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Towards the development of superhydrophobic carbon nanomaterial coatings on wood

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ABSTRACT

Carbon nanomaterials (CNMs) have recently been used to form superhydrophobic coatings on metals, synthetic polymers or textiles. Here we investigate the possibility of using carbon black (CB), graphene (Gr) and carbon nanotubes (CNTs), as water repellent agents on naturally hydrophilic wood. We show that it is possible to form homogeneous CNM coatings on any type of wood via simple methods of drop casting and dip coating, using CNMs dispersed in organic solvents or water. Contact angle measurements of wood coated with only 0.05 g/m^2 CNTs and 0.25 g/m^2 Gr gave the results exceeding 130°, indicating apparent hydrophobicity. Yet, high adhesion of the droplets was observed, simultaneously suggesting a "rose petal" type of superhydrophobic behavior. That may be explained by the formation of micro-nano architectures in which low surface energy CNMs deposited on microrough surface of wood cause superhydrophobicity. Yet, due to heterogeneity of wood, some part of hydrophilic surface is still uncovered, resulting in high adhesion of water. Finally, although Gr and CNT were only physically bond to wood surface, the hydrophobic properties of CNM coatings were maintained after sandpaper abrasion test. Moreover, wood fibers and particles covered with Gr showed the decrease of water absorption equal 98% and 87%, respectively.

1. Introduction

Although, wood has been successively replaced by concrete and steel, over the years, it is still one of the extremely widely used construction materials. On the contrary to the past trends modern world sees wood as an extremely valuable resource due to the fact that it is a fully natural, renewable and biodegradable material and has low carbon footprint. Unfortunately, wood still has several disadvantages, which have been the limiting factors before. One of them is low resistance to water which deteriorates its mechanical performance, increases construction mass [1] and enables the growth of mold [2]. Although many techniques of surface preservation has been recently developed [3–5], none of them can fully solve the hydrophobicity problem in every application of wood, therefore any new exploration in this area may be highly valuable. Here we propose, for the first time ever, that recent developments in nanotechnology area could help in this respect and offer inexpensive and easily up-scalable method of hydrophobization of wood and its composites by the use of carbon nanomaterials (CNMs).

Due to unique properties and rapidly decreasing price, carbon nanomaterials, such as graphene (Gr) and carbon nanotubes (CNTs), have recently found interest in both science and industry. It has been observed that these carbon allotropes properly structured at micro-nanoscale may show superhydrophobic effect and form various types of water repellent materials and coatings. Particularly pronounced effect imitating water repellent properties of lotus leaves (also referred to as "lotus effect") has been observed in CNT forests i.e. vertically aligned CNT arrays [6–9]. Lotus leaves exhibit high water contact angles – above 130° and low adhesion with sliding angles below 5°. Therefore, raindrops can easily roll off the surface, without wetting the leaves. This effect is caused by the unique micro-nanoscale hierarchical and rough architectures of the leaves surfaces [10]. Various types of water repellent behavior have been also found in other CNT, Gr architectures including graphene sponges [11], films [12,13], and foams [14,15],

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carbon black (CB) coatings [16] or nanoengineered CNTs [17].

Further studies showed that powders of CNMs may act as hydrophobic agents for other materials, including metal meshes [18,19], polymer sponges [20], textiles and cellulosic materials like cotton [21–26]. All of these substrates are characterized by micro-porous structure, which in combination with low surface energy of carbon allotropes is responsible for superhydrophobicity of the final product. Graphene has been used in cellulose nanocomposites [22,23], showing both increased hydrophobicity and enhanced mechanical performance.

In this paper, we test the possibility of using Gr and CNT and for comparison of CB which is a well-known high specific surface area hydrophobic carbon allotrope of amorphous structure as superhydrophobic agents in wood technology applications. Although wood is a cellulose rich material and its chemical composition is therefore somehow similar to cotton (90% cellulose content), yet wood consists of comparably significant amounts of lignin and hemicelluloses which together may exceed even 50% of woods mass. Moreover, wood comprises fatty acids, resin acids, waxes and terpenes [27]. Woods with higher extract content in general are more hydrophobic [28]. Therefore, it is a much more complex material than cotton and its interaction with CNMs might be considerably different from cotton-CNM ones. Additionally, wood has also its own specific surface microstructure which is influenced by processing such as sawing or polishing. All these aspects could influence the formation of CNM coatings and their wetting characteristics. Moreover, wood consists of nano, micro- and mesopores, which are specific to each wood species. The influence of roughness onto wetting of wood is not straightforward, but it was presented that while increasing the roughness of wood the contact angle at first slightly decreases, but for very rough surface it increases and wood becomes even more hydrophobic [28].

The purpose of this paper is fourfold. First, we demonstrate that most popular types of wood may be coated by CNMs using simple methods of dip coating and drop casting. We show that both organic solvent and water-based dispersions of CNMs may ensure the deposition of pure CNM materials on the surface of wood. Next, we analyze the morphology of coatings formed on bulk wood and wood-based products such as fibers and wood particles used for the production of popular wood composites such as fiberboards or particleboards, and prove their hydrophobic properties. Finally, the model of hydrophobicity of our coatings is proposed.

