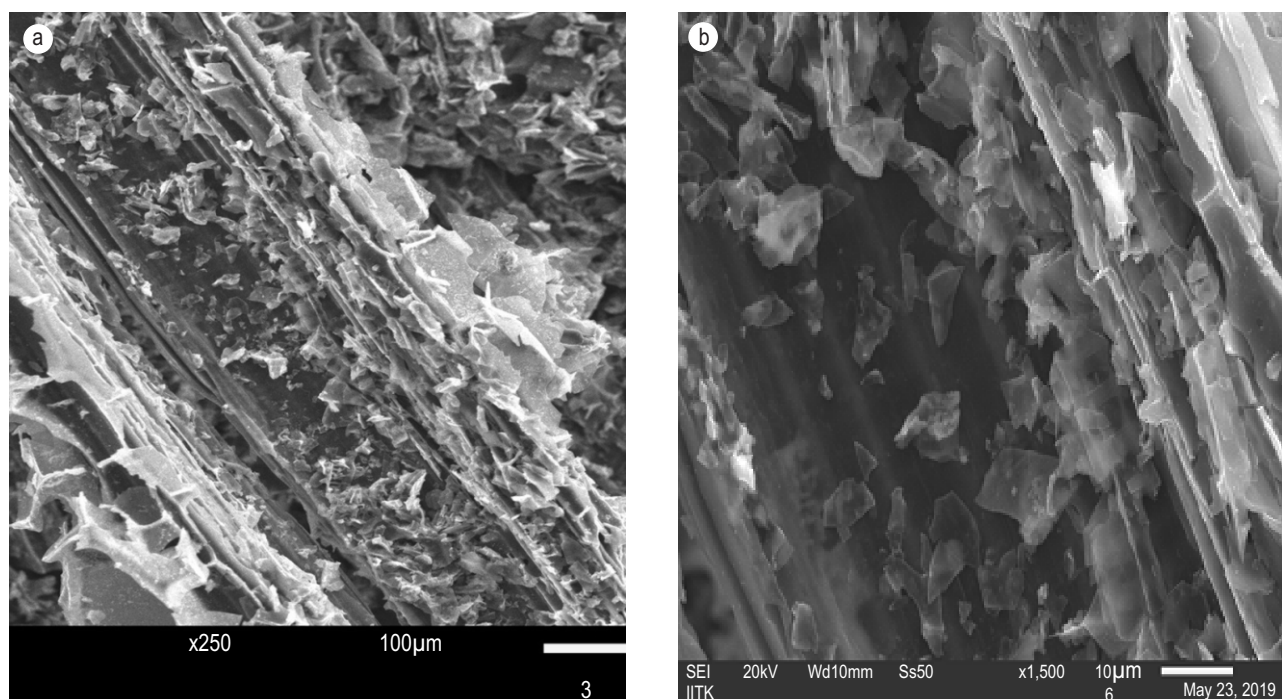
**Fig. 1:** Phytoaccumulation of arsenic by *Typha tripholia* growing naturally in contaminated water and soil in a pond of Rada village in Bijua block of district Lakhimpur Kheri (U.P.), India.  $\pm$ SEM value, there is a statistically significant difference ( $P < 0.001$ )



**Fig. 2:** Morphological characterization of ACTT (a) X-ray diffraction pattern, (b) isotherm, (c) EDXRD showed the elemental composition and (d) Removal of arsenic by ACTT.

leaves, with an average level of  $4273 \text{ mg kg}^{-1}$  in plant tissues (Brankovi *et al.*, 2015). Other workers who have reported accumulating arsenic from water through other aquatic macrophytes (Robinson *et al.*, 2003; Mkandawire *et al.*, 2004b; Mkandawire and Dudel, 2005; Robinson *et al.*, 2006; Rahman *et al.*, 2007; Alvarado *et al.*, 2008). The characteristic properties of ACTT were studied with XRD, carried out by the Rigaku Ultima-IV X-ray diffractometer is shown in Fig. (3a). The XRD pattern showed broad diffuse scattering at  $10^\circ$ - $28^\circ$ , which confirmed the short-range order

characteristics of the amorphous nature of ACTT and few crystalline peaks were also observed. The Energy-dispersive X-ray diffraction was used to assess the elemental composition of ACTT. It contained 81.71% carbon while other elements like oxygen (10.65%), chloride (2.12%), and K (5.52%) were also found (Table 1, Fig. 2c). Textural characterization was done through scanning electron microscopy (SEM) and Brunauer-Emmett-Teller analyzer. Surface area and pore volumes (or pore size distribution) were determined using the Brunauer-Emmet-



**Fig. 3:** Surface morphology of ACTT through scanning electron microscopy at two magnifications (a) Magnification x 250 pixels and (b) high magnification x1500 pixels.

