



Removal of phenolics from aqueous media using quaternised maize tassels



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ABSTRACT

This paper reports on the preparation and modification of powdered maize tassels with polydiallyldimethylammonium chloride (polyDADMAC). The modified tassel were applied for the removal of phenolic compounds from water, through adsorption. The effect of contact time, sorbent dose, pH of the sample and the adsorption capacity were investigated at fixed temperature (25 °C). The optimum pH was 6.0 and the uptake was more than 90% within the first 10 min of contact. The adsorption prescribed to Langmuir model of monolayer adsorption implying a chemisorption process. The adsorption capacities were found to be 7.09, 8.23, 8.84 and 4.74 mg g⁻¹ for chlorobenzoic acid, 2,4,6-trichlorophenol, 2,4-dichlorophenol and 1,2-dihydroxybenzene respectively. These were fairly higher than many other reported systems. The removal efficiency was found to be 75, 64, 55 and 40% for Chlorobenzoic acid, 2,4,6-Trichlorophenol, 2,4-dichlorophenol and 1,2-dihydroxybenzene, respectively. This proved that quaternised maize tassels can be used as an efficient adsorbent material for removal of phenolic compounds from water and wastewater.

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1. Introduction

Organic compounds containing hydroxy groups directly attached to the structure of single or condensed aromatic rings are classified as phenols. They are similar to alcohols but tend to ionise easily and display a more acidic character than alcohols. This is due to the presence of a delocalised electron cloud that cause stabilization in the ring by withdrawing electrons from substituted groups thus weakening the bond between oxygen and hydrogen (Siggel and Thomas, 1986). Different ring substituents in the phenolic ring give rise to different effects on the properties of phenolic compounds such as ionization (Crisponi et al., 2002). Thus the reactivity of phenolic compounds may depend on functional groups attached to the ring.

Most phenolic compounds are soluble in water, or those that are not, disintegrate into simple water soluble phenolic compounds. Then they can get incorporated into various sources of drinking water and eventually be a source of pollution. There are some serious health effects that can occur when humans or animals are exposed to certain phenolic compounds. It has been established

that a concentration of as low as 10 µg dm⁻³ (1 ppb) can affect the taste and odour of water and impact negatively on the quality of fish in certain aquatic environments (Martos et al., 2005). Others such as chlorophenols are poorly biodegraded and potentially carcinogenic (Marchessault, 1962; Torres et al., 2003). It is therefore important to determine the level of these compounds in drinking water within the areas where there is their indiscriminate use. This is with a view to ascertain the safety of water and their eventual removal to minimise the dangers associated with these compounds.

Phenolic compounds are known to react with quaternary ammonium compounds to form stable complexes (Jin and Preston, 1991). Quaternary ammonium salts are ionic surface-active chemical agents that consist generally of one nitrogen atom, surrounded by substitutes of carbon atoms on the four sights of the nitrogen atom (R₄N⁽⁺⁾). These cations are permanently charged and as a result can interact with any neutral or negatively charged material to form strong bonds (Assem et al., 2007; Xia et al., 2007). This property has been exploited by making quaternary ammonium compounds preferred material for wood preservative owing to their chemical interaction with lignocellulosic substrates in the wood (Jin and Preston, 1991). The reaction forms the basis addressing the challenge of removal of phenolic compounds in water with a view to offer solutions.

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Several methods have been reported on the removal of phenols from water. Degradation of phenol and chlorophenols from water by ozone using a pH range of 6.2–6.5 was reported by Manojlovica and co-workers (2007). In their study, degradation of 6.0 mg L^{-1} took 24 h. Activated carbon has been used as an adsorbent because of its large surface area (Qadeer and Rehan, 2002). Potgieter et al. (2009) also used fly ash as an adsorbent to remove phenols in aqueous solution. In both cases, the best adsorption was found to be between pH 3.12 and 3.4 and the adsorption capacity was below $1.00 \text{ } \mu\text{g g}^{-1}$. This is an extremely acidic environment and as a result of such limitations, other adsorbents such as quaternised maize tassels were considered with a view of removing phenolic compounds at the physiological pH of water.

Thus, in our study we report on the preparation of chemically modified maize tassels with a quaternary ammonium compound, polydiallyldimethylammonium chloride (polyDADMAC) and its application for the removal of selected phenolic compounds in water followed by UV–Vis spectrophotometric determination. The adsorbed (attached) phenolic compounds can easily be stripped off from the adsorbent by the use of dilute hydrochloric acid.

2. Material and methods

2.1. Chemicals and reagents

All the solutions were prepared in double distilled water and all the reagents used were of analytical grade. The standard phenols, 2,4 Dichlorophenol (24DCP), Chlorobenzoic acid (CBA), 2,4,6 Trichlorophenol (246TCP) and Dihydroxybenzene (DHB) hydrochloric acid 32%, methanol, sodium hydroxide, ammonium acetate and polyDADMAC were supplied by Sigma Aldrich, Gauteng-Johannesburg, South Africa. Dimethyl phthalate, propylene oxide, and epichlorohydrin were obtained from Lehlabile chemical supplies Ltd, Gauteng-Johannesburg, South Africa.

