

Water and grease resistance of paperboard coated with long chain cellulose fatty acid esters using electrostatic powder coating

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ABSTRACT: Fiber-based materials used for foodservice, takeaway, and other packaging applications must typically provide water and grease resistance. Simultaneously, there is growing interest towards bio-based and renewable barrier coatings. In this study we applied thermoplastic long chain fatty acid cellulose esters onto paperboard using electrostatic powder coating to create barrier properties of interest. Electrostatic powder coating provides a water-free process to tackle moisture-induced quality issues and to provide an even coating layer.

Cellulose octanoate and palmitate esters were produced in pilot scale. These were ground into fine particles using liquid nitrogen and applied onto paperboard sheets using an electrostatic powder gun. The loosely packed coatings were fixed and fused onto the paperboard by hot pressing. We characterized the coated sheets for coating thickness, contact angles with water, water absorption, grease resistance (KIT), and olive oil barrier. Two commercial polyethylene powders were used as references.

Our results indicated that the long chain cellulose fatty acid ester coatings were thermoplastic and hydrophobic with contact angles above 100°. Water absorption was similar to the reference coatings. While grease resistance was lower than with the references, the cellulose ester coatings slowed down diffusion of olive oil through the paperboard. Cellulose octanoate ester with a lower melt viscosity already provided smooth coatings after the initial hot pressing step, while in most cases, the second post hot pressing step further improved the barrier properties.

Application: This paper presents a method for creating water and grease resistance utilizing a combination of cellulose grafted with long chain fatty acids and electrostatic powder coating. This approach can be used for different fiber-based packaging materials, especially when tackling moisture-induced quality issues and aiming at even barrier coating layers.

Barrier coatings are widely applied in packaging to protect packed products from permeants, such as moisture, gases, and lipids, throughout various stages such as production, transportation, storage, and retail [1,2]. Barrier coatings for paper and paperboard are dominated today by fossil-based thermoplastic polymers. The market for barrier coated papers alone has been forecasted to rise from US\$5.2 billion to US\$8.2 billion by 2035 [3]. Despite their widespread use, there are growing environmental concerns regarding non-renewable polymers in packaging materials. These concerns have led to an increased interest towards renewable material options, driven not only by environmental concerns of consumers but also by legislation [4]. Innovative bio-based barrier and coating materials can offer safe alternatives, reduce greenhouse gas emissions, and decrease our dependency on fossil-based raw materials [5].

Cellulose, the Earth's most abundant biopolymer, serves as a versatile raw material for various applications, including textiles, paper, food, and cosmetics [6]. Cellulose is a

linear polymer of D-glucose units with hydroxyl groups forming strong hydrogen bonds and van der Waals interactions, which create a resilient microfibril network that gives cellulose its strength and reactivity. Cellulose is, however, hydrophilic and insoluble in typical organic solvents and does not possess thermoplastic properties, which limits its feasibility for various industrial applications for films, coatings, and composites [7]. One solution is chemical modification (e.g., esterification). Whereas short chain cellulose esters with substituent length of no more than six carbon atoms have been used commercially for several decades, longer chain cellulose fatty acid esters (CFAEs) have gained interest more recently, providing hydrophobicity, mechanical strength, and thermoplastic properties for melt processing [8-11].

In this study, we explored electrostatic powder coating of paperboard using CFAEs to create water and oil/grease resistance that is important for various packaging applications. Electrostatic powder coating is a water-free coating process used previously for paper coating [12-15]. In elec-

trostatic powder coating, fine powder particles are charged and guided with an electric field and air to the surface to be coated [16]. A loosely packed layer of powder particles is formed and attached to the surface followed by a thermomechanical treatment step to form the final coating layer. This technology helps to eliminate moisture-induced quality issues and enables even coating layers.

MATERIALS AND METHODS

Materials

The cellulose used for preparing the fatty acid esters was commercial softwood dissolving pulp produced by Domsjö Fabriker AB, Örnsköldsvik, Sweden. We purchased all reagents and solvents from commercial sources in high purity grades and used them as received. The paperboard was a precoated folding boxboard with basis weight of 190 g/m² provided by Kemira (Helsinki, Finland).

We used Coathylene HX 1681 (low-density polyethylene [LDPE]; R1) and NB 5374-F (high-density polyethylene [HDPE]; R2) powders from Axalta Coating Systems (Cologne, Germany) as reference powders in powder coating. These powders had a mean particle size of 10–15 µm and 40–60 µm, respectively, and they were used as received.

