



## (51) International Patent Classification:

*D21H 27/10* (2006.01)      *D21H 19/58* (2006.01)  
*D21H 19/20* (2006.01)      *D21H 19/84* (2006.01)  
*D21H 19/28* (2006.01)      *D21H 21/16* (2006.01)

## (21) International Application Number:

PCT/US2017/067614

## (22) International Filing Date:

20 December 2017 (20.12.2017)

## (25) Filing Language:

English

## (26) Publication Language:

English

## (30) Priority Data:

62/436,615      20 December 2016 (20.12.2016)      US

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(81) Designated States (unless otherwise indicated, for every  
kind of national protection available): AE, AG, AL, AM,  
AO, AT, AU, AZ, BA, BB, BG, BH, BN, BR, BW, BY, BZ,  
CA, CH, CL, CN, CO, CR, CU, CZ, DE, DJ, DK, DM, DO,  
DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, GT, HN,  
HR, HU, ID, IL, IN, IR, IS, JO, JP, KE, KG, KH, KN, KP,  
KR, KW, KZ, LA, LC, LK, LR, LS, LU, LY, MA, MD, ME,  
MG, MK, MN, MW, MX, MY, MZ, NA, NG, NI, NO, NZ,  
OM, PA, PE, PG, PH, PL, PT, QA, RO, RS, RU, RW, SA,  
SC, SD, SE, SG, SK, SL, SM, ST, SV, SY, TH, TJ, TM, TN,  
TR, TT, TZ, UA, UG, US, UZ, VC, VN, ZA, ZM, ZW.

(84) Designated States (unless otherwise indicated, for every  
kind of regional protection available): ARIPO (BW, GH,  
GM, KE, LR, LS, MW, MZ, NA, RW, SD, SL, ST, SZ, TZ,  
UG, ZM, ZW), Eurasian (AM, AZ, BY, KG, KZ, RU, TJ,  
TM), European (AL, AT, BE, BG, CH, CY, CZ, DE, DK,  
EE, ES, FI, FR, GB, GR, HR, HU, IE, IS, IT, LT, LU, LV,  
MC, MK, MT, NL, NO, PL, PT, RO, RS, SE, SI, SK, SM,

TR), OAPI (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW,  
KM, ML, MR, NE, SN, TD, TG).

## Declarations under Rule 4.17:

- as to applicant's entitlement to apply for and be granted a  
patent (Rule 4.17(ii))
- as to the applicant's entitlement to claim the priority of the  
earlier application (Rule 4.17(iii))

## Published:

- with international search report (Art. 21(3))

(54) Title: COATED SUBSTRATES INCLUDING COMPOSTABLE COATINGS AND METHODS FOR PRODUCING THE SAME

(57) Abstract: Embodiments of the present disclosure are directed to coated substrates including a substrate having first and second major surfaces and a compostable coating disposed on at least one of the first and second major surfaces of the substrate. The substrate includes a cellulosic material. The compostable coating includes at least one compostable polymer and a dispersant and may disposed on the substrate at a coat weight less than about 20 grams per square meter. The coated substrate has a grease resistance ranging from about 4 to about 12 as measured in accordance with the TAPPI 559 kit test. In some embodiments, the compostable coating is formed from an aqueous dispersion of the compostable polymer and the dispersant.

- 1 -

## **COATED SUBSTRATES INCLUDING COMPOSTABLE COATINGS AND METHODS FOR PRODUCING THE SAME**

### **CROSS-REFERENCE TO RELATED APPLICATIONS**

**[0001]** This application claims the benefit of U.S. Provisional Application Serial No. 62/436,615 filed December 20, 2016.

### **TECHNICAL FIELD**

**[0002]** Embodiments of the present disclosure generally relate to compostable coated substrates and methods for producing compostable coated substrates. More particularly, embodiments of the present disclosure relate to compostable coated substrates comprising cellulosic substrates coated with compostable coatings including at least one compostable polymer and a dispersant, the compostable coated substrates having improved grease resistance.

### **BACKGROUND**

**[0003]** Aqueous-based polymer dispersions are used in a wide variety of technical applications such as adhesives, binders, sizing agents, sealants, primers, inks, paints, seed or grain coating systems, and paper coatings. Such dispersions are also used in pharmaceutical industries for drug delivery, in medical testing kits, or as additives in cosmetics. One use of such dispersions has been to form barrier coatings on paper, paperboard packaging, plastic films, and containers. However, the synthetic polymers used to form such dispersions and coatings are typically sourced from petroleum. This can sometimes make the polymers difficult to recycle or compost, and, as a result, the polymers may be problematic in landfills where biodegradability or compostability is required.

**[0004]** Moreover, even in cases where compostable or biodegradable polymers are utilized, conventional aqueous based polymer coatings utilize significant non-compostable materials which prevent the resultant polymer coating from being biodegradable. Specifically, these

- 2 -

aqueous based polymer coatings involve dissolving the compostable or biodegradable polymers in volatile organic solvents, coating the dissolved materials onto substrates, and then removing the solvents. However, this generates problems in the handling and disposal of such volatile compounds. These problems included toxicity, flammability, and environmental pollution. In addition, such films can also leave some residual solvent in the coating which can adversely affect and prohibit compliance with food contact regulations.

**[0005]** Alternative efforts for making coatings comprising biodegradable polymers involve melting such polymers and then extruding them onto films. However, this often requires working the polymers at high temperatures which can cause premature degradation of the polymers. Other methods employ extrusion and drawing techniques that were material and energy intensive. Additionally, melt extruded polymer coatings typically yield coat weights which are undesirable for many coating applications, for example, coating on paper or other cellulosic substrates. Depending on the substrate or substrates involved, these limitations of melt extruded polymers may be prohibitive.

