

Adsorption of wood extractives and model compounds onto bentonite



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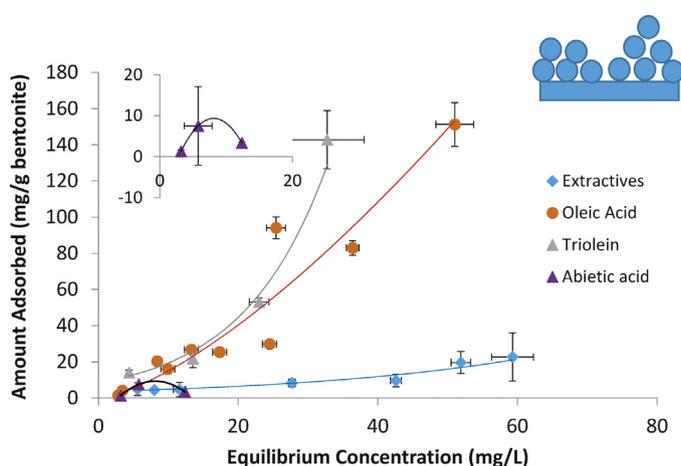
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HIGHLIGHTS

- Adsorption isotherms of wood extractives onto bentonite show multilayer adsorption.
- Wood extractives have a higher affinity for bentonite than triolein or oleic acid.
- Na-bentonite with large surface area adsorbed more wood extractives.
- Addition of ethanol to dispersions decreased adsorption of extractives to bentonite.

GRAPHICAL ABSTRACT



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ABSTRACT

The adsorption of wood extractives and representative model compounds into two different forms of bentonite was studied. The colloidal dispersion showed limited solubility which affected the shape of the adsorption isotherm. Modelling of the adsorption isotherms showed excellent fits with Freundlich and BET isotherm models indicating that multilayer adsorption with cooperative binding was occurring. The wood extractives were found to have greater affinity with the bentonite surface than the model compounds. Bentonite B was found to be a better adsorbent than bentonite A due to the differences in exchangeable cations present and its greater swelling capacity and surface area. The addition of ethanol during the preparation of the colloidal dispersions, as done by previous researchers when studying adsorption of the oleic acid, triolein and abietic acid, appears to reduce the affinity and capacity of the adsorption onto bentonite.

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1. Introduction

Inorganic mineral fillers are used as adsorbents in many industrial processes to remove troublesome material from the process. In papermaking, this troublesome material causes major problems with fouling of equipment, lost production, reduced efficiency and

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increased costs to the industry. The troublesome material arises from colloidal dispersed material originating from the wood and other additives used in the process. In particular, the wood resins which are released from the wood are a major contributor to the problem. The wood resins, also known as wood extractives, are a mixture of different chemical classes. The main classes are the resin acids, fatty acids, triglycerides, sterols and steryl esters. In an aqueous phase, these compounds form negatively charged colloidal droplets typically 0.2–1 μ in size [1–6]. The stability of the droplets is affected by a number of factors including the process pH [3,7–10], temperature [7,10,11], shear conditions [9,10], composition of wood resins [2,7,8,12–14], presence of bacteria [6], and dissolved salts [1,11,15,16], as well as dissolved organic matter originating from the wood hemicelluloses which partially dissolve under the temperature conditions used in the pulping process [3,11,12,17–20].

A range of natural and surface modified materials including talc, calcium carbonate, kaolinite, bentonite and zeolite are used to adsorb dissolved and colloidal material originating from the wood and pulping process [21–33]. Talc was one of the earliest adsorbents used in the pulp and paper industry for control of sticky material such as wood resins [34–36]. However it has been found to be difficult to disperse, requiring surfactants to aid dispersal and these cause significant foaming problems [37], and does not significantly reduce the hydrophobicity of wood resin colloids compared to other inorganic adsorbates [38]. It has also been found that talc is non selective and many other substances such as dissolved organics from hemicellulose and lignin as well as defoamers compete with the wood resin and so decrease the efficiency of talc to remove wood resins from the process water [23,36,39,40].

Calcium carbonate is used as a filler and a coating additive in neutral and alkaline papermaking. Ground calcium carbonate (GCC) is generally anionic in charge while precipitated calcium carbonate (PCC) is cationic [31]. Several studies have shown that PCC and modified GCC are effective for adsorbing sticky materials [24,29,31,38]. The adsorption of oleic acid and abietic acid onto calcium carbonate is believed to be a chemical adsorption process through the formation of calcium soaps precipitating on the surface [24]. The use of calcium carbonate as an adsorbent is restricted to neutral and alkaline processes.

