



Research paper

Sorption of Congo red and reactive blue on biomass and activated carbon derived from biomass modified by ionic liquid



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ABSTRACT

Ionic liquid modified sorbent materials derived from peanut shell were prepared and used in the sorption of Congo red (CR) and reactive blue 4 (RB) dyes from aqueous solution. 1-methyl-3-decahexyl imidazolium ionic liquid (IL) was used to modify peanut shell (NS) and activated carbon (AC) from NS, to produce IL modified peanut shell (ILNS) and IL modified AC (ILAC), respectively. The adsorbents were characterised by fourier transform infrared (FTIR), Brunauer – Emmett – Teller (BET), x-ray diffraction (XRD), thermal gravimetric analysis (TGA) and scanning electron microscopy (SEM). The effects of sorbent dosage, initial solution pH and contact time (min) were evaluated. Adsorption was pH dependent, the modified materials (ILNS and ILAC) showed improved adsorption capacities of 136.4 and 150.0 mg g⁻¹ for CR and 290.0 and 364.4 mg g⁻¹ for RB respectively. Adsorption followed pseudo-second order. Also, adsorption of CR and RB onto ILAC followed the Freundlich isotherm, while ILNS was described by Freundlich and Langmuir.

1. Introduction

Thousands of different dyes are produced annually for use in various industries ranging from textiles and tanning to paper and pulp. Of these, between 35 and 70 thousand tonnes of this dye ends up in industrial effluent which in most cases is simply dumped into natural water ways (Roy Choudhury, 2013). The extremely adverse effects these dyes have on aquatic flora and fauna are exacerbated by the compounds recalcitrance in nature. Congo red (CR) and reactive blue (RB) are known to have a negative impact on the environment. Benzidine, one of the products resulting from the degradation of CR is a known carcinogen (Boeniger, 1980). Extended exposure to RB has been noted to cause renal complications in addition to central nervous and reproductive system disorders (Dawood and Sen, 2012; Yagub et al., 2014). Also, RB which is a triazine dye with an anthraquinone structure, is reported to be a potential carcinogenic, mutagenic and allergenic pollutant (Aksu and Tezer, 2000; Bayramoğlu et al., 2006; Heiss et al., 1992; Özer et al., 2005).

Their many methods of wastewater remediation which include chlorination, oxidation, and ultrasonic irradiation. Photo degradation, coagulation, precipitation, biodegradation, membrane separation and adsorption have also been utilized (Akpotu and Moodley, 2016; Babalola et al., 2016c; Crini and Badot, 2008; Lawal and Moodley, 2015). Adsorption is commonly applied in the removal of organic

compounds and dyes from wastewater due to its high removal efficiency. Activated carbon (AC) is the most used adsorbent, but the use is limited not only due to its high cost, but also inefficiency in removing oil, grease, natural organic matter, and other large organic molecules (Alther, 2002). There is therefore need for inexpensive yet efficient and competitive adsorbent materials to be developed.

Biosorbents, which include chitosan, peat, sugarcane bagasse, straw and rice husks, activated bamboo and many more, are some of the recent low-cost materials used for the removal of pollutants from water bodies. They are cheap, easy to use and readily available compared to AC. Low adsorption capacity has been the bane of biosorption, particularly its industrial use in cleaning up toxic pollutants from wastewater (Singh et al., 2012). This has led to the modification of biosorbents to enhance its adsorption capacity. Modifications ranging from acid or alkaline treatment, acetone and ethanol has been reported (Montazer-Rahmati et al., 2011). Surfactants has also been used in the modification of biosorbents for the adsorption of organics pollutants. Researchers reported improved biosorption when biomasses were modified with surfactants (Akar et al., 2013; Aksu et al., 2010; Brandão et al., 2010).

Activated carbon from biomass is another way of improving the adsorption capacity of biomass. Foo and co-workers reported superior adsorptive capabilities of AC compared to its natively prepared biomass (Foo et al., 2012). Recently, to further improve the adsorption capacity

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of AC, surfactants have been used to modify them as well. Since AC is not efficient in removing oil, grease, natural organic matter, and other large organic molecules (Alther, 2002), therefore modification of this AC is essential to make it more suitable for the adsorption of organic pollutants. Researchers reported the modification of AC with surfactants for the removal of organic pollutants (Choi et al., 2008; Cota-Espericueta, 2003).

Ionic liquids as surfactants have recently attracted an increasing amount of interest, owing to their low volatility, non-flammability, high chemical and thermal stabilities, high ionic conductivity and broad electrochemical windows (Zhang et al., 2006). Recently, they have been used as surfactants for the modification of materials for adsorption of organic pollutant (Absalan et al., 2011; Kamran et al., 2014a; Kamran et al., 2014b; Lawal and Moodley, 2016a; Lawal and Moodley, 2015).

Therefore, the aim of this study was to investigate and evaluate the adsorption behaviour and compare the biosorption capacities of sorbent materials derived from peanut shells (sp *Arachis hypogaea*) (native (NS), activated carbon (AC) derived from NS and their modification with ionic liquid to produce ILNS and ILAC respectively). A deep literature search has shown that no work has been reported on the modification of *Arachis hypogaea* biosorbent and its AC derivative with ionic liquid (1-methyl-3-decahexyl imidazolium) for the biosorption of dyes.

2. Experimental

2.1. Materials

The raw peanuts (sp *Arachis hypogaea*) was sourced in Durban, and ionic liquid, 1-methyl-3-decahexyl imidazolium was synthesised in our laboratory. All other chemicals used were of analytical grade and sourced from Sigma Aldrich (99.999% purity). The structures of Congo red and reactive blue is given in the supplementary information (SI).