Synthesis of cellulose fatty acid esters

Pulp was first treated enzymatically to adjust the molar mass and reactivity prior to esterification [17]. Detailed properties of the pulps are tabulated in **Table I**. We prepared CFAE with two different side chain lengths in pilot scale using octanoyl and palmitoyl fatty acid (C8 and C16, respectively) chlorides. The homogenous esterification of CFAE was conducted with the method presented by Willberg-Keyriläinen et al. [10,18] using a 500-L enamel Büchi reactor (Büchi AG; Uster, Switzerland). In this scale, 10 kg cellulose was dissolved in lithium chloride/*N,N*-dimethylacetamide (LiCl/DMAc) solution (12.5 kg LiCl/250 kg DMAc), and then anhydrous pyridine, 3.6 equivalents to cellulose anhydroglucose unit (AGU), was mixed with the cellulose solution. After this, acyl chloride (C8 or C16, 3.0 equivalents to AGU) was added dropwise to the cellulose mixture. The mixture was stirred for 16 h at 80°C. The obtained CFAE materials were precipitated with water (C8) or ethanol (C16) using Seitz filter unit (Pall Corp.; New York). The C8 CFAE was washed once with water and twice with warm ethanol. The C16 CFAE was washed once with ethanol and twice with warm acetone. Finally, the CFAE materials were dried using vacuum.

Grinding of polymers

The CFAEs were received as coarse powders and flakes. We employed a laboratory electric grain mill grinder (Model XZ-10B, Vevor Ltd.; Shanghai) with liquid nitrogen for 60 s followed by sieving to remove most of the particles with a diameter larger than 50 µm prior to the powder coating tests. These powders were used as such without any additives.

Powder coating

We carried out powder coating with an electrostatic powder gun (Encore LT, Nordson; Westlake, OH, USA) equipped with a sample feeding cup. Powder was transferred from the cup with pressurized air. Powder particles were charged (50 kV) when passing a sharp electrode and guided by both air and the electrostatic forces onto a paperboard sheet backed with a grounded metal plate. The powder coating system is shown in **Fig. 1**. This setup was placed in a powder coating cabin. We adjusted coating thickness by the time used to apply the coating (2, 5, and 10 s). For the reference powders, the application time was 10 s.

The coated paperboard sheets were transferred to an L&W Rapid Dryer (ABB/Lorenzen & Wettre; Kista, Sweden), covered with a siliconized aluminum (Al) foil or a label backing paper, and pressed (~0.8 kPa) for 2 min at 160°C to fuse the coating. Some of the coated sheets with powder deposition time of 2 s and 5 s were also post hot pressed (~3.5 kPa) for an additional 5 min for improved film formation.

Characterization of polymers, powders, and coatings

Quantitative ¹³C nuclear magnetic resonance (NMR) spectra were recorded utilizing a Avance III 500 NMR spectrometer (Bruker; Billerica, MA, USA). The samples were prepared by dissolving the samples in deuterated chloroform with concentration of approximately 100 mg/mL with a small amount of chromium(III)acetylacetonate as a relaxation reagent. The samples were stirred at room temperature overnight to achieve a visually homogenous solution. The spectra were collected with 15000 scans and 3-s delay between the scans.

The melt flow rate (MFR) describing the fluidity of a melted thermoplastic resin was measured with GOETT-FERT mi40 (GOETTFERT; Buchen, Germany) using procedure B (ISO 1133 “Plastics — Determination of the melt

Pulp	Viscosity, mL/g	$M_{w,r}$ kDa	$M_{n,r}$ kDa	Polydispersity
Untreated	490	462	49	9.5
Enzymatic treated	230	271	22	12.1

M_w : molecular weight/mass; kDa: kilodalton; M_n : number-average molecular weight.

I. Properties of pulp before and after enzymatic treatment.



1. Powder feeding cup (left) and powder coating setup (right).

mass-flow rate [MFR] and melt volume-flow rate [MVR] of thermoplastics — Part 2: Method for materials sensitive to time-temperature history and/or moisture”) and a test weight of 2.16 kg. Results are expressed as the mass (grams) extruded in 10 min.

We measured the particle size distribution (PSD) for the CFAE powders with a Mastersizer 3000 particle size analyzer (Malvern Panalytical; Malvern, United Kingdom). Four parallel particle size measurements were performed for the ground and sieved CFAE powders.

Coating thickness was measured using Micrometer Model 51 (Lorenzen & Wettre). The thickness of each coated sample tested for water absorption and oil barrier was measured. The thickness of the uncoated sheet was subtracted from that of the coated samples to obtain the coating thickness.

We measured contact angles with Milli-Q water (MilliporeSigma; Burlington, MA, USA) using the sessile drop method. These tests were carried out using Theta One Attension optical tensiometer with OneAttension Version 4.0.4 software (Biolin Scientific; Gothenburg, Sweden). Five parallel measurements were performed for each sample, and contact angles were analyzed from the contact angle data after 1 min.