#### SUMMARY

**[0006]** Accordingly, there remains a need for aqueous based polymer coating processes which achieve compostable or biodegradable polymers coatings without volatile organic solvents. Moreover, further needs exist for compostable or biodegradable coatings, which achieve desirable lower coat weights while maintaining desirable barrier properties, for example, oil and grease resistant properties. The embodiments of the present disclosure meet these needs.

**[0007]** Various embodiments include a coated substrate including a substrate having first and second major surfaces and a compostable coating disposed on at least one of the first and second major surfaces of the substrate. The substrate includes a cellulosic material. The compostable coating, which has a coat weight of less than about 20 grams per square meters (gsm) on a dry weight basis, includes at least one compostable polymer and a dispersant. The

- 3 -

coated substrate has a grease resistance ranging from about 4 to about 12 as measured in accordance with the TAPPI 559 kit test.

**[0008]** Various embodiments described herein also include a method of producing a compostable coated substrate comprising a substrate and a compostable coating. The method includes producing the compostable coated substrate by applying an aqueous dispersion comprising at least one compostable polymer and water onto the substrate. The substrate may include cellulosic material and the compostable coating may have a coat weight less than or equal to about 20 grams per square meter (gsm) on a dry weight basis. Further, the compostable coated substrate may exhibit a grease resistance ranging from 4 to 12 as measured in accordance with the TAPPI 559 kit test.

**[0009]** Accordingly, various embodiments herein provide processes which are effective to apply aqueous dispersions of compostable polymers to compostable substrates. Further, various embodiments herein provide for compostable coated substrates with oil and grease resistant properties. Other features and will be apparent from the following detailed description and the appended claims.

## DETAILED DESCRIPTION

### *Definitions*

**[0010]** As used herein, a “compostable” substance is a substance that undergoes degradation by biological processes during composting to yield CO<sub>2</sub>, water, inorganic compounds, and biomass at a rate consistent with other known compostable materials and leaves no visible, distinguishable, or toxic residue. The term “composting,” as used in the present disclosure, refers to a managed process that controls the biological decomposition and transformation of materials into humus through aerobic mesophilic and thermophilic degradation. This definition of “compostable” is consistent with the requirements defined in ASTM D6868-11 for “plastics and Polymers as Coatings or Additives with Paper and Other Substrates.” Compostable polymers may refer to polymeric materials that are compostable; also included

within the scope of compostable polymers are copolymers and blends of compostable polymers.

**[0011]** As used herein, a “biodegradable polyester” refers to a polyester that undergoes degradation as a result of the activity of naturally occurring microorganisms, bacteria, archaea, fungi, algae, or combinations thereof. This definition of “biodegradable polyester” is consistent with the definition of biodegradable plastic as defined in ASTM D6868-11.

**[0012]** As used herein, “volatile organic solvents” refers to organic compounds having vapor pressures at ambient temperatures such that at least a portion of the compound evaporates, sublimates, or enters the surrounding atmosphere and is measurable by U.S. Environmental Protection Agency Test Method 24.

**[0013]** As used herein, a dispersion is “hydrolytically stable” if the polymers in the dispersion substantially retain their structure and molecular weight without degrading into monomeric forms or other decomposition materials over a specific period of time while stored at specific temperatures. In the context of the present disclosure, a dispersion is hydrolytically stable if it maintains a loss of solids of no more than 10% over a time period from thirty days to one year when stored at temperatures ranging from ambient conditions to 50°C.

### ***Compostable Coated Substrate***

**[0014]** Embodiments of the present disclosure are directed to a coated substrate including a cellulosic substrate having a compostable coating disposed thereon. In various embodiments, the compostable coating includes at least one compostable polymer and a dispersant.

**[0015]** In various embodiments, the substrate includes first and second major surfaces and comprises cellulosic material. Cellulosic materials may include, but are not limited to, paper materials such as a cellulosic paper, construction paper, kraft paper, art paper, paper board, coated paper, or the like. The cellulosic material may also be, for example, a material, such as a fabric or textile formed from cellulose fibers. In one or more embodiments, the substrate is compostable.

**[0016]** In various embodiments, a compostable coating is disposed on at least one of the first and second major surfaces of the substrate. In other embodiments, the compostable coating is disposed on both the first and second major surfaces of the substrate. The compostable coating of various embodiments may be applied to the substrate as an aqueous-based dispersion of a compostable polymer.

**[0017]** The compostable polymer may be any of the known classes of compostable polymers. For example, the compostable polymer may comprise a biodegradable polyester. In other embodiments, the compostable polymer may be selected from the group consisting of polylactic acid (PLA), polymalate (PMA), polyhydroxyalkanoate (PHA), polycaprolactone (PCL), polyesteramide (PEA), polyhydroxyvalerate (PHV), polyhydroxyhexanoate (PHH), aliphatic copolyesters (PBSA), polyethylene furanoate (PEF), polybutylene succinate (PBS), polyglycolic acid, aliphatic polyaspartic polyurea, soybase polymers, polybutyl styrene (PBS), and mixtures and co-polymers thereof. In a specific embodiment, the dispersant comprises polyvinyl alcohol.

**[0018]** In one or more embodiments, the compostable coating comprises from about 60 wt.% to about 99 wt.% of at least one compostable polymer based on the total dry weight of the compostable coating. In other embodiments, the compostable coating comprises from about 60 wt.% to about 95 wt.%, from about 70 wt.% to about 99 wt.%, from about 75 wt.% to about 95 wt.%, or from about 75 wt.% to about 90 wt.% based on the total dry weight of the compostable coating.

