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(54) Title: FILMS OF POLYMER-OIL COMPOSITIONS

(57) Abstract: Films formed from compositions comprising thermoplastic polymers and oils are disclosed, where the oil is dispersed throughout the thermoplastic polymer. Also disclosed are articles formed from films of these compositions.



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## FILMS OF POLYMER-OIL COMPOSITIONS

## FIELD OF THE INVENTION

The present invention relates to films formed from compositions comprising intimate admixtures of thermoplastic polymers and oils. The present invention also relates to articles  
5 made of these films.

## BACKGROUND OF THE INVENTION

Thermoplastic polymers are used in a wide variety of applications. However, thermoplastic polymers, such as polypropylene and polyethylene pose additional challenges compared to other polymer species, especially with respect to formation of, for example,  
10 fibers. This is because the material and processing requirements for production of fibers are much more stringent than for producing other forms, for example, films. For the production of fibers, polymer melt flow characteristics are more demanding on the material's physical and rheological properties vs other polymer processing methods. Also, the local shear/extensional rate and shear rate are much greater in fiber production than other processes  
15 and, for spinning very fine fibers, small defects, slight inconsistencies, or phase incompatibilities in the melt are not acceptable for a commercially viable process. Moreover, high molecular weight thermoplastic polymers cannot be easily or effectively spun into fine fibers. Given their availability and potential strength improvement, it would be desirable to provide a way to easily and effectively spin such high molecular weight polymers. The use  
20 of high molecular weight polymers is also beneficial for use in film and injection molding applications as it generally improves strength and toughness.

Most thermoplastic polymers, such as polyethylene, polypropylene, and polyethylene terephthalate, are derived from monomers (e.g., ethylene, propylene, and terephthalic acid, respectively) that are obtained from non-renewable, fossil-based resources (e.g., petroleum,  
25 natural gas, and coal). Thus, the price and availability of these resources ultimately have a significant impact on the price of these polymers. As the worldwide price of these resources escalates, so does the price of materials made from these polymers. Furthermore, many consumers display an aversion to purchasing products that are derived solely from petrochemicals, which are non-renewable fossil based resources. In some instances,  
30 consumers are hesitant to purchase products made from non-renewable fossil-based resources. Other consumers may have adverse perceptions about products derived from petrochemicals as being "unnatural" or not environmentally friendly.

Thermoplastic polymers are often incompatible with, or have poor miscibility with additives (e.g., oils, pigments, organic dyes, perfumes, etc.) that might otherwise contribute to a reduced consumption of these polymers in the manufacture of downstream articles.

Heretofore, the art has not effectively addressed how to reduce the amount of thermoplastic

5 polymers derived from non-renewable, fossil-based resources in the manufacture of common articles employing these polymers. Accordingly, it would be desirable to address this

deficiency. Existing art has combined polypropylene with additives, with polypropylene as the minor component to form cellular structures. These cellular structures are the purpose

behind including renewable materials that are later removed or extracted after the structure is

10 formed. U.S. Patent No. 3,093,612 describes the combination of polypropylene with various fatty acids where the fatty acid is removed. The scientific paper *J. Apply. Polym. Sci* 82 (1)

pp. 169-177 (2001) discloses use of diluents on polypropylene for thermally induced phase separation to produce an open and large cellular structure but at low polymer ratio, where the diluent is subsequently removed from the final structure. The scientific paper *J. Apply.*

15 *Polym. Sci* 105 (4) pp. 2000-2007 (2007) produces microporous membranes via thermally induced phase separation with dibutyl phthalate and soy bean oil mixtures, with a minor component of polypropylene. The diluent is removed in the final structure. The scientific

paper *Journal of Membrane Science* 108 (1-2) pp. 25-36 (1995) produces hollow fiber microporous membranes using soy bean oil and polypropylene mixtures, with a minor

20 component of polypropylene and using thermally induced phase separation to produce the desired membrane structure. The diluent is removed in the final structure. In all of these

cases, the diluent as described is removed to produce the final structure. These structures

before the diluent is removed are oily with excessive amounts of diluent to produce very open microporous structures with pore sizes  $> 10\mu\text{m}$ .

25 Thus, a need exists for films from compositions of thermoplastic polymers that allow for use of higher molecular weight and/or decreased non-renewable resource based materials,

and/or incorporation of further additives, such as perfumes and dyes. A still further need is for films from compositions that leave the additive present to deliver renewable materials in

the final product and that can also enable the addition of further additives into the final

30 structure, such as dyes and perfumes, for example.