Water absorption (TAPPI Standard Test Method T 441 “Water absorptiveness of sized [non-bibulous] paper, paperboard, and corrugated fiberboard [Cobb test]”) was determined by exposing the sample to water at room temperature inside a Cobb test apparatus (Gurley Precision Instruments; Troy, NY, USA) for 30 min. The excess water was removed by pressing the sample between blotting papers. The initial mass of the sample was subtracted from that of the sample exposed to water to obtain the water absorption (g/m^2).

The KIT test (TAPPI T 559 “Grease resistance test for paper and paperboard”) was used to assess the grease resistance of the coated samples. Originally developed for evaluating oil and grease resistance in paper with fluorochemical sizing agents, the test applies drops of reagents (KIT No. 1–12) to the sample. The highest numbered re-

agent that stays on the surface for 15 s without staining the coated sample is recorded as the “KIT number.” Tests were performed using mixtures of castor oil, *n*-heptane, and toluene, with higher solvent content indicating greater aggressiveness. Results are averaged over five measurements for each sample.

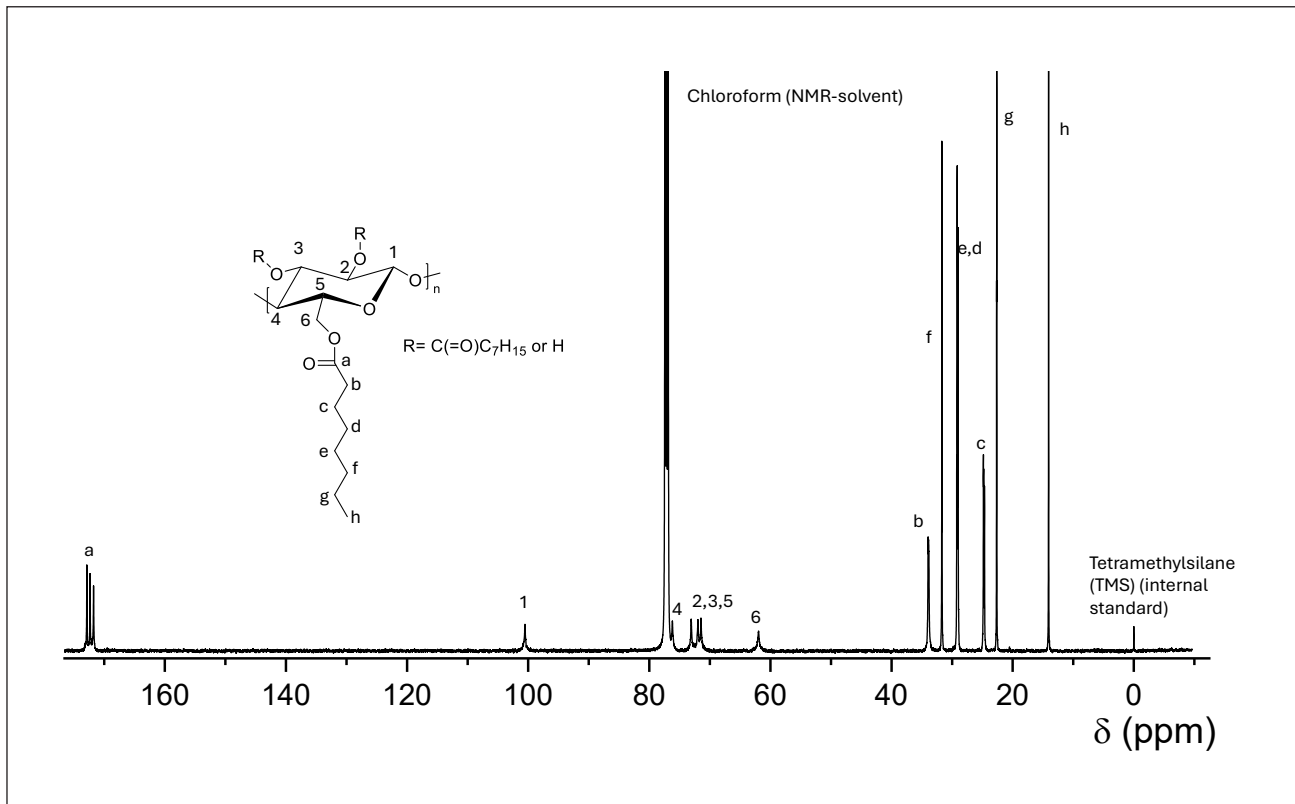
Our oil barrier test was based on measuring the size of the area stained by a red dyed virgin olive oil (Borges, Spain) on the reverse side of the coated sample as a function of time [19]. Five square pieces of each sample were placed on a glass plate with the coated side up. Circular discs of blotting paper were placed onto the samples and saturated with 200 μL of oil. Weights of 50 g were then placed onto the saturated discs. The reverse side of the samples was scanned through the glass plate after preselected time intervals, and the stained area was analyzed from the scanned images. Between the scans, the test setup was kept in a laboratory oven at 40°C. We determined three characteristics from the data:

- Breakthrough time when at least two squares indicate stained area of 2%.
- Average relative area (%) stained by oil after one day of testing.
- Average relative stained area (%) after one week of testing.