**[0019]** In addition to the compostable polymer, the compostable coating includes at least one dispersant. Without wishing to be bound by theory, the dispersant is believed to act to at least partially encapsulate particles of the compostable polymers to enhance the stability of such particles in the dispersion to prevent particles from agglomerating and settling or floating out of the dispersion. This encapsulation may reduce the interfacial tension between the at least one compostable polymer and the surrounding continuous phase of the dispersion. In some instances, the dispersant may act to form a coating on the surface of the compostable polymer particles. Further without being bound by theory, the dispersant helps facilitate the coating of the aqueous dispersion without other volatile organic solvents.

- 6 -

**[0020]** In one or more embodiments, the dispersant is selected from the group consisting of polyvinyl alcohol, cellulosic polysaccharides, including carboxymethyl cellulose, carboxymethyl hydroxyethyl cellulose, ethyl hydroxyethyl cellulose, hydroxyethyl cellulose, hydroxypropyl methyl cellulose, and methyl cellulose; biopolymer polysaccharides including gellan gum, rhamnan gum, wheilan gum, xanthan gum, guar gum, hydroxypropyl guar gum, and sodium alginate; synthetic hydrocarbon polymers including polyacrylamide and copolymers thereof, polyethylene oxide, polyhydroxyethyl (meth)acrylate, poly(meth)acrylic acid and copolymers thereof, polyolefinic sulfonate copolymers, polyvinylpyrrolidone, copolymers of polystyrene and maleic anhydride, copolymers of polymethylene ether and maleic anhydride; and saponified fatty acids including stearic acid, palmitic acid, oleic acid, linoleic acid, lauric acid, behenic acid, erucic acid, lignoceric acid, nervonic acid, brassidic acid, gadoleic acid, vaccenic acid, caproic acid, lauroic acid, myristoleic acid, elaidic acid, palmitoleic acid, icosanoic acid, myristic acid, octanoic acid, hexanoic acid, butanoic acid, or other long chain carboxylic acids having carbon chain lengths from 12 carbons to 60 carbons and combinations and copolymers thereof. In other embodiments, the dispersant comprises UNICID™ 350, a commercially available blend of long chain carboxylic acids from Baker Hughes Incorporated (Houston, Texas, United States). In a specific embodiment, the dispersant comprises polyvinyl alcohol.

**[0021]** In various embodiments, the compostable coating includes from about 0.1 wt.% to about 3.0 wt.% dispersant based on a total dry weight of the compostable coating. For example, the compostable coating includes from about 0.5 wt.% to about 3.0 wt.%, from about 1.0 wt.% to about 3.0 wt.%, or from about 1.5 wt.% to about 2.5 wt.% dispersant based on the total dry weight of the compostable coating.

**[0022]** In further embodiments, the compostable coating may also include waxes. The wax can be, for example, an animal, plant, mineral, or petroleum wax. Example waxes may include, by way of example and not limitations, an emulsion or micronized wax including a Fischer-Tropsch wax, a carnauba wax, a polyethylene wax, a soy wax, a paraffin wax, a scale wax, a slack wax, other vegetable waxes, or mixtures thereof. The compostable coating may include a single wax or a blend of multiple waxes. Various amounts of wax are contemplated.

For example, the compostable coating may include from 0 to 20 wt.% based on a dry weight of the compostable coating. In a further embodiment, the compostable coating may include from 1 to 15 wt.% based on a dry weight of the compostable coating.

**[0023]** In optional embodiments, other additives may be added to the compostable coating. According to some embodiments, the additives are compostable. However, in some other embodiments, one or more non-compostable additives may be included. In such embodiments, the amount of the non-compostable additives is less than 5 wt.% based on a dry weight of the compostable coating. Additionally, the compostable coating comprises no greater than 1 wt.% of any one non-compostable additive on a dry weight basis. Without being bound by theory, it is believed that limiting the presence of non-compostable additives in the compostable coating to less than 5 wt.% on a dry weight basis will not affect the compostable properties of embodiment coated substrates.

**[0024]** In one or more embodiments, the compostable coating has a coat weight less than or equal to about 20 grams per square meter (gsm) on a dry weight basis. In other embodiments, the compostable coating may have a coat weight less than about 20 gsm, less than or equal to about 15 gsm, less than or equal to about 10 gsm, from about 1 gsm to about 5 gsm, or from about 1 gsm to about 20 gsm on a dry weight basis. Lower coat weights use less material and can be more industrially efficient, but often fail to impart the desired properties to the coated substrate. Surprisingly, using the compostable coating aqueous dispersions, compostable substrates, and methods described herein, the desired properties may be imparted to the coated substrate at relatively low coat weights, for example, less than 20 gsm.

### ***Coating Process***

**[0025]** In a further embodiment, the compostable coating may be applied as an aqueous dispersion onto the cellulosic. In some embodiments, the aqueous dispersion may be substantially free of volatile organic compounds, fluorocarbons, or both. As used herein, “substantially free” means that the aqueous-based dispersion includes less than 0.2 weight percent (wt.%) based on a total weight of the aqueous-based dispersion.



**[0026]** Various embodiments are contemplated for making the aqueous-based dispersion. In one embodiment, the process comprises emulsifying the compostable polymer at a temperature of from between about 25° C to about 170° C by blending together the compostable polymer, water, and a dispersant to form a hydrolytically stable aqueous based dispersion of particles of the compostable polymer. According to one embodiment, the compostable polymer is heated to a temperature above its melting point before blending with water to form an aqueous dispersion. An aqueous solution of the dispersant is then blended into the dispersion.

