



Paper coating performance of hemicellulose-rich natural polymer from distiller's grains



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ABSTRACT

Coatings contribute to about 10% of the overall cost of paper production of which, the binder is the highest cost component in the formulation. In addition to costs, the binders that are currently being used are synthesized from fossil fuel feedstock raising concerns towards the sustainability of paper coatings. Furthermore, papers coated with waxes, polyolefins and other synthetic materials are difficult to recycle or compost. These challenges inherent with synthetic binders can be addressed by using renewable, plant based binders. This research focused on an abundant and underutilized source of hemicellulose based binders (DG gum) that can be extracted from distiller's dried grains. The extracted binder was compared with polyvinyl alcohol (PVA) in a coating formulation consisting of 10:1 CaCO₃ to binder formulation coated on light weight paper. Cobb test, dry and wet tensile tests, optical properties (brightness, color, opacity), water vapor transmission rate (WVTR) analysis were performed to compare the hemicellulose based binder with PVA. The results indicate water absorptivity reduction by 25% for both DG gum and PVA coatings from the base paper value. Dry tensile index was lower with higher coating weights for both coatings with similar performance. The performance on WVTR analysis for both coatings was also comparable. No significant differences in optical properties after coating were observed from that of the base paper. These results suggest that hemicellulose based binder extracted from distiller's grains has the potential to replace synthetic binders in paper coating formulations.

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1. Introduction

Depending on the end-use, coatings are applied onto paper to provide the desired properties such as gloss and smoothness in magazines and catalogues, printability and ink-retention in office paper, wear resistance, and water and grease resistance in packaging applications [7]. Coating formulations typically consist of a pigment(s) such as calcium carbonate, clays and others to provide the desired characteristic; a binder such as styrene butadiene, styrene butadiene acrylonitrile, styrene acrylic and vinyl acetate polymers to hold the pigment(s) together; and certain additives to improve processability and handling [10,13]. Coatings contribute about 10% of the overall cost of paper production of which, the binder is the highest cost component in the formulation [7]. In addition to costs, the binders that are currently being used are synthesized from fossil fuel feedstock raising concerns towards the sustainability of paper coatings. Furthermore, papers coated with

waxes, polyolefins and other synthetic materials are difficult to recycle or compost [3].

These challenges inherent with synthetic binders have triggered an interest in using renewable resources for paper coating applications [31]. Bio-polymers have found use in edible coatings for food applications for over a century now, however, their use as coatings and films in paper and packaging is relatively recent [3]. The basic bio-materials used for films and coatings are broadly classified as polysaccharides, lipids, and proteins compounds [15]. Polysaccharide compounds used as films include celluloses, starches, hemicelluloses, chitosan and their derivatives having excellent oil and grease resistant properties [4]. Several proteins including collagen, zein, wheat gluten, cottonseed and soybean proteins have also been researched for films and coatings [23,1]. Water resistivity in fruits and vegetables have been controlled by edible lipids films made from waxes, animal fats and vegetable oils due to their non-polar nature [9].

For this study, we have concentrated our research on polysaccharide coatings due to its abundance, ease of modification and diversity of its sources. One of the most widely used polysaccharides for environmentally friendly coatings is starch. Starches are biodegradable, renewable and inexpensive however, they are

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limited by their poor water vapor resistance and mechanical properties. To overcome these limitations, starches are typically combined with certain plasticizers such as polyhydroxy compounds and/or modified by crosslinking or by incorporating hydrophobic groups to improve film forming and other physical properties [3]. Chitosan, which is a partially deacetylated derivative of chitin is also used as a biodegradable, non-toxic and biocompatible coating material [14]. Coatings prepared from chitosan exhibit low oxygen permeability [6], grease resistance [29], antifungal and antibacterial properties [24], and superior mechanical strength [2]. On the contrary, cellulose by itself has poor film forming characteristics and therefore cellulose derivatives namely cellophane, cellulose esters (e.g. cellulose acetate, cellulose triacetate, and cellulose acetate butyrate), and cellulose ethers (e.g. methylcellulose carboxymethylcellulose, hydroxyethylcellulose, etc.) have been widely synthesized to exhibit low air, vapor, and grease resistance for food packaging applications [25].

Another abundant polysaccharide that has been used widely in coatings is hemicelluloses. Hemicelluloses are one of the three structural molecules in plants along with cellulose and lignin and are found abundantly in agricultural wastes [16]. While cellulose and lignin have homogeneous compositions, hemicelluloses are composed of noncrystalline hexose and pentose sugars. Four main groups of hemicelluloses are defined as xyloglycans (xylan), β -glucans, mannoglycans (mannans), and xyloglycans [11]. Hemicelluloses from various sources such as barley husks, aspen wood, oats, corn hulls, birchwood, grass, corncob and their derivatives have been tested for products ranging from packaging to medical applications to provide oxygen barriers, water and grease resistance, tablet coatings and others [16]. A detailed review on hemicellulose applications and films is provided by Aulin et al. [3] and Hansen et al. [16]. However, these authors and other literature on hemicelluloses have not considered any coating applications from abundant and underutilized source of hemicelluloses that can be obtained from distiller's grains (DG).