2. Experimental

2.1. Materials

The initial experiments were performed on balsa (*Ochroma pyramidale*), as a highly water absorptive wood, with a significant cellulose content and homogeneity [29]. The comparative tests were further carried out on scots pine (*Pinus sylvestris*), oak (*Quercus*), birch (*Betula*), beech (*Fagus*) and poplar (*Populus alba*) woods, characterized by different pore distribution and chemical composition [30,31]. Industrial grade, sphere-like CB (N-550) was obtained from Konimpex Ltd. Gr with average diameter of 300 nm and thickness below 1 nm was obtained from Cambridge Nanosystems Ltd. (Camgraph G1). Industrial grade, short and tangled multiwall carbon nanotubes (MWCNTs) (NC-7000) were ordered from Nanocyl SA. Sodium dodecylbenzenesulfonate (SDBS), technical grade, was purchased from Sigma Aldrich. Dichloromethane (DCM), spectroscopy grade, was ordered from Avantor Performance Materials Poland SA. Water was filtrated by Milli-Q® ultrapure water system.

2.2. Preparation of coatings

The procedure started from preparation of CNM dispersions in an organic solvent. At first, all CNMs were mixed with DCM in the concentrations of 1 mg/2 ml CB, 1 mg/30 ml and 1 mg/2 ml Gr and 1 mg/60 ml MWCNT. Then the samples were sonicated for 10–20 mins (power 60 W per 40 ml of mixture) to obtain uniform dispersions. These CNM dispersions were further deposited onto wood via drop-casting and the samples were dried at 40 °C on a hot plate. Due to very high vapor pressure of DCM, the applied heating should be sufficient to remove any DCM trapped between CNM and wood surface. The droplets were applied slowly in order to let DCM to evaporate before next droplet was casted. Due to capillary forces in wood pores the suspension was naturally distributed onto the surface, but no CNMs were found inside wood.

The amount of deposited dispersion was set to obtain two selected values of mass per square meter. Thus, in order to obtain 0.05 g/m^2 Gr (1.5 ml of 1 mg/30 ml) and CNT (3 ml of 1 mg/60 ml) were drop casted onto 10 cm^2 of balsa wood (BW). Whilst for 0.25 g/m^2 (0.5 ml of 1 mg/2 ml) CB and Gr was deposited on 10 cm^2 of BW. Gr was chosen for CNM deposition on various wood types, due to good stability of highly concentrated dispersion. Gr was deposited by dip-coating in 1 mg/2 ml Gr repeated 10 times. The same procedure was applied for wood fibers and wood particles.

Although the use of organic solvent such as DCM enables investigation of CNM deposition on wood, it is not convenient in potential industrial applications. Thus, the aqueous dispersion was tested as well. The aqueous dispersions were prepared using 0.3 g of SDBS and 0.3 g of CNM per 100 ml of ultrapure water. The concentration was relatively high in order to obtain more hydrophobic surface. Then, it was sonicated for 10–20 mins similarly as for organic solvent dispersions. CNMs were deposited on wood surface via dip-coating, followed by drying at 90 °C. Afterwards, the samples were washed 3 times in order to remove the outer layer of the surfactant and again immersed in CNM dispersion. The procedure was repeated three times, to obtain 3 layers of CNMs. Then, according to Makowski et al. [24] SDBS was removed by several washing and drying cycles.

2.3. Surface imaging

CNMs were dispersed via Hielsher 400 St (400 W) ultrasonic horn. The SEM images were taken by FEI Nova NanoSEM 650 and the EDS by Bruker XFlash® 5010 detector. Due to excessive charging under SEM, wood coated with CB was covered with 10 nm thick layer of platinum (Pt) which enabled successful imaging of the surface. The fluorescent microscopy images were taken by Zeiss Laser Scanning Microscope (LSM) 510, using a mercury lamp HXP 120 and a filter set 38. Each image was taken with the exposure time of 300 ms. The roughness, calculated as quadratic mean deviation of all profile height values (R_{qa}), was measured using the confocal mode of Zeiss LSM 510.

2.4. Roughness and robustness measurements

The roughness of wood was measured by Zeiss LSM 510. The measurement and calculations were performed according to [32,33]. Briefly, topography map was obtained by LSM, using argon laser (458 nm) and EC Epiplan-Nefluar 20 × lens. Then, the roughness was calculated by ZEN2011software, as the arithmetic mean of total surface height values RS_a, according to DIN EN ISO 428. The topography map was taken in five random spots on each wood type and the average value is presented in manuscript. The standard deviation of the mean was used as measurement uncertainty.

To demonstrate the robustness of CNM coated wood we performed sandpaper abrasion test (P100 grit) according to Chen et al. [34]. Briefly, 3 cm^2 wood coated with CNM were placed onto sandpaper facedown under a weight of 17 g (approximately 550 Pa). Then, the sample was dragged thirty times 10 cm longitudinally and transversely in each direction.

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