**[0027]** In one or more such aqueous dispersions, the at least one compostable polymer may have a particle size of from about 0.2  $\mu\text{m}$  to about 50  $\mu\text{m}$ . In other embodiments, the at least one compostable polymer of the aqueous dispersion may have a particle size of from about 0.2  $\mu\text{m}$  to about 30  $\mu\text{m}$ , from about 0.2  $\mu\text{m}$  to about 20  $\mu\text{m}$ , from about 0.2  $\mu\text{m}$  to about 10  $\mu\text{m}$ , from about 0.2  $\mu\text{m}$  to about 5  $\mu\text{m}$ , or from about 0.2  $\mu\text{m}$  to about 1  $\mu\text{m}$ .

**[0028]** Alternatively, the compostable polymer may be provided as a micronized powder, and the blending process carried out at a temperature below the melting point of the compostable polymer. Any suitable mixing apparatus may be utilized including, for example, wet milling, a rotor stator, high pressure homogenization using a cavitation mixer, or ultrasonic mixing. Alternatively, other mixing devices and processes may be used.

**[0029]** In one embodiment, the compostable polymer, water, and dispersant are blended together to form a dispersion, and an aqueous solution optionally containing a rheology modifier is then blended into the dispersion. In one embodiment, a dispersion of the polymer melt, water, and dispersant is formed, and the dispersion is allowed to cool. An aqueous solution of a rheology modifier is blended into the cool dispersion. An optional pH buffering agent may also be added to the dispersion to stabilize it.

**[0030]** The rheology modifier may include various compositions suitable for improving the flow and stability of the aqueous dispersion. In one or more embodiments, the rheology modifier is selected from the group consisting of inorganic clays including bentonite, calcium carbonate, and fumed silica; cellulosic polysaccharides including carboxymethyl cellulose,

- 9 -

carboxymethyl hydroxyethyl cellulose, ethyl hydroxyethyl cellulose, hydroxyethyl cellulose, hydroxypropyl methyl cellulose, methyl cellulose, and nanocrystalline cellulose; synthetic hydrocarbon polymers including polyacrylamide and copolymers thereof, polyethylene oxide, polyhydroxyethyl (meth)acrylate, poly(meth)acrylic acid and copolymers thereof, polyolefinicsulfonate and copolymers thereof, polyvinylpyrrolidone, copolymers of polystyrene and maleic anhydride, copolymers of polymethylene ether and maleic anhydride; biopolymer polysaccharides including gellan gum, rhamsan gum, whelan gum, xanthan gum, guar gum, hydroxypropyl guar gum, and sodium alginate.

**[0031]** In at least one embodiment, the dispersion includes particles of polylactic acid as a compostable polymer, polyvinyl alcohol as a dispersant, and xanthan gum as a rheology modifier. In other embodiments, the dispersion includes particles of polylactic acid as a compostable polymer, polyvinyl alcohol as a dispersant, and carboxymethyl cellulose as a rheology modifier.

**[0032]** Further as stated above, the aqueous dispersion may optionally include one or more pH buffering agents. The optional pH buffering agent may be used to sequester any residual acid monomers present in the compostable polymer composition. It may also aid in maintaining pH stability near a neutral pH of 7.0, thereby minimizing any hydrolytic instability, as described subsequently. In accordance with the present embodiments, the dispersion is hydrolytically stable over a broad pH range of from about 3.0 to about 11.5. Without wishing to be bound by theory, the optional pH buffering agent may also act to create a salt effect, causing the water soluble dispersant, rheology modifier, or both to precipitate onto the surface of the compostable polymer particles, providing additional protection from hydrolysis of the compostable polymer particles. In one embodiment, the pH buffering agent is selected from the group consisting of salts of weak acids or bases or other known buffering agents having  $pK_a$  values of between 6 and 8.

**[0033]** In one embodiment, the compostable coating aqueous dispersion may include from about 10 wt.% to about 60 wt.% of at least one compostable polymer, from about 1.0 wt.% to about 8.0 wt.% of at least one dispersant, from about 0.01 wt.% to about 1.0 wt.% of the rheology modifier, and the balance water, based on the total weight of the aqueous

dispersion. In another embodiment, the aqueous dispersion may include from about 21 wt.% to about 49 wt.% of the compostable polymer, from about 1.0 wt.% to about 8.0 wt.% of the dispersant, and from about 0.01 wt.% to about 1.0 wt.% of the rheology modifier, and the balance water, based on the total weight of the aqueous dispersion. In some embodiments, the dispersion may further include from about 3.0 wt.% to about 15.0 wt.% of a pH buffering agent and from about 0.1 to about 2.0 wt.% of a salt, based on the total weight of the aqueous dispersion. In at least one embodiment, the dispersion may include from about 3.0 wt.% to about 11.5 wt.% of a pH buffering agent and from about 0.1 wt.% to about 0.5 wt.% salt, based on the total weight of the aqueous dispersion.

**[0034]** In an example embodiment, the process for making an aqueous-based hydrolytically stable dispersion of polylactic acid comprises emulsifying the polylactic acid, water, polyvinyl alcohol, and xanthan gum (Keltrol® from CP Keltrol) at a temperature of from about 25 °C to about 170 °C and for a time sufficient to form an aqueous-based dispersion of particles of polylactic acid. The process can be carried out by first blending together the polylactic acid polymer with a first portion of water and polyvinyl alcohol to form a first mixture. The first mixture is then blended with a second portion of water and xanthan gum to form the dispersion.

**[0035]** Various suitable coating methods are contemplated for applying the aqueous dispersion onto the cellulosic substrate. The aqueous dispersion may be applied to cellulosic substrates via coating rod deposition, or spray coating. The dispersion may be applied to a substrate to provide a coated substrate including a functional coating without sacrificing the recyclability, repulpability, or the ability to compost the coated substrates.