Kaolinite and bentonite are also used as fillers and adsorbents in papermaking and other applications. These materials are hydrophilic flake-like particles comprising crystalline layered structures of tetrahedral silica sheets and octahedral alumina sheets [41]. The particles are amphoteric and develop cationic charges along their edges and anionic charges on their basal surfaces under neutral to low pH conditions. They also swell and expand when dispersed in water, greatly increasing their surface areas. Kaolinite has a 1:1 layered alternating structure of silica and alumina with a low cation exchange capacity. Bentonite (which belongs to the montmorillonite class of minerals) is a 2:1 layered structure of silica:alumina with a high cation exchange capacity. Adsorption of resin acids, fatty acids and triglycerides onto bentonite occurs primarily through the exchangeable cations of the bentonite [41] and it has been found to be superior to other adsorbents in reducing hydrophobicity of wood resin and reducing deposition tendency [38].

Only a limited number of studies have measured adsorption isotherms of wood extractives and the model compounds present in the wood extractives onto mineral surfaces such as talc, calcium carbonates, kaolin and bentonite [21,22,28,30]. No studies have been reported for the adsorption of extractives from resin acid rich *Pinus radiata*. This paper compares isotherms of model compounds and wood extractives, from *P. radiata*, onto two types of commercially available bentonite and proposes mechanisms for the difference in behaviour that occurs.

2. Experimental

2.1. Materials

Model compounds of abietic acid (85% ACROS Organics), oleic acid ($\geq 99\%$ purity, Fluka) and triolein (glyceryl trioleate, $\geq 99\%$, Sigma–Aldrich) were used. Solutions of the model compounds were prepared by weighing out the required amount and dissolving the compound in acetone (AR grade, Chem-Supply) unless otherwise specified.

Wood resin was extracted from *P. radiata* thermo-mechanical pulp (TMP) sourced from the primary refiner at the Norske Skog Mill in Boyer, Tasmania. The pulp was air-dried and then freeze-dried using a Breda Scientific freeze drier. Extraction was performed on the dried pulp using hexane (LR grade, Chem-Supply) in a soxhlet apparatus for 8 h. The extract was stored in the freezer until required and then mixed in acetone before use. Two commercial bentonite samples (termed bentonite A and bentonite B) were obtained from Norske Skog Boyer Mill.

2.2. Preparation of bentonite

Bentonite was prepared by size fractionation from a 2% slurry in deionised water after stirring at 700 rpm for 15 min. The suspension was then centrifuged at 1550 rpm for 15 min in an Eppendorf 5810 centrifuge. The supernatant was removed and the settled bentonite was made up to its initial volume with deionised water. This process was repeated until at least 98% of the particles had a diameter greater than 0.45 μ m as determined by laser particle size analysis (LPSA). The final settled material was removed and dried in an air oven at 60 °C.

Prior to adsorption experiments, a 0.45% bentonite slurry was prepared using deionised water. The slurry was stirred overnight at 480 rpm to allow hydration of the bentonite to occur.

2.3. Characterisation of bentonite

2.3.1. Particle size analysis

A Micromeritics Saturn DigiSizer 5200 fitted with a Sonics Vibra Cell Ultrasonicator was used to measure the particle size distribution. The flow rate of the instrument was set at 12 L/min. For the dispersant, the refractive index was set at 1.331 with a density of 0.998 g/cm³. The material properties were set at a refractive index of 1.57 and a density of 2.650 g/cm³ when measuring bentonite samples and 1.54 refractive index and 0.890 g/cm³ density when using wood resin samples. The dispersant used was either deionised water or 1 mM potassium nitrate solution.

2.3.2. Zeta potential measurements

Zeta potential measurement was performed at 25 °C using a Malvern Nano ZS Zetasizer and Malvern disposable folded capillary cell. The dispersant used was either deionised water or 1 mM potassium nitrate solution pH adjusted to 5.5.

2.3.3. BET surface area determination

A Micromeritics TriStar II 3020 V1.01 was used to determine the BET surface area of bentonite samples. The sample density was set at 1.000 g/cm³ and the samples were run with an equilibrium interval of 25 s.

2.3.4. Exchangeable cation determination

A method derived from Sumner and Miller [42] was used to extract the exchangeable cations of fractionated bentonites. Approximately 0.5 g of bentonite was weighed accurately and added to a 50 mL centrifuge tube with 20 mL of 0.2 M ammonium

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