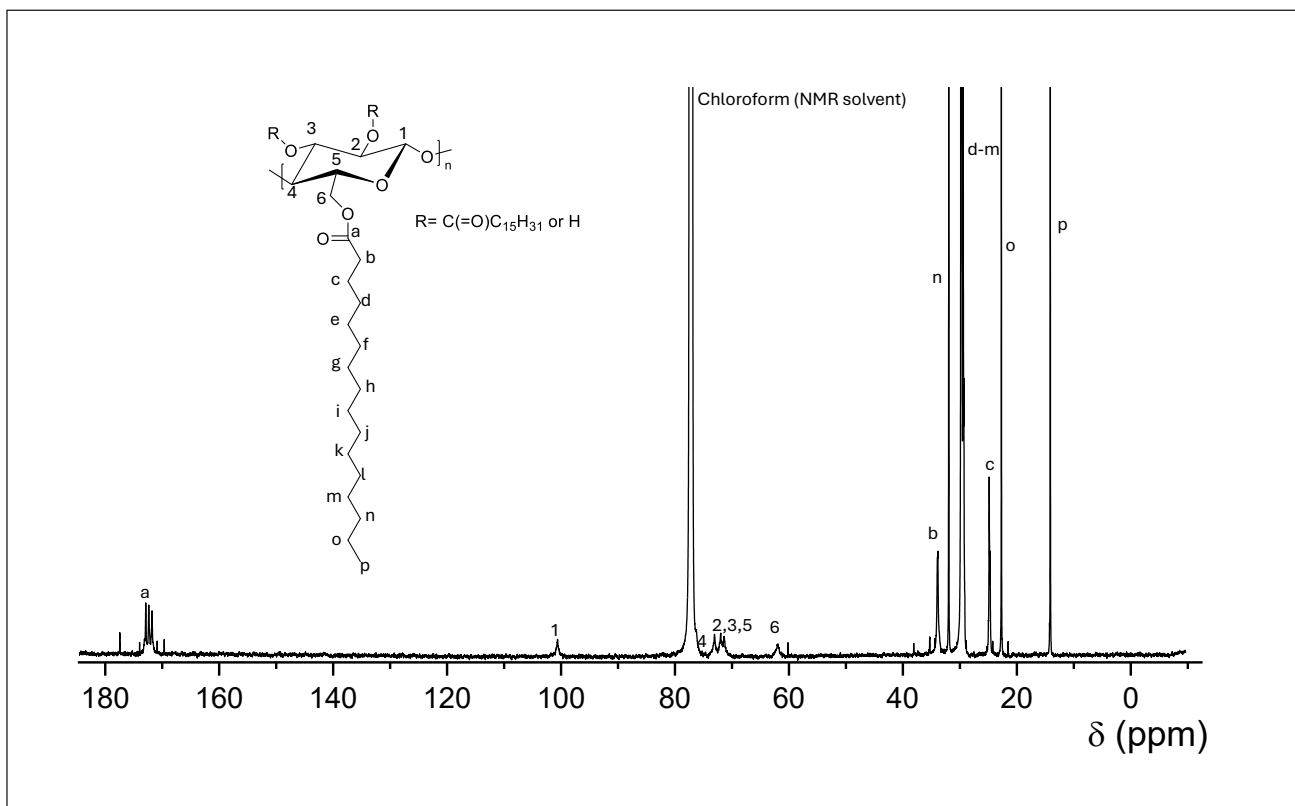
RESULTS

Cellulose ester synthesis

The cellulose esters were synthesized in a pilot scale reactor utilizing homogenous conditions in DMAc/LiCl. Degrees of substitution (DS) of the materials were determined by quantitative ^{13}C NMR (Fig. 2 and Fig. 3). The DS for the cellulose esters were obtained by comparing the integrals of cellulose carbon 1 (labeled “1” in the spectra) and the carbonyl carbon in the ester part (labeled “a” in the spectra). The obtained DS values were 3.0 for both C8 and C16 CFAEs. Thus, the reaction has essentially achieved completion, signaling that making cellulose esters under homogenous conditions provides efficient reactions even with a 1:1 stoichiometric reagent ratio between the cellulose OH groups and the acylating agent.