### ***Barrier Properties***

**[0036]** Moreover, in various embodiments, the coating may provide water resistance, oil and grease resistance, or both to the substrate. In some embodiments, the coating may provide barrier functionality to the coated substrate, and the coated substrate may exhibit resistance to water, oxygen transmission barrier, or moisture vapor transmission barrier properties. In

various embodiments, barrier functionality for resistance to water can be measured by the Cobb Sizing Test, as defined in ASTM D-3285 (TAPPI T-441).

**[0037]** In various embodiments, the coated substrate exhibits a Cobb Value less than or equal to about 30, less than or equal to about 20, or less than or equal to about 10 after about 2 minutes. In other embodiments, the coated substrate has a grease resistance ranging from 4 to 12 or from 7 to 12, as measured in accordance with the TAPPI 559 kit test. For example, the coated substrate may have a TAPPI 559 kit test rating greater than or equal to 4, greater than or equal to 5, greater than or equal to 6, greater than or equal to 7, greater than or equal to 8, greater than or equal to about 9, greater than or equal to 10, greater than or equal to 11, or 12.

### *Applications*

**[0038]** Without being bound by theory, packages and foodservice items made from the coated substrates of various embodiments may exhibit longer useful life as the coating may aid in protecting the substrates and the contents of the package from the elements within the environments to which these items are exposed. In particular, it is believed that by providing barrier properties, the coating may resist the migration of the elements (including, but not limited to, oil, grease, and water) which can be present in the environment and in the contents of the packages or items. Typically, when exposed to such elements, the substrates used in the construction of such packages or items may degrade, break down, or otherwise change in undesirable ways. However, by protecting the substrates from such migration, the coating may increase the useful life of the package or foodservice item. Moreover, by protecting the package from the environmental elements, the coating may not only help to protect the substrates, but may vicariously help to protect the contents of the package, thereby extending the shelf life of the product.

## TEST METHODS

***Cobb Sizing Test for Water Resistance***

[0039] In the Cobb Sizing Test, a sample of coated substrate to be tested is cut approximately 12.5 x 12.5 centimeter (cm) square. The sample is weighed and placed on a neoprene mat, and a hollow metal cylinder is clamped upon the sample. The metal cylinder has an inside area of 100 cm<sup>2</sup>, 25 cm<sup>2</sup>, or 10 cm<sup>2</sup>. If the sample material is textured, a neoprene gasket is placed between the sample and the cylinder, carefully aligning the inner edges of each. Water is then poured into the test cylinder at an amount of 100 mL for a 100 cm<sup>2</sup> cylinder. Proportionately less liquid is used for smaller cylinders. After pouring the liquid, a timer is started to measure the test period. The test period may vary depending on the sample being tested, but generally is 2 minutes, 15 minutes, or 30 minutes. At fifteen seconds before the expiration of the predetermined test period, the water is quickly poured from the cylinder, using care in not dropping any liquid on the untreated (outside) portion of the test specimen. The cylinder is removed from the sample and the sample is placed with wetted side up on a sheet of blotting paper. At exactly the end of the predetermined test period, a second sheet of blotting paper is placed on top of the sample to remove the surplus liquid by moving a stainless steel roller having a smooth face about 20 cm wide and weighing about 10 kg once forward and once backward over the sample and blotting paper without exerting downward force on the roller. The sample is then folded after removing it from between the blotter sheets and re-weighed. The initial weight of the sample is subtracted from the final weight of the sample and the gain in weight in grams is multiplied by 100 for a 100 cm<sup>2</sup> cylinder to obtain the weight of liquid absorbed in grams per square meter (g/m<sup>2</sup>).

***TAPPI 559 Kit Test for Oil/Grease Resistance***

[0040] Oil and grease resistance may be measured according to TAPPI 559 kit test, also referred to as a 3M Kit test. In the TAPPI 559 kit test, twelve increasingly aggressive oil solutions are prepared using different amounts of castor oil, toluene, and n-Heptane. The mixtures of the kit solutions are provided in Table 1 below. The castor oil used in the TAPPI 559 kit test has a density of 0.969 g/cm<sup>3</sup>.

**Table 1: Compositions of Kit Test Solutions**

Kit No.	Castor Oil* (g)	Toluene (mL)	n-Heptane (mL)
1	969.0	0	0
2	872.1	50	50
3	775.2	100	100
4	678.3	150	150
5	581.4	200	200
6	484.5	250	250
7	387.6	300	300
8	290.7	350	350
9	193.8	400	400
10	96.9	450	450
11	0	500	500
12	0	450	550

**[0041]** In a TAPPI 559 kit test, a drop of a kit solution 1 is released onto the surface of a sample of the coated substrate from a height of about 13 millimeters and wiped off after 15.0 seconds. The surface of the sample is then immediately visually inspected for any darkening spots. Darkening spots indicate penetration of the kit solution into the sample coated substrate due to the presence of pinholes. The presence of darkening spots, and penetration of the kit solution into the sample, results in failure of the sample to pass the test. If the sample passes the test with kit solution 1, then the test is repeated with kit solution 2. Each

- 14 -

time the sample passes the kit test, the next numbered kit solution is used. The test is repeated in the same manner with a higher numbered kit solution, until the highest numbered kit that remains on the surface of the sample for 15 seconds without causing darkening spots on the sample is identified. The highest numbered kit that does not cause failure is identified as the TAPPI 559 kit test rating. In various embodiments, the test may be repeated 5 times and the results averaged for each sample. Accordingly, possible TAPPI 559 kit test ratings may range from 1 to 12, with 12 being the highest TAPPI 559 kit test rating, indicating oil/grease resistance to the most aggressive oil mixture.