Distiller's grains are a byproduct of the ethanol production process and for our research we have focused on DGs from the corn ethanol production process. Current practice involves fermentation of the pretreated corn feedstock and distillation of ethanol from the mixture leaving behind the distiller's grains, which is dried and utilized as animal feed due to its abundance of proteins, and fats [8]. However, various studies have shown that hemicelluloses contained within the DGs have limited digestibility in ruminants [18,20] and higher content of hemicelluloses in the feed have shown to reduce voluntary intake by the animals [21]. Thus separation of hemicelluloses from DG prior to its use as animal feed is likely to improve the feed value and marketability of DG and recover hemicelluloses for alternative applications.

In this study, hemicelluloses from DG were extracted by alkali following the procedure adopted by Xiang et al. [32,33]. The recovered hemicelluloses, henceforth known as distiller's grains gum (DG gum) were characterized for preparing paper coating formulations. Furthermore, the DG gum was chemically modified by succinic anhydride to impart wet and dry strength to the coated paper and improve water repellency. The objective of this research is to evaluate performance of paper coatings formulated with DG gum and modified DG gum as binder and compare with and polyvinyl alcohol (PVA).

2. Materials and methods

All chemicals and reagents were obtained from Sigma Aldrich unless noted otherwise. Precipitated Calcium Carbonate (PCC) was provided by Mineralstech (New York, NY). Distiller's dried grains were provided by Didion Milling (Cambria, WI). Procedure for

extraction of gum from DG was adopted from Xiang et al. [32,33] who performed factorial experiments to optimize the purity of the extracted gum to keep protein content low. Based on those experiments, we extracted DG gum from 2 kg of DG using 3% NaOH solution at 50 °C for 3 h. After extraction, the mixture was centrifuged and the DG gum in the supernatant was precipitated and washed using ethanol. The gum was freeze dried and ground for further use.

2.1. DG gum characterization

DG gum samples were analyzed for carbohydrates following the procedure adopted by [22,22]. Briefly, 100 mg of DG gum was hydrolyzed with 1.5 mL 72% H₂SO₄ at room temperature for 1.5 h. The mixture was then diluted to a 3% H₂SO₄ concentration and autoclaved at 121 °C for 1.5 h. The hydrolysate was separated by centrifugation, which was then analyzed through a Dionex ICS-3000 Ion Chromatography (IC) System. The molecular weight of the DG gum was determined using Gel Permeation Chromatography (GPC). The pre-treatment and analysis procedure was adopted from Xiang et al. [32,33].

2.2. DG gum modification

For DG gum modification, the unmodified gum was dissolved in DI water to make a 5 wt% solution. 0.5 mol/mol arabinoxylan unit amount of succinic anhydride was added to the solution slowly. The mixture was maintained at 30 °C and stirring on a magnetic plate for 1 h. The mixture was then added to 3 volume of 95% ethanol. The precipitate was collected and washed two times with 70% ethanol solution and one time with acetone in order to remove the unreacted succinic anhydride. The collected precipitate was then freeze-dried and ground for further use. A full characterization of the succinylated gum including NMR, FTIR, and carboxyl group titration may be found at Xiang and Runge [34].

2.3. Paper coating

Lightweight coated (LWC) paper (60 g/m²) was selected for the coating experiments. Prior to coating, papers were dried in an oven at 105 °C for 30 min and placed in a desiccator for 24 h. The basis of coating formulation was 100 pph CaCO₃ and 8 pph binder. The formulations were prepared at 10%, 20%, and 40% solids to control the coating grammage. Typical target coating grammage was 8–14 g/m², which corresponded to coating thickness of 6–9 μ m [12]. Coatings were applied manually on a bench top coater using RDS Mayer rod 7, hot air dried for 1 min and further oven dried at 105 °C for 30 min. The oven dried samples were placed in a desiccator for 24 h. After 24 h, the samples were weighed immediately to ascertain the coating grammage. Prior to testing, the samples were conditioned at 50% RH and 23 °C in accordance with TAPPI standard T-402 sp3.

2.4. Paper testing

Scanning Electron Microscopy was performed using Sputter Coater, Hitachi S-3200 SEM to determine the surface structure of the coated samples. Water absorptiveness of coated paper (Cobb Test) was performed in triplicate in accordance with TAPPI standard T-441 om-04 in triplicate. Contact angles were measured at 25 °C using DI water drops by a Dataphysics OCA 15 Optical Contact Angle Measuring System (Dataphysics, Garden City, NY, USA). Three measurements at random places of the tested surface were conducted. Dry and Wet tensile tests were performed using the Lorentzen & Wettre (L&W) Tensile Tester following the procedure outlined in TAPPI Standard T 494 om-06 and TAPPI Standard T 456

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