



Potential of activated carbon from waste rubber tire for the adsorption of phenolics: Effect of pre-treatment conditions



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ABSTRACT

Rubber tire activated carbon modification (RTACMC) and rubber tire activated carbon (RTAC) were prepared from waste rubber tire by microwave assisted chemical treatment and physical heating respectively. A greater improvement in porosity and total pore volume was achieved in RTACMC as compared to that of RTAC. But both have a predominantly mesoporous structure. Under identical operating conditions, an irradiation time of 10 min, chemical impregnation ratio of 1.50 and a microwave power of 600 W resulted in maximizing the efficiency of RTACMC for p-cresol (250 mg/g) at a contact time of 90 min while RTAC showed a 71.43 mg/g adsorption capacity at 150 min. Phenol, due to its higher solubility was adsorbed to a lesser extent by both adsorbents. Physical nature of interactions, pore diffusion mechanism and exothermicity of the adsorption process was operative in both adsorbents. The outcomes support the feasibility of preparing high quality activated carbon from waste rubber tire by microwave assisted chemical activation.

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1. Introduction

The excellent properties of activated carbons (AC) as adsorbent materials have rendered the adsorption technology a versatile technique for wastewater remediation [1]. Some of these properties which have been widely documented are its large interparticulate surface area, controllable pore structure, thermostability, low acid/base reactivity and highly modifiable surface chemistry [2]. But the high cost associated with the commercial ACs has led to the widespread use of various low cost precursors like biobased agricultural residues, biomass etc. for the production of ACs [3]. Besides their high relative abundance and easy processing costs, such precursors have been identified to have high carbon content, are insoluble in nature, have good chemical stability, high mechanical strength and also have a favorable surface chemistry. Such properties have facilitated their adsorption properties.

Carbon black from waste rubber tire has been another interesting alternative as a low cost adsorbent basically because its carbonaceous nature resembles that of a commercial AC [4,5]. Also since it constitutes approximately 32% by weight of carbon, its recovery and reuse as adsorbent would solve the waste tire disposal problem thereby benefiting the environment in a dual way. But the carbon black obtained by untreated tire pyrolysis has a less developed porous structure and a lower internal surface area [6]. Appropriate activation conditions of temperature, time

etc. have been found to enhance both surface area and pore volume of activated carbons developed from carbon black [7–18] and this has resulted in improved adsorption behavior. Mui et al. in their review on preparation and characterization of activated carbon from carbon black have documented the use of physical and chemical methods of activation of carbon black from rubber tire [19].

Recent progress has shown the use of microwave assisted physical and chemical methods in the preparation of activated carbon from various low cost precursors as a substitute to conventional heating [20]. The advantage associated with using microwave heating is that the treatment time and consumption of gases can be considerably reduced, which further results in the reduction in energy consumption as well. In comparison with conventional heating techniques, microwave heating has been seen to demonstrate interior heating, higher heating rates, selective heating, greater control of the heating process, no direct contact between the heating source and heated materials, and reduced equipment size and waste. Additionally, microwave furnaces are generally smaller than conventional furnaces. Also, the activated carbons prepared by microwave heating have been seen to demonstrate lesser oxygenated functionalities from their carbon surfaces [21]. The textural and chemical properties of the carbons thus prepared have been found to be comparable to those from conventional heating but the time of preparation of the activated carbon is seen to be far shortened [22].

Based on a vast literature review on the use of microwave technology for the preparation and regeneration of activated carbons, it

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is concluded that till date this technology has not been used for the preparation of activated carbons from rubber tire. The objective of the present study is focused in preparing activated carbons (RTACMC and RTAC) by microwave assisted chemical treatment as well as by conventional physical activation respectively from waste rubber tire followed by the assessment of their physical and chemical characteristics for the ultimate objective of determining their adsorption capacity for organic pollutants like p-cresol and phenol. Experimental data were fitted to various adsorption isotherms and kinetic models in order to determine the adsorption capacities and rates.

Phenolic compounds were selected as model adsorbates because of their widespread prevalence in industrial wastewater as a result of their rampant use in many chemical and petrochemical industries, oil refinery, ceramic and steel plants, disinfectant manufacturing, or metal refining [23]. Their concentration in some of the industrial wastewaters in India has been found to be very high varying from 1000 to 2000 ppm in coal mining, 50–700 ppm in petrochemical, 1000 ppm in pharmaceutical and 2000–20,000 ppm in oil refining industries [24]. Also, such phenolic compounds are known to have high toxicity and carcinogenicity. Because of their stability and bioaccumulation, they remain in the environment for longer periods and hence cause considerable damage and threat to the eco-system in water bodies and human health [25]. They are considered toxic for some aquatic life forms in concentrations above 50 ppb and the ingestion of one gram of phenol has shown fatal consequences in humans. Phenols have the capacity to combine with existing chlorine in drinking water, giving rise to chlorophenols, compounds that are even more toxic and difficult to eliminate. Hence phenolic compounds have been included in the list of priority pollutants by the Environmental Protection Agency (EPA) and the European Environmental Agency (EEA) [26–28].

