



## Development of *in situ* soft colloidal probe atomic force microscopy for probing the adhesion between wood extractives and model surfaces



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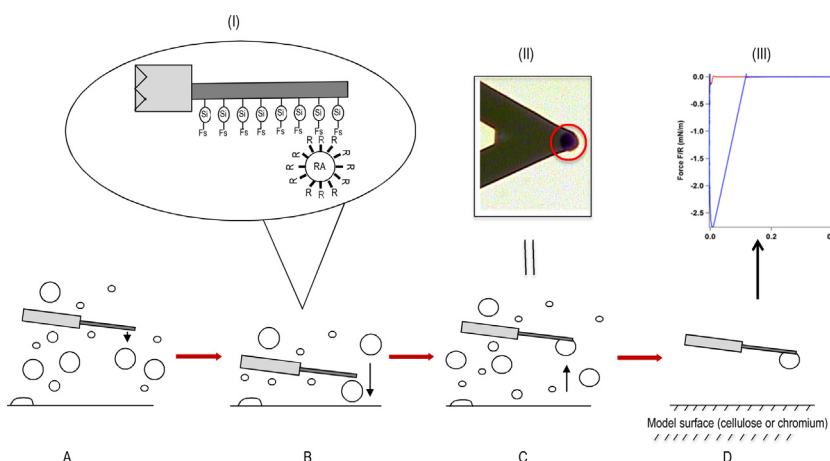
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### HIGHLIGHTS

- A new method developed to attach soft colloid onto tipless AFM cantilever.
- Adhesion forces of abietic acid and wood extractives (WE) are lowest on cellulose.
- On chromium, adhesion forces are highest for abietic acid (AA) and WE.
- Adhesion forces of AA and WE on cellulose and chromium are comparable.
- AA, instead of TrO and OA, preferentially transfers from cellulose to chromium.

### GRAPHICAL ABSTRACT



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### ABSTRACT

A new method to attach colloidal particles *in situ* directly onto atomic force microscopy (AFM) cantilevers was developed and used as colloidal probes. In this method, cantilever surfaces were hydrophobically functionalised and the colloids adhered without glue in an aqueous environment. Experiments measuring the interaction forces between colloidal probes, composed of either wood extractives or a range of model compounds normally present in wood extractives, and glass, cellulose, and chromium surfaces were carried out. Wood extractive colloids exhibited the strongest adhesion to glass control surface ( $7.0 \text{ mN m}^{-1}$ ), followed in decreasing order by triolein, abietic acid and oleic acid colloids ( $3.9 \text{ mN m}^{-1}$ ,  $2.2 \text{ mN m}^{-1}$  and  $1.2 \text{ mN m}^{-1}$  respectively). Adhesion onto a cellulose surface was strongest for triolein colloids ( $8.1 \text{ mN m}^{-1}$ ) followed by oleic acid colloids ( $6.1 \text{ mN m}^{-1}$ ), while adhesion forces for abietic acid and wood extractives colloids were similar ( $4.9 \text{ mN m}^{-1}$  and  $4.3 \text{ mN m}^{-1}$ , respectively). When attached to a chromium surface adhesion forces of abietic acid and wood extractives were strongest and similar ( $3.6 \text{ mN m}^{-1}$  and  $3.9 \text{ mN m}^{-1}$ , respectively); the weakest interaction at this surface was recorded for oleic acid colloids ( $0.8 \text{ mN m}^{-1}$ ). Similar adhesion forces

**Abbreviations:** AA, abietic acid; AFM, atomic force microscopy; NaCl, sodium chloride; OA, oleic acid; PEI, poly(ethylene)imine; TrO, triolein; WE, wood extractives.

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for wood extractive colloids and abietic acid colloids (onto cellulose and chromium surfaces) reinforces the proposal that abietic acid occupies the outer layer of wood extractive colloids. The differences in the interaction between the colloids and the cellulose and chromium surface helps to explain the transfer of resin acid exclusively over triolein or oleic acid from a cellulose paper surface onto chromium plated printing cylinders.

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## 1. Introduction

During the paper making process, dissolved and colloidal substances are released from thermomechanical pulp fibres into the process water [1]. The colloidal substances consist of fatty and resin acids, triglycerides and sterols, which are collectively referred to as wood extractives [1–5]. When dispersed in water, the wood extractives adopt a layered droplet structure with hydrophobic steryl esters and triglycerides at the core of the droplet, while the fatty acids and resin acids form an outer layer with their carboxyl and hydroxyl groups extending into the water phase [4].