Teller (BET) equation. Based on BET results, a graph was plotted between the volume of nitrogen gas and its relative pressure (Fig. 2b). In order to characterize the surface morphology of adsorbents, scanning electron microscopy, was conducted to characterize the activated carbon (Aljeboree *et al.*, 2017).

SEM showed that the surface area and the pore size increased on increasing the dose of ACTT. The large surface area and the large pore size of ACTT were recommended for As removal from the contaminated water (Fig. 3). Brunauer-Emmett-Teller analysis indicated ACTT porosity to be  $1.271 \times 10^{-3} \text{ cc g}^{-1}$  with pore volume  $3.7 \times 10^{-3} \text{ cc g}^{-1}$  while pore size, pore width, and pore diameter were 4.498 nm, 2.0208 nm, and 4.498 nm, respectively. Micro-pore volume and micro-pore areas were  $2.528 \times 10^{-3} \text{ cc g}^{-1}$  and  $2.253 \text{ m}^2 \text{ g}^{-1}$ , respectively (Table 2). Brunauer-Emmett-Teller analysis also indicated that cumulative adsorption and desorption surface area was 2.716 and  $2.943 \text{ m}^2 \text{ g}^{-1}$  while adsorption and desorption pore volume was  $3.546 \times 10^{-3} \text{ cc g}^{-1}$  and  $1.47 \times 10^{-3} \text{ cc g}^{-1}$  respectively. The volume of the micropores was substantially greater than that of the mesopores, indicating that the micropores are involved in the kinetics of adsorption (Table 2).

Activated carbons are extensively used adsorbent due to high adsorption capacity, high surface area, microporous structure, and a high degree of surface. Carbon is extremely valuable due to its large specific surface area and great chemical and mechanical stability. The sorption activity is typically determined by the chemical composition and pore structure

(Arivoli *et al.*, 2008). Precursor sources and the manufacturing process were the main factors in the development of pore system in ACTT. The amount of pores mainly depends on the concentration of impregnation agent (Jankowska *et al.*, 1991). The surface structures of ACTT showed cleaner and burnout pores with tunnel structures. The ACTT obtained from the physical activation process showed the pore amount, which depends on the washing process and played a crucial role in the development of pores as most of the chemical activator remains in the particle are eliminated during intense washing after being activated at high temperatures (Anisuzzaman *et al.*, 2015).

Different doses of ACTT were used to assess the removal of As [V] from contaminated water. Results indicated that the percent removal of As [V] was 94.13, 96.40, 99.6, 92.71 and 91.3%, respectively (Fig. 3d). The maximum removal (99.6%) was observed at  $1.5 \text{ g } 100 \text{ ml}^{-1}$  dose of ACTT and further, it decreased on increasing the dose while the minimum removal was noted at the maximum dose ( $2.5 \text{ mg } 100 \text{ ml}^{-1}$ ). It implies that the initial concentration of chromium ions has a significant impact on the adsorption, because the initial number of chromium ions to the accessible surface area is minimal at lower concentrations, the fractional adsorption doesn't depend on the starting concentration. The percentage of chromium ion removal is dependent on the initial concentration since there are fewer accessible sites for adsorption at high concentrations (Arivoli *et al.*, 2008). Budinova *et al.* (2009) used bean pods to make activated carbon by conventional physical activation (water

**Table1:** Elemental composition of activated carbon prepared from cattail (*Typha tripholia*) plant

| Chemical formula | Mass (%) | Atom (%) | Sigma | Net   | K ratio   | Line |
|------------------|----------|----------|-------|-------|-----------|------|
| Carbon ©         | 81.71    | 88.70    | 0.05  | 98300 | 0.0306341 | K    |
| Oxygen(O)        | 10.65    | 8.68     | 0.11  | 4876  | 0.0051603 | K    |
| Chlorine (Cl)    | 2.12     | 0.78     | 0.02  | 15167 | 0.0076167 | K    |
| Potassium (K)    | 5.52     | 1.84     | 0.05  | 33224 | 0.0195482 | K    |
| Total            | 100      | 100      |       |       |           |      |