## SUMMARY OF THE INVENTION

In one aspect, the invention is directed to films having at least one layer of a composition comprising an intimate admixture of a thermoplastic polymer and about 5 wt%

to about 40 wt% of an oil, based upon the total weight of the composition, wherein the oil has a melting point of 25°C or less and a boiling point greater than 160°C. The at least one layer can have a thickness of about 10 µm to about 300 µm. The film can further comprise a second layer, and the second layer can be of a composition as disclosed herein. The second layer can have a thickness of about 10 µm to about 300 µm. The films disclosed herein can have a tensile strength at 10% elongation from about 8 N/mm<sup>2</sup> to about 24 N/mm<sup>2</sup>. The films disclosed herein can have a tensile strength at break from about 20 N/mm<sup>2</sup> to about 60 N/mm<sup>2</sup>.

Further disclosed herein are fluid impervious webs formed from the films as disclosed herein.

The thermoplastic polymer can comprise a polyolefin, a polyester, a polyamide, copolymers thereof, or combinations thereof. . The thermoplastic polymer can be selected from the group consisting of polypropylene, polyethylene, polypropylene co-polymer, polyethylene co-polymer, polyethylene terephthalate, polybutylene terephthalate, polylactic acid, polyhydroxyalkanoates, polyamide-6, polyamide-6,6, and combinations thereof. Polypropylene having a melt flow index of greater than 0.5 g/10 min or of greater than 10 g/10 min can be used. The polypropylene can have a weight average molecular weight of about 20 kDa to about 700 kDa. The thermoplastic polymer can be derived from a renewable bio-based feed stock origin, such as bio polyethylene or bio polypropylene, and/or can be recycled source, such as post consumer use. The oil can be present in the composition in an amount of about 8 wt% to about 30 wt% or about 10 wt% to about 20 wt%, based upon the total weight of the composition. The oil can comprise a lipid, which can be selected from the group consisting of a monoglyceride, diglyceride, triglyceride, fatty acid, fatty alcohol, esterified fatty acid, epoxidized lipid, maleated lipid, hydrogenated lipid, alkyd resin derived from a lipid, sucrose polyester, or combinations thereof. The oil can comprise a mineral oil, such as a linear alkane, a branched alkane, or combinations thereof. The oil can be selected from the group consisting of soy bean oil, epoxidized soy bean oil, maleated soy bean oil, corn oil, cottonseed oil, canola oil, castor oil, coconut oil, coconut seed oil, corn germ oil, fish oil, linseed oil, olive oil, oiticica oil, palm kernel oil, palm oil, palm seed oil, peanut oil, rapeseed oil, safflower oil, sperm oil, sunflower seed oil, tall oil, tung oil, whale oil, triolein, trilinolein, 1-stearo- dilinolein, 1-palmito-dilinolein, lauroleic acid, linoleic acid, linolenic acid, myristoleic acid, oleic acid, palmitoleic acid, 1,2-diacetopalmitin, and combinations thereof.

The oil can be dispersed within the thermoplastic polymer such that the oil has a droplet size of less than 10  $\mu\text{m}$ , less than 5  $\mu\text{m}$ , less than 1  $\mu\text{m}$ , or less than 500 nm within the thermoplastic polymer. The oil can be a renewable material.

The compositions disclosed herein can further comprise an additive. The additive can be oil soluble or oil dispersible. Examples of additives include perfume, dye, pigment, surfactant, nucleating agent, clarifying agent, anti-microbial agent, nanoparticle, antistatic agent, filler, or combination thereof.

In another aspect, provided is a method of making a composition as disclosed herein, the method comprising a) mixing the thermoplastic polymer, in a molten state, with the oil, also in the molten state, to form the admixture; and b) cooling the admixture to a temperature at or less than the solidification temperature of the thermoplastic polymer in 10 seconds or less to form the composition. The method of making a composition can comprise a) melting a thermoplastic polymer to form a molten thermoplastic polymer; b) mixing the molten thermoplastic polymer and oil to form an admixture; and c) cooling the admixture to a temperature at or less than the solidification temperature of the thermoplastic polymer in 10 seconds or less. The mixing can be at a shear rate of greater than  $10\text{ s}^{-1}$ , or about 30 to about  $100\text{ s}^{-1}$ . The admixture can be cooled in 10 seconds or less to a temperature of  $50^{\circ}\text{C}$  or less. The composition can be pelletized. The pelletizing can occur after cooling the admixture or before or simultaneous to cooling the admixture. The composition can be made using an extruder, such as a single- or twin-screw extruder. Alternatively, the method of making a composition can comprise a) melting a thermoplastic polymer to form a molten thermoplastic polymer; b) mixing the molten thermoplastic polymer and a oil to form an admixture; and c) extruding the molten mixture to form the films.