2. Quantitative ^{13}C nuclear magnetic resonance (NMR) spectrum of the C8 cellulose fatty acid ester (CFAE).



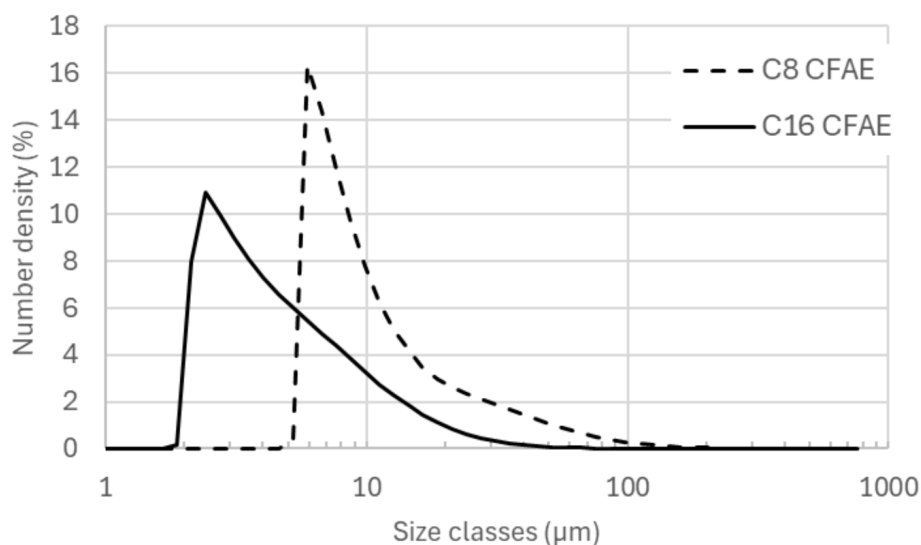
3. Quantitative ^{13}C NMR spectrum of the C16 CFAE.

Powder particle size

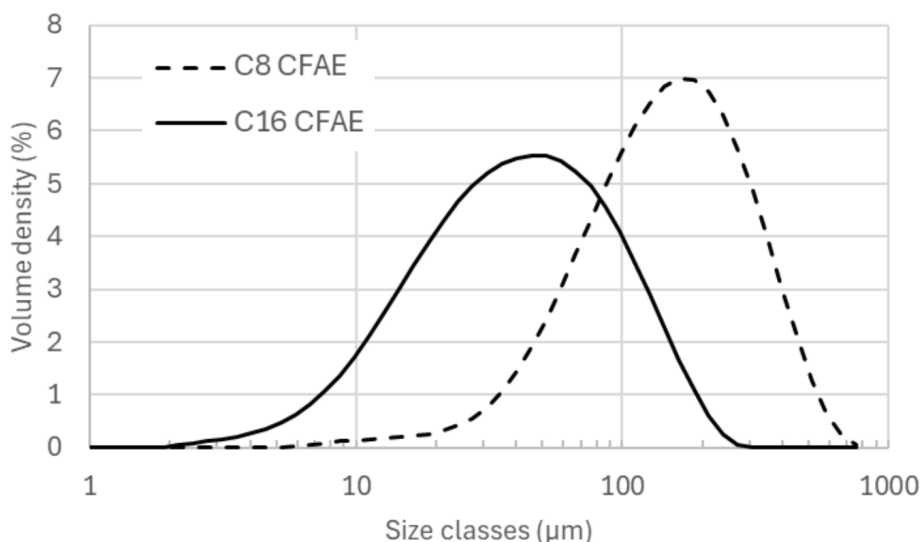
The CFAEs are thermoplastic polymers with the original crystalline structure of cellulose largely destroyed [20]. Ordered phases have been observed in cellulose fatty acid esters, which has been attributed to the packing of the long alkyl substituent chains [21]. There is no sharp melting point, and grinding must therefore be performed at reduced temperature with liquid nitrogen. During the synthesis, the cellulose is molecularly dissolved in DMAc/LiCl, which destroys the cellulose crystalline structure.

An assessment of the particle size distribution (PSD) for the two cellulose ester powders is depicted in **Fig. 4** and

Fig. 5. Overall, the number-based PSD was skewed towards smaller particles, with only a minor number of particles exceeding 100 μm in diameter. Based on the volume-based PSD, a significant part of the polymer was present as larger particle agglomerates, likely formed after the sieving step. Although finer CFAE powders were obtained with other grinding methods not reported here, the cryogrinding method was successful for achieving small enough particles to avoid clogging the powder gun. Ninety-percent of the particles were in the size range of 6–33 μm for C8 CFAE, while for C16 CFAE, the range was 2–13 μm . The number-based average particle size was 9.5 and 4.3 μm , and the



4. Number-based particle size distribution for the C8 and C16 CFAE powders.



5. Volume-based particle size distribution for the C8 and C16 CFAE powders.

Sample	120°C	130°C	140°C
C8 CFAE	51 ± 1	143 ± 2	N.A.
C16 CFAE	4.5 ± 0.3	15 ± 1	57 ± 2