#### EXAMPLES

[0042] In order that the embodiments may be more easily understood, reference is made to the following examples which are intended to illustrate embodiments disclosed and described herein. These examples are in no way limiting in scope.

##### Examples 1–5

[0043] Five compostable coating aqueous dispersions were prepared by mixing the components together until uniform in a paddle mixer at room temperature. Compostable coated substrate samples were prepared by passing a size 4 Buschman rod coated in each one of the five different formulations of compostable coating aqueous dispersions over a paper substrate twice, in order to apply two layers of the aqueous dispersion. The second layer was applied before the first layer was allowed to dry. After the coatings were applied to the substrate, there were put in a forced air oven at 105°C for one minute. The samples were then allowed to cool to room temperature before testing. The formulation for the aqueous dispersion used in each sample is provided in Table 2 below. Table 2 also details the dry weight formulations of the corresponding cured coatings. The polylactic acid used is a commercially available polylactic acid sold under the tradename Vercet A1000 from NatureWorks (Minnetonka, Minnesota, United States) and the polyvinyl alcohol used is a commercially available produce sold under the tradename Selvol 203 from the Sekisui Chemical Company (Japan). Wax 1 and Wax 2 are commercially available wax emulsions

produced by Michelman, Inc. (Blue Ash, Ohio, United States). Wax 1 is sold under the tradename Michem® Emulsion 77150 and Wax 2 is sold under the tradename Michem® Emulsion 80939M2.

**Table 2: Compostable Coating Formulations**

	Example 1	Example 2	Example 3	Example 4	Example 5
<b>Weight percentage based on the total weight of the aqueous dispersion (%)</b>					
Polylactic acid	43.875	38.025	33.35	38.025	33.350
Polyvinyl alcohol	1.125	0.975	0.855	0.975	0.855
Wax 1	0	2.3	4.6	0	0
Wax 2	0	0	0	1.00	2.00
Water	54.00	54.50	55.35	57.00	59.40
Total Solids	46.00	45.50	44.65	43.00	40.60
<b>Weight percentage based on the total dry weight of the coating (%)</b>					
PLA	95.01	83.57	74.69	88.43	82.14
Polyvinyl alcohol	2.44	2.14	1.91	2.27	2.11
Wax 1	0.00	5.05	10.30	0.00	0.00
Wax 2	0.00	0.00	0.00	2.33	4.93



Comparative Example A

[0044] Comparative Example A, a commercially available paper cup coated with extruded PLA, was obtained to evaluate the effectiveness of the compostable coated substrates of Examples 1–5.

Comparative Example B

[0045] Comparative Example B was prepared by coating a paper substrate with a non-compostable coating. The non-compostable coating was prepared as an aqueous dispersion by combining water, a styrene-acrylate copolymer, a wax emulsion, and emulsifying agents in a paddle mixer at room temperature until uniform. The coated substrate sample was then prepared by passing a size 4 Buschman rod coated in the prepared aqueous dispersions over a paper substrate twice, in order to apply two layers of the aqueous dispersion. The second layer was applied before the first layer was allowed to dry. After the coating was applied to the substrate, it was put in a forced air oven at 105°C for one minute. The samples were then allowed to cool to room temperature before testing.

[0046] To determine coat weights of all samples, the uncoated substrate was first weighed. Then after the coating was applied and cured, and the coated substrates were weighed again. The difference in coated and uncoated weight of the substrate was calculated in grams per square meter (gsm). All samples were then tested for oil and grease resistance according to TAPPI 559 kit test, as described in greater detail previously. Samples were additionally tested for water resistance using the Cobb Sizing Test, according to ASTM D-3285, also described in greater detail previously. The time interval for the Cobb Sizing Test was two minutes. The coat weight and results for the TAPPI 559 kit test and Cobb Sizing Test are shown in Table 3 for all samples. An uncoated substrate was also tested as a control.

**Table 3: Coat Weight, Oil and Grease Resistance, and Water Resistance**

<b>Sample</b>	<b>Coat Weight (gsm)</b>	<b>TAPPI 559 Kit Test</b>	<b>Cobb Sizing Test</b>
Uncoated Substrate	N/A	0	27
Comparative Example A	30	12	0.4
Comparative Example B	2.8	12	0.4
Example 1	3.8	12	12
Example 2	3.1	10	8
Example 3	3.4	9	8
Example 4	3.8	7	4
Example 5	2.5	9	7

**[0047]** As demonstrated by the data in Table 3, Comparative Example A, which included an extruded PLA coating, had similar values of oil and grease resistance as demonstrated by the TAPPI 559 kit test results of Examples 1–5. However, surprisingly, Examples 1–5 were able to achieve this oil and grease resistance with less than 10% of the coat weight as Comparative Example A. Additionally, the data in Table 3 illustrates that the compostable coatings of Examples 1-5 are able to provide similar oil and grease resistance as the non-compostable coating of Comparative Example B.

**[0048]** Examples 1–5 demonstrate that compostable coatings may be applied at coat weights less than the melt extruded coated substrates and exhibit the same oil and grease resistant properties. However, some applications of the compostable coated substrates described herein

require fine-tuned oil and grease resistance. Embodiments of the present disclosure provide for compostable coated substrates having an oil and grease resistance of from 4 to 12, as measured in accordance with the TAPPI 559 kit test. By varying the concentration and type of dispersant and rheology modifier in the compostable coating aqueous dispersion, the oil and grease resistance of embodiment compostable coated substrates may be tuned to exhibit a particular grease resistance.