2. Materials and methods

2.1. Reagents

Technical grade p-cresol and phenol of 98% purity were obtained from M/s Merck, India. Stock solution and all working solutions were prepared with deionized water. All reagents used in the study were of analytical grade. The ground tire granules used in the study were purchased from Wiswani Chemicals. Deionized water was used for making synthetic samples.

2.2. Adsorbent development

The ground tire granules were cleaned, thoroughly washed with deionized water, and then dried in an oven at 100 °C for 2 h. Carbonization of the ground tire granules was done as per our previous study [29] in which the dried material was heated to 500 °C for 5 h. This was followed by treatment with hydrogen peroxide solution for 24 h at 60 °C to oxidize adhering organic impurities. The material was washed with deionized water for three times to remove hydrogen peroxide and dried at 110 °C for 2 h in vacuum oven. The dried material was activated to 900 °C for 2 h in a covered silica crucible by heating in a muffle furnace. The crucibles were removed from the furnace and cooled in a desiccator. The material was then treated with 1 M HCl solution to remove the ash content and was further washed with deionized water. This was followed by drying of the material at 100 °C for 24 h. The dried product of particle size 150–200 μm is referred to as RTAC throughout the study and stored in separate vacuum desiccators until required.

The char obtained after carbonization was mixed with potassium hydroxide (KOH) pellets with varied KOH/char impregnation ratio of 0.5, 1, 1.5 and 2.

The impregnation ratio = $\text{weight(KOH)(g)}/\text{weight(char)(g)}$

The activation step was performed in a glass reactor placed inside a microwave oven of input power set at 200, 400, 600 and 800 W and an irradiation time of 5, 10, 15 min. After several trials, the process conditions of microwave cum chemical impregnation activation were optimized. A maximum 94% p-cresol removal was demonstrated by the resultant AC viz. RTACMC which was achieved under optimum conditions of chemical impregnation ratio of KOH/char of 1.50, microwave power of 600 W and an irradiation time of 10 min. This sample was washed with deionized water until the pH of the solution was neutral and stored for further characterization and adsorption tests.

2.3. Characterization of the adsorbent

pH measurements were made using a pH meter (Model Cyberscan 510, Singapore). The concentration of p-cresol was determined spectrophotometrically on a UV-visible spectrophotometer (Model 2450, Shimadzu). LEO 435 VP (Leo Elektronenmikroskopie GmbH, Germany) scanning electron microscopy was used for scanning the adsorbent surface. Carbon, hydrogen, and nitrogen analyses of the adsorbent were carried out on an Elementar Vario ELHI CHNS analyzer. For the FTIR study, 0.1 g of finely sized particle of the adsorbent was encapsulated in 1 g of KBr keeping the ratio 1:10, in order to prepare the translucent sample disks. The textural characteristics of the adsorbents were determined by N₂ gas adsorption isotherm at 77 K on micromeritics ASAP 2010 (UK), mercury porosimetry (Pascal 440; M/s Spektron Instrument Inc., India), and density measurements by Automatic Gas pycnometer (Ultrapyc 1200e, Quantachrome Instruments, India). The mesopore and the micropore volume of the adsorbents were determined by the mercury porosimeter plots of total pore volume vs. pore radius and the reading obtained by the volume adsorbed at $p/p_0 = 0.10$ from the N₂ adsorption isotherm plots respectively. The total pore volume was obtained from the density measurement of mercury (Mercury porosimeter) and density of helium (Automatic Gas pycnometer).

2.4. Adsorption and kinetic studies

The batch kinetic and isothermal adsorption experiments were conducted in a series of 250 mL Erlenmeyer flasks filled with an equal amount (0.6 g/L) of RTAC and RTACMC along with 100 mL of p-cresol and phenol solution of varying concentrations (3–300 ppm) maintained at the desired temperature and pH. The pH of the solution was kept constant by adding 0.1 M NaOH or 0.1 M HNO₃. The parameters considered for study were pH (2–12), initial concentration of adsorbate (3–300 ppm), contact time (5–210 min) and temperature (25–45 °C). The flasks were agitated in an orbital shaker at 100 rpm and liquid samples (10 mL) were taken out at a given time interval after centrifugation at 1500 rpm for 10 min on a centrifuge. The filtrate was collected and diluted before analysis. The concentration of remaining phenolic solution in the adsorption medium was determined using UV-Visible spectrophotometer at 277 and 269.9 nm for p-cresol and phenol respectively. The adsorption capacities were then obtained by mass balance calculations. The p-cresol and phenol uptake, q_e (mg/g), were determined as follows:

$$q_e = (C_0 - C)V/W \quad (1)$$

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