#### DETAILED DESCRIPTION OF THE INVENTION

Films disclosed herein are made from compositions of an intimate admixture of a thermoplastic polymer and an oil. The term “intimate admixture” refers to the physical relationship of the oil and thermoplastic polymer, wherein the oil is dispersed within the thermoplastic polymer. The droplet size of the oil within in the thermoplastic polymer is a parameter that indicates the level of dispersion of the oil within the thermoplastic polymer. The smaller the droplet size, the higher the dispersion of the oil within the thermoplastic polymer, the larger the droplet size the lower the dispersion of the oil within the thermoplastic polymer. As used herein, the term “admixture” refers to the intimate admixture

of the present invention, and not an “admixture” in the more general sense of a standard mixture of materials.

The droplet size of the oil within the thermoplastic polymer is less than 10  $\mu\text{m}$ , and can be less than 5  $\mu\text{m}$ , less than 1  $\mu\text{m}$ , or less than 500 nm. Other contemplated droplet sizes of the oil dispersed within the thermoplastic polymer include less than 9.5  $\mu\text{m}$ , less than 9  $\mu\text{m}$ , less than 8.5  $\mu\text{m}$ , less than 8  $\mu\text{m}$ , less than 7.5  $\mu\text{m}$ , less than 7  $\mu\text{m}$ , less than 6.5  $\mu\text{m}$ , less than 6  $\mu\text{m}$ , less than 5.5  $\mu\text{m}$ , less than 4.5  $\mu\text{m}$ , less than 4  $\mu\text{m}$ , less than 3.5  $\mu\text{m}$ , less than 3  $\mu\text{m}$ , less than 2.5  $\mu\text{m}$ , less than 2  $\mu\text{m}$ , less than 1.5  $\mu\text{m}$ , less than 900 nm, less than 800 nm, less than 700 nm, less than 600 nm, less than 400 nm, less than 300 nm, and less than 200 nm.

The droplet size of the oil can be measured by scanning electron microscopy (SEM) indirectly by measuring a void size in the thermoplastic polymer, after removal of the oil from the composition. Removal of the oil is typically performed prior to SEM imaging due to incompatibility of the oil and the SEM imaging technique. Thus, the void measured by SEM imaging is correlated to the droplet size of the oil in the composition.

One exemplary way to achieve a suitable dispersion of the oil within the thermoplastic polymer is by admixing the thermoplastic polymer, in a molten state, and the oil. The thermoplastic polymer is melted (e.g., exposed to temperatures greater than the thermoplastic polymer's solidification temperature) to provide the molten thermoplastic polymer and mixed with the oil. The thermoplastic polymer can be melted prior to addition of the oil or can be melted in the presence of the oil.

The thermoplastic polymer and oil can be mixed, for example, at a shear rate of greater than  $10\text{s}^{-1}$ . Other contemplated shear rates include greater than 10, about 15 to about 1000, about 20 to about 200, or up to  $500\text{s}^{-1}$ . The higher the shear rate of the mixing, the greater the dispersion of the oil in the composition as disclosed herein. Thus, the dispersion can be controlled by selecting a particular shear rate during formation of the composition.

The oil and molten thermoplastic polymer can be mixed using any mechanical means capable of providing the necessary shear rate to result in a composition as disclosed herein. Non-limiting examples of mechanical means include a mixer, such as a Haake batch mixer, and an extruder (e.g., a single- or twin-screw extruder).

The mixture of molten thermoplastic polymer and oil is then rapidly (e.g., in less than 10 seconds) cooled to a temperature lower than the solidification temperature of the

thermoplastic polymer. The mixture can be cooled to less than 100°C, less than 75°C, less than 50°C, less than 40°C, less than 30°C, less than 20°C, less than 15°C, less than 10°C, or to a temperature of about 0°C to about 30°C, about 0°C to about 20°C, or about 0°C to about 10°C. For example, the mixture can be placed in a low temperature liquid (e.g., the liquid is at or below the temperature to which the mixture is cooled). The liquid can be water.

### **Thermoplastic polymers**

Thermoplastic polymers, as used in the disclosed compositions, are polymers that melt and then, upon cooling, crystallize or harden, but can be re-melted upon further heating. Suitable thermoplastic polymers used herein have a melting temperature (also referred to as solidification temperature) from about 60°C to about 300°C, from about 80°C to about 250°C, or from 100°C to 215°C.