II. Melt flow rate (MFR; g/10 min) for the C8 and C16 CFAEs at different temperatures.

volume-based average particle size was 160 and 43 μm for C8 and C16 CFAE, respectively. As expected, as a more challenging material, the C8 CFAE provided a coarser powder with a wider particle size distribution. A narrow PSD typically improves powder flowability and enables a more uniform charging behavior and a better final coating quality [22-24]. Small particles tend to decrease flowability due to stronger cohesive forces between the particles and carry more charge due to a larger relative surface area. Large particles, on the other hand, are detrimental when aiming at low coating thickness due to a more uneven initial mass distribution on the surface to be coated. It has also been observed that C16 CFAE requires a higher processing temperature compared to C8. Therefore, the smaller particle size of C16 CFAE can be rationalized to arise from a higher temperature resistance.

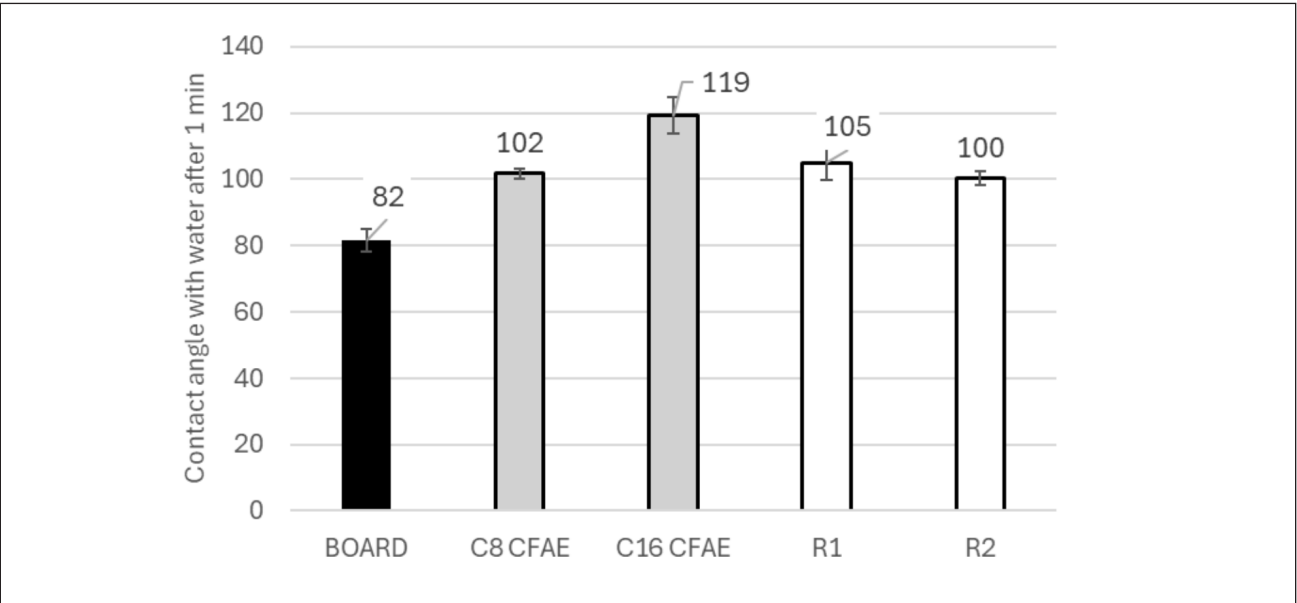
Powder coated sheets

The C8 CFAE powder coated sheets had some unevenly fused areas, but in general, the coating was visually of higher gloss and smoothness compared to C16 CFAE due to a lower melt viscosity (Table II). We did not observe any adhesion related issues with the hot-pressed samples. After

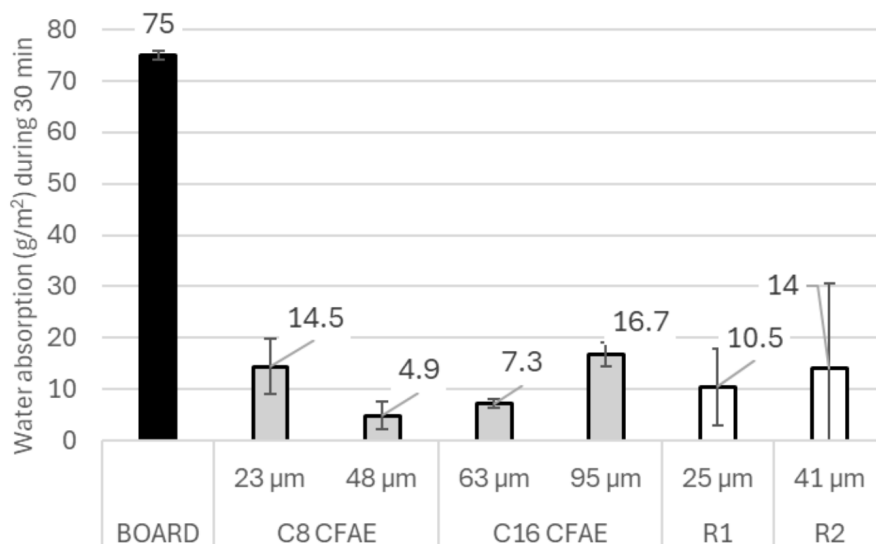
the additional post hot pressing step, the coating quality was improved. Sheets coated with the HDPE reference powder (R2) had stronger curling tendency after hot pressing.