Accumulation of wood extractives in the process water, which leads to the formation of larger agglomerates or 'pitch', results in their deposition onto surfaces of machinery that come into contact with the process water during the paper making process [6]. A commonly used method to reduce accumulation of wood extractives has been to actively adsorb the extractives onto the paper using organic polymers or polymers mixed with minerals, without compromising paper strength [6]. However, this merely transfers the problem to the production and printing machinery contributing to desorption of extractives from the paper onto surfaces of high-pressure rollers, and even onto chromium surfaces of printing presses that are employed well-beyond the production stage (Richardson D, pers. comm.).

Most previous studies on wood extractive colloids have focused on the chemical structure of the colloids [5,7–9]. For example Sundberg et al. [5] studied the effect of pH on the distribution of fatty and resin acids within the wood extractive colloidal solution and Stack et al. [10] showed how composition of wood extractives, especially the amount of triglycerides, will affect the colloidal stability. There have been fewer reports on subsequent dynamic changes to the colloids once they have formed and little is understood in regard to their surface chemistry-dependent interactions with different surfaces [11,12]. The formation of pitch deposits and thin films onto the surfaces is believed to originate from the complex interplay of adsorption/desorption of the colloidal wood extractives and their individual compounds (e.g. fatty and resin acids) from process water and/or paper surfaces. This process may be influenced by various factors such as pH, temperature, pressure (e.g. of the metal rollers) and surface chemistry [13]. Even though many components make up wood extractives, it has been shown that in the resultant films of deposit is almost exclusively resin acids that deposit onto surface during paper manufacture and printing (Richardson D, pers. comm.).

There is a need to understand why only resin acids from wood extractives desorb from paper and fatty acids and triglycerides are retained in the paper web. Therefore, to gain a better understanding of the interactions between colloidal wood extractives and different surfaces, including those encountered in the paper making and printing processes, this study employed the use of colloidal probe atomic force microscopy (AFM). Previously when this technique was applied to colloidal materials, a hard inorganic particle (e.g. silica) with a defined surface chemistry and geometry was typically attached (glued) to an AFM cantilever to measure interaction forces between the colloid and different surfaces [14–16]. Different types

of colloids were also attached to mimic and study a colloidal system of interest, with the sphere-flat geometry enabling modelling of the interaction [15]. Hard microspheres such as silica, zirconia, alumina and polymeric microspheres, have traditionally been used as colloidal probes [14,16]. Of direct relevance to this study is the work by Wallqvist et al. [2], more specifically the use of colloidal probe in force measurements, in which the adhesion forces between abietic acid and talc surfaces were investigated. In their study, the colloidal probes were prepared in air by attaching either silica colloids coated with wood extractives, dried pitch particles that were non-spherical (precipitation method), or pitch particles that were subsequently heated and melted to produce smoother particle surfaces (melt method). Force measurements in 6 mM NaCl showed that the interaction forces between abietic acid and talc surfaces were initially repulsive then became strongly attractive on approach due to bridging of sub-microscopic bubbles existing on the hydrophobic surface of abietic acid and talc. In contrast, the attractive interaction on approach and subsequent pull-off adhesion to the talc was significantly less for collophonium and pitch colloids, suggesting a greater presence of polar surface groups.

Considering that the wood extractives are effectively soft colloids in a liquid, it is of interest to study them in this form during their interactions with surfaces under relevant conditions. Study of the interactions of soft colloids has recently provided an improved understanding of soft matter in various biomedical, engineering and industrial applications [14,17,18]. There have been studies involving soft colloids in solution between rigid microspheres and droplets [17], between attached bubbles and surfaces [18] or between two liquid droplets/bubbles [19–21]. Chan et al. [22] and Tabor et al. [23] have presented separate reviews on the development and theories involved in AFM force studies of liquid droplets and bubbles. In this study, we specifically use soft colloidal probe-AFM to measure the interaction forces between various model soft colloids and surfaces, including colloidal forms of oleic acid (represents fatty acids), abietic acid (represents resin acids), triolein (represents triglycerides) and natural wood extractives, and model surfaces that are representative of paper (microcrystalline cellulose) and metal-coated printing presses (chromium). This study investigates why there is a greater tendency of resin acids to desorb from paper surfaces onto metal surfaces in preference to other components in wood extractives such as fatty acids or triglycerides. In doing so, we present a novel method for attaching soft colloids *in situ* (in liquid) prior to undertaking the AFM force measurements, which is advantageous for maintaining the native structure of these colloidal systems.

## 2. Method and materials

### 2.1. Preparation of model surfaces

#### 2.1.1. Glass surface

Glass coverslips (ProSciTech, Coverglass No. 1, 18 mm) were wiped with 70% ethanol solution then with 18 M $\Omega$  MilliQ water on lens paper. The cleaned glass cover slips were dried under a gentle stream of nitrogen before use.

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