The thermoplastic polymers can be derived from renewable resources or from fossil minerals and oils. The thermoplastic polymers derived from renewable resources are bio-based, for example such as bio produced ethylene and propylene monomers used in the production of polypropylene and polyethylene. These material properties are essentially identical to fossil based product equivalents, except for the presence of carbon-14 in the thermoplastic polymer. Renewable and fossil based thermoplastic polymers can be combined together in the present invention in any ratio, depending on cost and availability. Recycled thermoplastic polymers can also be used, alone or in combination with renewable and/or fossil derived thermoplastic polymers. The recycled thermoplastic polymers can be pre-conditioned to remove any unwanted contaminants prior to compounding or they can be used during the compounding and extrusion process, as well as simply left in the admixture. These contaminants can include trace amounts of other polymers, pulp, pigments, inorganic compounds, organic compounds and other additives typically found in processed polymeric compositions. The contaminants should not negatively impact the final performance properties of the admixture, for example, causing defects in a extruded film.

The molecular weight of the thermoplastic polymer is sufficiently high to enable entanglement between polymer molecules and yet low enough to be melt spinnable. Addition of the oil into the composition allows for compositions containing higher molecular weight thermoplastic polymers to be spun, compared to compositions without an oil. Thus, suitable thermoplastic polymers can have weight average molecular weights of about 1000 kDa or less, about 5 kDa to about 800 kDa, about 10 kDa to about 700 kDa, or about 20 kDa to about

400 kDa. The weight average molecular weight is determined by the specific method for each polymer, but is generally measured using either gel permeation chromatography (GPC) or from solution viscosity measurements. The thermoplastic polymer weight average molecular weight should be determined before addition into the admixture.

5           More specifically, however, the thermoplastic polymers preferably include polyolefins such as polyethylene or copolymers thereof, including low density, high density, linear low density, or ultra low density polyethylenes such that the polyethylene density ranges between 0.90grams per cubic centimeter to 0.97 grams per cubic centimeter, most preferred between 0.92 and 0.95 grams per cubic centimeter. The density of the polyethylene  
10 will is determined by the amount and type of branching and depends on the polymerization technology and comonomer type. Polypropylene and/or polypropylene copolymers, including atactic polypropylene; isotactic polypropylene, syndiotactic polypropylene, and combination thereof can also be used. Polypropylene copolymers, especially ethylene can be used to lower the melting temperature and improve properties. These polypropylene  
15 polymers can be produced using metallocene and Ziegler-Natta catalyst systems. These polypropylene and polyethylene compositions can be combined together to optimize end-use properties. Polybutylene is also a useful polyolefin.

Other suitable polymers include polyamides or copolymers thereof, such as Nylon 6, Nylon 11, Nylon 12, Nylon 46, Nylon 66; polyesters or copolymers thereof, such as maleic  
20 anhydride polypropylene copolymer, polyethylene terephthalate; olefin carboxylic acid copolymers such as ethylene/acrylic acid copolymer, ethylene/maleic acid copolymer, ethylene/methacrylic acid copolymer, ethylene/vinyl acetate copolymers or combinations thereof; polyacrylates, polymethacrylates, and their copolymers such as poly(methyl methacrylates).

25           Other nonlimiting examples of polymers include polycarbonates, polyvinyl acetates, poly(oxymethylene), styrene copolymers, polyacrylates, polymethacrylates, poly(methyl methacrylates), polystyrene/methyl methacrylate copolymers, polyetherimides, polysulfones, or combinations thereof. In some embodiments, thermoplastic polymers include polypropylene, polyethylene, polyamides, polyvinyl alcohol, ethylene acrylic acid, polyolefin  
30 carboxylic acid copolymers, polyesters, and combinations thereof.