2.2. Instrumentation

The concentration of phenolic ions in aqueous solutions was determined using a Shimadzu UV-2450 UV–Vis spectrophotometer (Tokyo, Japan). Characterization of the adsorbent was carried out on a Fourier transform infrared (FT-IR) spectrophotometer (Perkin Elmer 100 with sampling accessory, Waltham, MA, USA) using the ATR mode, to find out the functional groups present in the modified adsorbent (Mukamel, 2000). The adsorbent was also characterized by means of a ^{13}C solid state NMR instrument with cross polarization/magic angle spinning (CP/MAS) facility (Bruker Avance 600 spectrometer, Hanau, Germany). The experiments were performed at a frequency of 10 MHz. The cross polarization was adjusted with glycine at 10 KHz spinning rate, set at 5000 Hz. The contact time for cross polarization was 1 ms and the delay time for acquisition was 5 s (Asakura et al., 2001). Characterization of thermal stability of the sorbents was done on a Perkin Elmer TGA 4000 instrument (Waltham, MA 02451, USA) (Pielichowski, 2007).

2.3. Preparation of cationic based adsorbents

2.3.1. Procedures

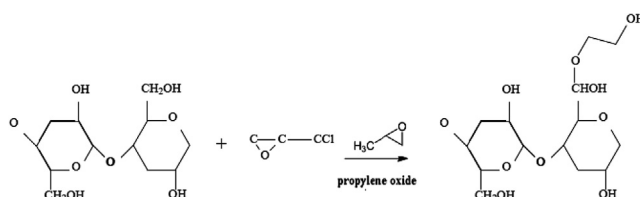
2.3.1.1. Preparation of Hydroxypropyl maize tassels (HP maize tassels). A dry powdered maize tassels sample (140 g) previously activated at 80°C for 12 h was weighed into a two litre three necked flask. The sample was mixed with cold 25% (w/v) aqueous sodium hydroxide (210 mL). To the mixture, a solution (8.40 mL) of epichlorohydrin dissolved in 70 mL of propylene oxide was added. The mixture was stirred thoroughly until the material finished swelling and all the liquid had been absorbed. The reaction vessel was then sealed and placed in a thermostated water bath at room temperature and later heated to 50°C and the temperature maintained for one hour. The reaction was cooled to room temperature and the damp, and the hydroxypropylated maize tassels (HP- maize tassels) sample was tested using a starch indicator paper for the extent of the hydroxypropylation process. Scheme 1 shows the preparation of hydroxypropyl cellulose or the maize tassels.

For alkylation reaction between HP-maize tassels and polyDADMAC, an epoxidation of the double bonds (alkenes) within its structure was carried out in-situ using hydrogen peroxide as an oxidant in a basic environment (Payne et al., 1961). These oxidation reactions of the double bonds within the structure of polyDADMAC gave cyclic ethers (epoxides) in which both carbons of a double bond become bonded to the same oxygen atom.

2.3.1.2. Epoxidation procedure. A standard epoxidation procedure as described by Bortolini et al. (2002) was adopted. The unsaturated compound, polyDADMAC, (25 mL) was treated with 30 mL of 30% hydrogen peroxide. The reaction mixture was made strongly basic by addition of 25 mL 14% sodium hydroxide solution. The mixture was then placed in a water bath at 60°C , and stirred vigorously for one hour. The product was analysed by UV–Vis spectrophotometry to determine the extent of epoxidation. It was then used for alkylation of the crosslinked material. Scheme 2 shows the epoxidation procedure on polyDADMAC.

2.3.1.3. Alkylation with 3-chloro 2-hydroxypropyldimethyldodecyl ammonium chloride. A solid sample of HP – maize tassels (20 g) and the epoxidated polyDADMAC prepared in Section 2.3.1.2 were placed in a 250 mL three neck flask and cooled to 4°C . A mass of 1.4 g of sodium hydroxide was introduced into the flask and thoroughly mixed for 30 min. The resulting mixture was then stirred continued for a further 30 min. The pH was monitored (maintained above 8.0) and the mixing was allowed to continue for 6 h in a thermostated water bath and the temperature was maintained at 60°C (Bortolini et al., 2002). The water-insoluble product was collected by vacuum filtration, washed with water until the pH was of the brought to neutral, dried in air and then the material was characterised. Scheme 3 shows the alkylation of the cross-linked material.

The product obtained was characterised with ^{13}C NMR, FTIR and DSC and then applied for the removal of phenolic compounds in aqueous media.



Scheme 1. Functionalizing maize tassel by attaching hydroxypropyl (HP) group to form a modified tassel (HP-maize tassel complex).

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