**[0049]** Five different compostable coating aqueous dispersions were prepared by mixing the components together in a paddle mixer at room temperature. Compostable coated substrate Examples 6–10 were prepared using the same coating process as Examples 1-5. The samples were then allowed to cool to room temperature before being tested for oil and grease resistance. The dry weight formulations for the aqueous dispersions used in each sample are provided in Table 4 below along with their oil and grease resistance as measured in accordance with a TAPPI 559 kit test. As used in Table 4, polyvinyl alcohol 1 is Selvol™ Polyvinyl Alcohol 203 from Sekisui Specialty Chemicals America, LLC and polyvinyl alcohol 2 is Selvol™ Polyvinyl Alcohol 523 from Sekisui Specialty Chemicals America, LLC. The polylactic acid used is a commercially available polylactic acid sold under the tradename Vercet A1000 from NatureWorks (Minnetonka, Minnesota, United States). Wax 1 and Wax 2 are commercially available wax emulsions produced by Michelman, Inc. (Blue Ash, Ohio, United States). Wax 1 is sold under the tradename Michem® Emulsion 77150 and Wax 2 is sold under the tradename Michem® Emulsion 80939M2.

**Table 4: Oil and Grease Resistance of Coated Substrates with Varying Dispersant Composition**

Sample	Weight percentage based on the total dry weight of the coating (%)					TAPPI 559 Kit Test
	Polylactic Acid	Polyvinyl alcohol 1	Polyvinyl alcohol 2	Wax 1	Wax 2	
Example 6	95	5	0	0	0	0
Example 7	85.5	4.5	0	0	10	3
Example 8	85.5	4.5	0	10	0	5

Example 9	85.5	3.4	1.1	10	0	8
Example 10	85.5	2.75	2.75	10	0	10

**[0050]** As can be seen in Table 4, by adjusting this relationship of additives in the aqueous dispersion, including the type of dispersant used, the oil and grease resistance may be tuned to the level appropriate, depending on the application.

**[0051]** It is noted that terms like “preferably,” “commonly,” and “typically” are not utilized herein to limit the scope of the claimed subject matter or to imply that certain features are critical, essential, or even important to the structure or function of the claimed subject matter. Rather, these terms are merely intended to highlight alternative or additional features that may or may not be utilized in particular embodiments.

**[0052]** For the purposes of describing and defining various embodiments it is noted that the term “device” is utilized herein to represent a combination of components and individual components, regardless of whether the components are combined with other components. For the purposes of describing and defining various embodiments, it is noted that the term “substantially” is utilized herein to represent the inherent degree of uncertainty that may be attributed to any quantitative comparison, value, measurement, or other representation. Unless the meaning is clearly to the contrary, all ranges set forth herein are deemed to be inclusive of all values within the recited range as well as the endpoints.

**[0053]** Having described various embodiments in detail and by reference to specific embodiments thereof, it will be apparent that modifications and variations are possible without departing from the scope of the claimed subject matter. Thus it is intended that the specification cover the modifications and variations of the various embodiments described herein provided such modification and variations come within the scope of the appended claims and their equivalents.

- 20 -

#### CLAIMS

1. A coated substrate comprising:  
a substrate comprising first and second major surfaces, wherein the substrate comprises cellulosic material; and  
a compostable coating comprising at least one compostable polymer and a dispersant, the compostable coating being disposed on at least one of the first and second major surfaces and having a coat weight of less than about 20 grams per square meters (gsm) on a dry weight basis,  
wherein the coated substrate has a grease resistance ranging from 4 to 12 as measured in accordance with the TAPPI 559 kit test.
2. The coated substrate of claim 1, wherein the coated substrate is compostable.
3. The coated substrate of claim 1, wherein the coated substrate comprises paper.
4. The coated substrate of claim 1, wherein the coated substrate exhibits a Cobb Value less than or equal to about 20 after about 2 minutes when measured according to ASTM D-3285.
5. The coated substrate of claim 1, wherein the compostable coating has a coat weight less than or equal to about 10 gsm on a dry weight basis.
6. The coated substrate of claim 1, wherein the compostable coating has a coat weight of from about 1 gsm to about 5 gsm on a dry weight basis.
7. The coated substrate of claim 1, wherein the compostable coating comprises less than 5 wt.% of non-compostable additives on a dry weight basis.
8. The coated substrate of claim 1, wherein the compostable coating comprises no greater than 1 wt.% of any one non-compostable additive on a dry weight basis.

9. The coated substrate of claim 1, wherein the compostable polymer comprises biodegradable polyester.
10. The coated substrate of claim 11, wherein the biodegradable polyester comprises a polymer selected from the group consisting of polylactic acid (PLA), polymalate (PMA), polyhydroxyalkanoate (PHA), polyhydroxybutyrate (PHB), polycaprolactone (PCL), polyesteramide (PEA), polyhydroxyvalerate (PHV), polyhydroxyhexanoate (PHH), aliphatic copolyesters (PBSA), polyethylene furanoate (PEF), polybutylene succinate (PBS), poly(poly sebate) (PPS), and polyglycolic acid.
11. The coated substrate of claim 1, wherein the dispersant is selected from the group consisting of polyvinyl alcohol, polyethylene glycols, cellulosic polysaccharides, biopolymer polysaccharides, synthetic hydrocarbon polymers, saponified fatty acids, acrylic copolymers, polyacrylate ammonium salts, polyether carboxylate polymers, and base-neutralized ethylene acrylic acid copolymers.
12. The coated substrate of claim 1, wherein the compostable coating further comprises a rheology modifier.
13. The coated substrate of claim 14, wherein the rheology modifier is selected from the group consisting of inorganic clays, cellulosic polysaccharides, synthetic hydrocarbon polymers, polyolefinic sulfonate and copolymers thereof, polyvinylpyrrolidone, copolymers of polystyrene and maleic anhydride, copolymers of polymethylene ether and maleic anhydride, biopolymer polysaccharides, acrylic copolymers, polyacrylate ammonium salts, polyether carboxylate polymers, and base-neutralized ethylene acrylic acid copolymers.
14. The coated substrate of claim 14, wherein the compostable coating comprises wax.
15. The coated substrate of claim 1, wherein the coated substrate has a grease resistance ranging from 7 to 12 as measured in accordance with the TAPPI 559 kit test.
16. A product package comprising the coated substrate of claim 1.