Biodegradable thermoplastic polymers also are contemplated for use herein. Biodegradable materials are susceptible to being assimilated by microorganisms, such as



molds, fungi, and bacteria when the biodegradable material is buried in the ground or otherwise contacts the microorganisms (including contact under environmental conditions conducive to the growth of the microorganisms). Suitable biodegradable polymers also include those biodegradable materials which are environmentally-degradable using aerobic or  
5 anaerobic digestion procedures, or by virtue of being exposed to environmental elements such as sunlight, rain, moisture, wind, temperature, and the like. The biodegradable thermoplastic polymers can be used individually or as a combination of biodegradable or non-biodegradable polymers. Biodegradable polymers include polyesters containing aliphatic components. Among the polyesters are ester polycondensates containing aliphatic  
10 constituents and poly(hydroxycarboxylic) acid. The ester polycondensates include diacids/diol aliphatic polyesters such as polybutylene succinate, polybutylene succinate co-adipate, aliphatic/aromatic polyesters such as terpolymers made of butylenes diol, adipic acid and terephthalic acid. The poly(hydroxycarboxylic) acids include lactic acid based homopolymers and copolymers, polyhydroxybutyrate (PHB), or other polyhydroxyalkanoate  
15 homopolymers and copolymers. Such polyhydroxyalkanoates include copolymers of PHB with higher chain length monomers, such as C<sub>6</sub>-C<sub>12</sub>, and higher, polyhydroxyalkanoates, such as those disclosed in U.S. Patent Nos. RE 36,548 and 5,990,271.

An example of a suitable commercially available polylactic acid is NATUREWORKS from Cargill Dow and LACEA from Mitsui Chemical. An example of a suitable  
20 commercially available diacid/diol aliphatic polyester is the polybutylene succinate/adipate copolymers sold as BIONOLLE 1000 and BIONOLLE 3000 from the Showa High Polymer Company, Ltd. (Tokyo, Japan). An example of a suitable commercially available aliphatic/aromatic copolyester is the poly(tetramethylene adipate-co-terephthalate) sold as EASTAR BIO Copolyester from Eastman Chemical or ECOFLEX from BASF.

25 Non-limiting examples of suitable commercially available polypropylene or polypropylene copolymers include Basell Profax PH-835 (a 35 melt flow rate Ziegler-Natta isotactic polypropylene from Lyondell-Basell), Basell Metocene MF-650W (a 500 melt flow rate metallocene isotactic polypropylene from Lyondell-Basell), Polybond 3200 (a 250 melt flow rate maleic anhydride polypropylene copolymer from Crompton), Exxon Achieve 3854  
30 (a 25 melt flow rate metallocene isotactic polypropylene from Exxon-Mobil Chemical), Mosten NB425 (a 25 melt flow rate Ziegler-Natta isotactic polypropylene from Unipetrol), Danimer 27510 (a polyhydroxyalkanoate polypropylene from Danimer Scientific LLC), Dow Aspun 6811A (a 27 melt index polyethylene polypropylene copolymer from Dow Chemical),

and Eastman 9921 (a polyester terephthalic homopolymer with a nominally 0.81 intrinsic viscosity from Eastman Chemical).

The thermoplastic polymer component can be a single polymer species as described above or a blend of two or more thermoplastic polymers as described above.

- 5            If the polymer is polypropylene, the thermoplastic polymer can have a melt flow index of greater than 5 g/10 min, as measured by ASTM D-1238, used for measuring polypropylene. Other contemplated melt flow indices include greater than 10 g/10 min, greater than 20 g/10 min, or about 5 g/10 min to about 50 g/10 min.

### Oils

- 10            An oil, as used in the disclosed composition, is a lipid, mineral oil, or combination thereof, having a melting point of 25°C or less and a boiling point of greater than 160°C. The lipid can be a monoglyceride, diglyceride, triglyceride, fatty acid, fatty alcohol, esterified fatty acid, epoxidized lipid, maleated lipid, hydrogenated lipid, alkyd resin derived from a lipid, sucrose polyester, or combinations thereof. The mineral oil can be a linear alkane, a  
15            branched alkane, or combinations thereof.

Because the oil may contain a distribution of melting temperatures to generate a peak melting temperature, the oil melting temperature is defined as having a peak melting temperature 25°C or below as defined when > 50 weight percent of the oil component melts at or below 25°C. This measurement can be made using a differential scanning calorimeter (DSC), where  
20            the heat of fusion is equated to the weight percent fraction of the oil.

The oil number average molecular weight, as determined by gel permeation chromatography (GPC), should be less than 2 kDa, preferably less than 1.5kDa, still more preferred less than 1.2kDa.