2.2. Adsorbent preparation

2.2.1. Synthesis and characterisation of 1-methyl-3-decahexyl imidazolium

The synthesis and characterisation of ionic liquid (1-methyl-3-decahexyl imidazolium) used in this research has been reported elsewhere (Lawal and Moodley, 2015).

2.2.2. Preparation of nut shell (NS)

The peanut was removed from the shell, washed successively with tap water and double distilled deionized water to remove any unwanted particles and membranes. Thereafter, the shell was dried in an oven at 85 °C until dry, ground in an Angstrom TE250 ring and puck pulveriser and sieved through a 120 µm sieve. A mass of 50 g powdered peanut shell was mixed with 100 mL of 1 M sodium hydroxide and then stirred for approximately three hours at room temperature. Thereafter, it was filtered off and washed repeatedly with double distilled deionised water until a neutral pH was achieved. The material was then dried in an oven at 85 °C until dry, labelled NS and stored in a desiccator until further use.

2.2.3. Preparation of activated carbon from NS

NS was calcined in a furnace under constant nitrogen flow, at a heating rate of 10 °C min⁻¹ until a temperature of 500 °C was reached. The temperature was then maintained for a further two hours. After two hours the calcined material was activated by increasing the temperature to 800 °C (under nitrogen) for a further one and a half hours. The AC obtained from the calcination was stored in a desiccator and labelled AC.

2.2.4. Modification of NS and AC

The modification of NS and AC by ionic liquid was done by adding ionic liquid in excess of its critical micelle concentration (CMC) (0.01 × 10⁻³ mol dm⁻³) as determined by Mandavi et al. (2008). A

mass (in grams) of NS or AC powder was added to an already dissolved and known amount of ionic liquid in methanol. The mixture was stirred at room temperature for 24 h, and thereafter, dried in an oven at 80 °C overnight. The resulting modified material was cooled down, washed several times with double distilled deionized water and dried in an oven (80 °C). The resulting materials were labelled ILNS and ILAC respectively and stored in a desiccator until further use.

2.3. Characterisation of NS, ILNS, AC and ILAC

The adsorbents NS, ILNS, AC and ILAC were characterised by FTIR using the KBr disc preparation method, and scanning electron microscopy (SEM) using the Leo 435 VP model. Thermal gravimetric analysis (TGA) (SDT Q 600 V 20.9 Build 20 instrument) was used to determine the thermal behaviour of the adsorbents. It was measured as a function of increasing temperature from ambient to 1000 °C (with a constant heating rate of 5 °C min⁻¹) under nitrogen atmosphere with a flow rate of 50 mL min⁻¹. The materials were analysed using x-ray diffraction (XRD) (Bruker D8, Cu Kα radiation, 45 kV, 40 mA and λ of 0.154 nm), and data were collected for 2θ ranging from 10° to 90° with a scan speed of 1° min⁻¹. BET (Tri-star II 3020.VI.03) was used to determine the surface area, pore-size, pore volume and pore size distribution. The materials were first degassed (Micromeritics vacprep 061, sample degas system) at 90 °C for 1 h and was increased to 200 °C for 12 h. The sample was then analysed under a nitrogen atmosphere at 77 K. The spectrum was scanned at 650–4000 cm⁻¹. The Crystallinity index (CrI) of the materials were determined using the XRD spectra of the materials. The crystallinity index CrI was determined by the Eq. (1) proposed by Segal et al. (1959)

$$\text{CrI} = (I_{002} - I_{\text{am}})/I_{002} \quad (1)$$

Where, I_{002} is the scattered intensity at the main peak for cellulose type I and I_{am} is the scattered intensity due to the amorphous portion evaluated as the minimum intensity between the main and secondary peaks.

2.4. Adsorption experiments

2.4.1. Batch equilibrium experiments

Batch experiments included the investigation of the effects of sorbent dosage, initial solution pH and contact time (min) for both CR and RB. The effect of dose of the adsorbents was carried out with each of the four materials (NS, ILNS, AC and ILAC) ranging from 0.01 to 0.15 g into 30 mL of 100 mg L⁻¹ of each dye in 250 mL capacity glass stopped Erlenmeyer flasks. The flasks were wrapped with aluminium foil and equilibrated on a shaker for 24 h in the dark at 120 rpm using a horizontal shaker. Thereafter, the dye samples were separated from the adsorbent by filtration through a 0.45 µm whatman filter paper. The effect of pH (2–11) and contact time (min) were carried out using the same concentration and volume as in the case of dose. Blank was also determined under the same conditions but without the adsorbents. The concentration of the supernatant was analysed using a double beam UV–vis–NIR spectrophotometer (Shimadzu Model UV 3600, Japan) at λ_{max} 498 nm for CR and 594 nm for RB.

2.4.2. Kinetic and isotherm experiments

A constant mass (0.05 g) of each material was added to separate conical flasks containing 150 mL of dye in the concentration range of 20–200 mg L⁻¹. The capped flasks were then wrapped in foil and placed on an orbital shaker set at 220 rpm. Aliquots of the solution from each flask were removed at regular time intervals, 5, 10, 15, 20, 30, 45, 60 min and subsequently at an interval of 1 h over a period of 24 h, filtered under vacuum and the filtrates collected for analysis by UV spectrophotometry.

The adsorption capacity q_e (mg g⁻¹) of the materials were calculated using the following equation.

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