Water contact angles and water absorption

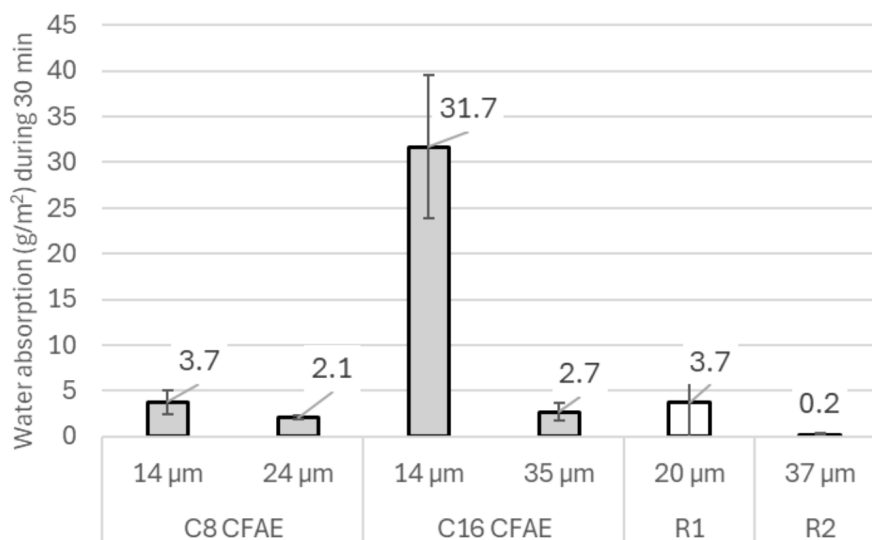
The water contact angles for the powder coated substrates are presented in Fig. 6. Contact angles greater than 90° indicate hydrophobicity. Our measurements showed that both the CFAE and the polyethylene coatings were hydrophobic, which was expected due to the long nonpolar molecules. Additionally, the contact angle for the C16 CFAE coating was higher than that for C8 CFAE, which is attributed to both the longer alkyl substituents and a higher surface roughness before the post hot pressing step. The finding concerning the impact of the substituent length on the water contact angle is consistent with previous research with cellulose fatty acid esters [10,18]. Also, Bras et al. [25] have reported similar contact angles for thermopressed films from cellulose octanoate and palmitate esters. Water absorption for the uncoated and powder coated paperboard before and after the post hot pressing step is presented in Fig. 7 and Fig. 8, respectively. Water absorption was improved with all the powder coatings, and the CFAE coatings were on average similar to the reference polyethylene coatings. The C16 CFAE, however, was less responsive to the initial hot pressing as indicated by an increase in water absorption with an increased coating thickness. A post hot pressing step in most cases improved film formation, and both the coating thickness and water absorption decreased. The most obvious decrease in coating thickness was observed with the C16 CFAE coatings. Water absorption with the C8 CFAE coatings was in the same range as the reference coatings, but with the C16 CFAE, a higher coating



6. Contact angle with water for uncoated and powder coated paperboard (CFAE: cellulose fatty acid ester; R1: low-density polyethylene; R2: high-density polyethylene).



7. Water absorption for uncoated and powder coated paperboard (CFAE: cellulose fatty acid ester; R1: low-density polyethylene; R2: high-density polyethylene).



8. Water absorption for powder coated paperboard after post hot pressing (CFAE: cellulose fatty acid ester; R1: low-density polyethylene; R2: high-density polyethylene).

thickness was required. The negative impact of the post hot pressing with a thin C16 CFAE coating was likely due to a limited lateral flow of a more viscous polymer melt that was not able to block the coating defects, which created a shorter pathway for the water to enter into the base board.

Oil and grease barrier

Cellulose ester coatings slowed down penetration of oil through the paperboard, especially with the C8 CFAE, which was attributed to a better film formation after the initial hot pressing and shorter nonpolar alkyl substituents

compared to the C16 CFAE (**Table III**). The best oil barrier was achieved with the HDPE reference (R2), although both reference coatings had occasional pinholes. Uncoated paperboard was already fully stained with oil after two days of testing. The increase in KIT value with coating thickness was minor for the CFAE coatings. Although widely used, the drawbacks of the KIT test include its subjectivity, volatility of the solvents on the coated surfaces, and possible interactions between the solvents and the coating components compared to different oils [26,27].