17. A method of producing a compostable coated substrate comprising a substrate and a compostable coating, the method comprising:

producing the compostable coated substrate by applying an aqueous dispersion comprising at least one compostable polymer and water onto the substrate which comprises cellulosic material,

wherein the compostable coating comprises a coat weight less than or equal to about 20 grams per square meter (gsm) on a dry weight basis, and

wherein the compostable coated substrate has a grease resistance ranging from 4 to 12 as measured in accordance with the TAPPI 559 kit test.

18. The method of claim 17, wherein the compostable polymer comprises polylactic acid (PLA).

19. The method of claim 17, wherein the coat weight of the compostable coating is less than or equal to about 10 gsm on a dry weight basis.

20. The method of claim 17, wherein the coat weight of the compostable coating is from about 1 gsm to about 5 gsm on a dry weight basis.

21. The method of claim 17, wherein the compostable coated substrate has a grease resistance ranging from 7 to 12 as measured in accordance with the TAPPI 559 kit test.

22. The method of claim 17, wherein the aqueous dispersion comprises at least one dispersant, wherein the dispersant is selected from the group consisting of polyvinyl alcohol, polyethylene glycols, cellulosic polysaccharides, biopolymer polysaccharides, synthetic hydrocarbon polymers, saponified fatty acids, acrylic copolymers, polyacrylate ammonium salts, polyether carboxylate polymers, and base-neutralized ethylene acrylic acid copolymers.

23. The method of claim 17, wherein the compostable polymer is selected from the group consisting of polylactic acid (PLA), polymalate (PMA), polyhydroxyalkanoate (PHA), polyhydroxybutyrate (PHB), polycaprolactone (PCL), polyesteramide (PEA),

polyhydroxyvalerate (PHV), polyhydroxyhexanoate (PHH), aliphatic copolyesters (PBSA), polyethylene furanoate (PEF), polybutylene succinate (PBS), poly(poly sebate) (PPS), and polyglycolic acid.

24. The method of claim 17, wherein the coated substrate exhibits a Cobb Value equal to or less than about 20 after about 2 minutes when measured according to ASTM D-3285.

25. The method of claim 17, wherein the at least one compostable polymer has a particle size of from about 0.2  $\mu\text{m}$  to about 20  $\mu\text{m}$ .

26. A coated substrate comprising:

a substrate comprising first and second major surfaces, wherein the substrate comprises cellulosic material; and

a compostable coating comprising at least one compostable polymer and a dispersant, the compostable coating being disposed on at least one of the first and second major surfaces and having a coat weight of less than about 20 gram per square meters (gsm) on a dry weight basis.

27. The coated substrate of claim 26, wherein the coated substrate comprises paper.

28. The coated substrate of claim 26, wherein the coated substrate exhibits a Cobb Value less than or equal to about 20 after about 2 minutes when measured according to ASTM D-3285.

29. The coated substrate of claim 26, wherein the compostable coating has a coat weight less than or equal to about 10 gsm on a dry weight basis.

30. The coated substrate of claim 26, wherein the compostable coating comprises less than 5 wt.% of non-compostable additives.

31. The coated substrate of claim 26, wherein the compostable polymer is selected from the group consisting of polylactic acid (PLA), polymalate (PMA), polyhydroxyalkanoate (PHA), polyhydroxybutyrate (PHB), polycaprolactone (PCL), polyesteramide (PEA),



- 24 -

polyhydroxyvalerate (PHV), polyhydroxyhexanoate (PHH), aliphatic copolyesters (PBSA), polyethylene furanoate (PEF), polybutylene succinate (PBS), poly(poly sebate) (PPS), and polyglycolic acid.

32. The coated substrate of claim 26, wherein the dispersant is selected from the group consisting of polyvinyl alcohol, polyethylene glycols, cellulosic polysaccharides, biopolymer polysaccharides, synthetic hydrocarbon polymers, saponified fatty acids, acrylic copolymers, polyacrylate ammonium salts, polyether carboxylate polymers, and base-neutralized ethylene acrylic acid copolymers.

## INTERNATIONAL SEARCH REPORT

International application No  
PCT/US2017/067614

## A. CLASSIFICATION OF SUBJECT MATTER

INV. D21H27/10 D21H19/20 D21H19/28 D21H19/58 D21H19/84  
D21H21/16

ADD.

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

D21H B65D

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

EPO-Internal, WPI Data

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	JP 2001 303478 A (TOPPAN FORMS CO LTD) 31 October 2001 (2001-10-31) paragraphs [0001], [0004] - [0007], [0010] - [0023]; claims -----	1-32
A	DE 10 2012 006760 A1 (INOVISCOAT GMBH [DE]) 20 June 2013 (2013-06-20) the whole document -----	1-32
A	US 2005/042443 A1 (MILLER GERALD D [US]) 24 February 2005 (2005-02-24) the whole document -----	1-32
A	EP 1 914 345 A1 (KURARAY EUROPE GMBH [DE]) 23 April 2008 (2008-04-23) paragraph [0027]; claims -----	1-32



Further documents are listed in the continuation of Box C.



See patent family annex.

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Date of the actual completion of the international search

14 March 2018

Date of mailing of the international search report

22/03/2018

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# INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No

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