**Table 2:** Morphological properties, i.e., surface area, pore area, pore volume, pore diameter, atom diameter, and adsorbent density of activated carbon prepared from cattail plant (*Typha tripholia*)

| Morphological properties                                  | Description | Langmuir | Multipoint Brunauer-Emmett-Teller (BET) | Mesopore Barrett-Joyner-Halenda (BJH Method) | Mesopore (NLDFT-Method) | MesoporeDH (Dollimore-Heal) Method | Micropore t-Method | Micropore DR Method |
|-----------------------------------------------------------|-------------|----------|-----------------------------------------|----------------------------------------------|-------------------------|------------------------------------|--------------------|---------------------|
| Cumulative surface area (m <sup>2</sup> g <sup>-1</sup> ) | Adsorption  | 1.744    | 1.130                                   | 2.130                                        | 1.564                   | 2.716                              | 2.253              | -                   |
| Pore area (m <sup>2</sup> g <sup>-1</sup> )               | Desorption  |          |                                         | 0.4862                                       |                         | 2.943                              |                    |                     |
| Pore volume (cm <sup>3</sup> g <sup>-1</sup> )            | Adsorption  | -        | -                                       | 3.318x10-3                                   | 3.695x10-3              |                                    | 2.528x10-3         | 1.080x10-3          |
|                                                           | Desorption  |          |                                         | 3.069x10-4                                   |                         |                                    |                    |                     |
| Cumulative pore volume (cm <sup>3</sup> g <sup>-1</sup> ) | Adsorption  | -        | -                                       | 3.314x10-3                                   | -                       | 3.546x10-3                         | -                  | -                   |
|                                                           | Desorption  |          |                                         | 2.810x10-4                                   |                         | 1.470x10-3                         |                    |                     |
| Pore diameter (nm)                                        | Adsorption  | -        | -                                       | 4.394                                        | 2.021                   | 4.394                              | -                  | -                   |
|                                                           | Desorption  |          |                                         | 1.654                                        |                         | 1.654                              |                    |                     |
| Adsorbent atom diameter (nm)                              | 0.3400      |          |                                         |                                              |                         |                                    |                    |                     |
| Adsorbent density (g cm <sup>-3</sup> )                   | 2.2460      |          |                                         |                                              |                         |                                    |                    |                     |

vapor) and find out the maximum adsorption capacity at 1.01 mg g<sup>-1</sup> for As [III]. Anisuzzaman *et al.* (2015) also reported up to 97.2% removal of Cd(II) by activated carbon prepared from cattail. Activated carbon should have variable surface chemistry for the removal of organo-metallic contaminants like large surface area as well as a high degree of surface reactivity which are further dependent on the source material, conditions of carbonization and activation and impregnation. According to Dambies (2004), the process and the optimum conditions are still not clear for the tendency to adsorb arsenic onto activated carbon.

Some researchers have reported that the activated carbon can remove both forms of arsenic species (Samsuri *et al.*, 2013). Many adsorbents are made up of agriculture by-products (Budanova *et al.*, 2009; Lodeiro *et al.*, 2013; Tuna *et al.*, 2013). The adsorption activity of AC may be enhanced by modifying through impregnation with metal or metal oxides. Liu *et al.* (2010) used the

waste biomass to form a low-cost composite Fe<sub>3</sub>O<sub>4</sub> loaded activated carbon for the removal of arsenate from water. The study of adsorption kinetics is important to evaluate the mechanism and efficiency of the adsorption process. The kinetics of As [III] by prepared AC was analyzed using pseudo-first-order and pseudo-second-order kinetic models in which contact times were the rate-determining step (Ho and McKay, 1998). There is the involvement of a sharing of electrons between the adsorbate and the surface of the adsorbent in the chemisorptions process (El Qada *et al.*, 2006). Similar adsorption kinetic results have also been obtained from the biosorption of Pb (II) by cattail leaves and tea waste (Liew *et al.*, 2011; Amarasinghe and Williams, 2007). The equilibrium was achieved when the capacity of the adsorbent is saturated, thus the rate of adsorption equals the rate of desorption (Payne and Abdel-Fattah, 2004). The concentration of adsorbate remaining in the wastewater at the equilibrium depends on the type of activated carbon and the

dose/concentration of adsorbent (Krishnaiah et al., 2013). It is also used for removing phosphorous and some heavy metals (Ciria et al., 2005). Thus, *Typha tripholia* may be used as phytoremediation for the removal of As from contaminated water as well as ACTT acts as a good adsorbent for arsenic due to its large surface area and pore space and a high degree of surface reactivity.

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**Authors' contribution:** A. Gupta: Analytical work and Manuscript writing; V. Kumar: Activated carbon preparation and activation; P. Singh: Characterization of AC; SG Pratap: Help in MS writing; P. K. Singh: Overall monitoring and writing of the manuscript.

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