Sample	KIT	Oil Barrier			Thickness
		Oil Breakthrough Time	Staining after 1 Day	Staining after 7 Days	
Board	1	Immediately	64 ± 10%	N.A. (100%)	-
C8 CFAE	0	2–3 h	13 ± 3%	30 ± 1%	23 µm
	1	5–23 h	8 ± 5%	23 ± 10%	48 µm
	2	77–145 h	0%	7 ± 7%	93 µm
C16 CFAE	0	0–10 min	11 ± 13%	20 ± 23%	63 µm
	2	0–10 min	12 ± 10%	15 ± 14%	95 µm
R1	8	Immediately	10 ± 14%	21 ± 30%	31 µm
R2	8	> 7 d	0%	0%	51 µm

KIT: grease resistance; CFAE: cellulose fatty acid ester; R1: low-density polyethylene; R2: high-density polyethylene.

III. Oil and grease barrier for uncoated and powder coated paperboard.

Sample	KIT	Oil Barrier			Thickness
		Oil Breakthrough Time	Staining after 1 Day	Staining after 7 Days	
C8 CFAE	2	5–24 h	13 ± 9%	31 ± 10%	15 µm
	3	24–31 h	0%	11 ± 9%	21 µm
C16 CFAE	3	40–50 min	6 ± 8%	16 ± 22%	15 µm
	2	> 7 d	0%	0%	35 µm
R1	10	> 7 d	1 ± 3%	2 ± 4%	21 µm
R2	10	> 7 d	0%	0%	40 µm

KIT: grease resistance; CFAE: cellulose fatty acid ester; R1: low-density polyethylene; R2: high-density polyethylene.

IV. Oil and grease barrier for powder coated paperboard after post hot pressing.

Similar to water absorption, post hot pressing had a positive impact on oil and grease barrier (**Table IV**), especially when taking into account the decreased apparent coating thickness. At low coating thickness, the main observation was a prolonged breakthrough time for the C8 CFAE, but as the coating thickness was increased, the impact of hot pressing on both breakthrough time and staining as a function of time became obvious with both CFAEs. The C16 CFAE coating with a thickness of 35 µm was the best of the CFAEs and comparable to the reference polyethylene powder coatings. The KIT value was also improved, likely due to a more closed coating surface.

CONCLUSIONS

We demonstrated that thermoplastic long chain fatty acid cellulose esters can be processed into powders using cryogrinding, and that these powders can be charged and deposited onto paperboard sheets using electrostatic pow-

der coating. We tested thermoplastic long chain fatty acid cellulose esters with octanoyl and palmitoyl substituents as powder coatings with specific interest in their hydrophobicity and water and grease resistance.

Both long chain cellulose fatty acid esters provided hydrophobic coatings similar to the polyethylene reference powders with water contact angles equal or higher than 100°. Water absorption was in the same range as the polyethylene coatings, and values below 4 g/m² were achieved depending on the type of the cellulose ester, the coating thickness, and the fusing process. Cellulose esters also improved grease and oil resistance, and the best sample provided moderate KIT value of 6 and oil barrier for at least seven days. The final coating properties were affected by the length of the substituents and were indirectly affected by the effect this had on the film formation through powder particle size and polymer melt viscosity during hot pressing used to fuse the coatings.

Our study also indicated possibilities for further improvement in water and grease resistance by optimizing the composition of the cellulose esters, the powder preparation process, and the parameters used for fusing the powder coating.

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ABOUT THE AUTHORS

Due to environmental concerns and legislation, there is an interest around alternatives for petroleum-based plastics and perfluoroalkyl substances (PFAS). Cellulose is an abundant raw material for biobased polymers, which together with a novel coating method such as electrostatic powder coating, could help to create specific barrier properties for fiber-based packaging materials, including molded products.

We have already been working with both fatty acid cellulose esters and powder coating of paper. However, in this study, we combined these technologies for the first time, thus providing a new means for converting fiber-based materials. The main challenges included producing small enough powder particles from the fatty acid esters feasible for powder coating and developing the actual powder coating procedure.

Our results supported the hypothesis that it is possible to achieve both grease and water resistance with biobased materials without using any water in the coating process. This research also provided an insight on how to develop the concept further. The results were in line with our recent work with the long-chain fatty acid cellulose esters, and the barrier properties of interest were comparable to the polyethylene references.

For mills, this water-free approach can be used for different fiber-based packaging materials, especially when tackling moisture-induced quality issues and aiming at even barrier coating layers. As a next step

in this research, process and materials could be tested for coating of molded products. This study also indicated possibilities for further improvement in barrier performance by optimizing the powder formulation and the processes used to prepare and apply the powders.

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