- 25            The amount of oil is determined via gravimetric weight loss method. The solidified mixture is placed, with the narrowest specimen dimension no greater than 1mm, into hexane (or acetone) at a ratio of 1g or mixture per 100g of hexane using a refluxing flask system. First the mixture is weighed before being placed into the reflux flask, and then the hexane and mixtures are heated to 60°C for 20hours. The sample is removed and air dried for 60 minutes and a final weight determined. The equation for calculating the weight percent oil is

- 30            
$$\text{weight \% oil} = ([\text{initial mass} - \text{final mass}] / [\text{initial mass}]) \times 100\%$$

Non-limiting examples of oils contemplated in the compositions disclosed herein include castor oil, coconut oil, coconut seed oil, corn germ oil, cottonseed oil, linseed oil, fish oil, olive oil, oiticica oil, palm kernel oil, palm oil, palm seed oil, peanut oil, cottonseed oil, hempseed oil, rapeseed oil, safflower oil, soybean oil, sperm oil, sunflowerseed oil, tall oil, tung oil, whale oil, and combinations thereof. Preferred oils are corn, soy bean, canola, cottonseed, and palm kernel oil. The preferred oils can be new or processed or recycled oils, such as those used at least once, for example as used in cooking. Non-limiting examples of specific triglycerides include triglycerides such as, for example, triolein, trilinolein, 1-stearo-dilinolein, and 1,2-diacetopalmitin. Coconut oil, palm oil and palm kernel oil all have melting temperatures close to or at 25°C and are classified as oils in the present application. The oils can be from edible plant sources and inedible plant sources. Edible plant sources, for example, include soy bean and corn. Inedible sources include jatropha oil and some variants of rapeseed oil. Other contemplated oils include 1-palmito-dilinolein, lauroleic acid, linoleic acid, linolenic acid, myristoleic acid, oleic acid, palmitoleic acid, and combinations thereof.

The oil can be from a renewable material (e.g., derived from a renewable resource). As used herein, a “renewable resource” is one that is produced by a natural process at a rate comparable to its rate of consumption (e.g., within a 100 year time frame). The resource can be replenished naturally, or via agricultural techniques. Non-limiting examples of renewable resources include plants (e.g., sugar cane, beets, corn, potatoes, citrus fruit, woody plants, lignocellulosics, hemicellulosics, cellulosic waste), animals, fish, bacteria, fungi, and forestry products. These resources can be naturally occurring, hybrids, or genetically engineered organisms. Natural resources such as crude oil, coal, natural gas, and peat, which take longer than 100 years to form, are not considered renewable resources. Mineral oil is viewed as a by-product waste stream of coal, and while not renewable, it can be considered a by-product oil.

The oil, as disclosed herein, is present in the composition at a weight percent of about 5 wt% to about 40 wt%, based upon the total weight of the composition. Other contemplated wt% ranges of the oil include about 8 wt% to about 30 wt%, with a preferred range from about 10 wt% to about 30 wt%, about 10 wt% to about 20 wt%, or about 12 wt% to about 18 wt%, based upon the total weight of the composition. Specific oil wt% contemplated include about 5 wt%, about 6 wt%, about 7 wt%, about 8 wt%, about 9 wt%, about 10 wt%, about 11 wt%, about 12 wt%, about 13 wt%, about 14 wt%, about 15 wt%, about 16 wt%, about 17

wt%, about 18 wt%, about 19 wt%, about 20 wt%, about 21 wt%, about 22 wt%, about 23 wt%, about 24 wt%, about 25 wt%, about 26 wt%, about 27 wt%, about 28 wt%, about 29 wt%, about 30 wt%, about 31 wt%, about 32 wt%, about 33 wt%, about 34 wt%, about 35 wt%, about 36 wt%, about 37 wt%, about 38 wt%, about 39 wt%, and about 40 wt%, based upon the total weight of the composition.

### **Additives**

The compositions disclosed herein can further include an additive. The additive can be dispersed throughout the composition, or can be substantially in the thermoplastic polymer portion of the thermoplastic layer or substantially in the oil portion of the composition. In cases where the additive is in the oil portion of the composition, the additive can be oil soluble or oil dispersible.

Non-limiting examples of classes of additives contemplated in the compositions disclosed herein include perfumes, dyes, pigments, nanoparticles, antistatic agents, fillers, and combinations thereof. The compositions disclosed herein can contain a single additive or a mixture of additives. For example, both a perfume and a colorant (e.g., pigment and/or dye) can be present in the composition. The additive(s), when present, is/are present in a weight percent of about 0.05 wt% to about 20 wt%, or about 0.1 wt% to about 10 wt %. Specifically contemplated weight percentages include about 0.5 wt%, about 0.6 wt%, about 0.7 wt%, about 0.8 wt%, about 0.9 wt%, about 1 wt%, about 1.1 wt%, about 1.2 wt%, about 1.3 wt%, about 1.4 wt%, about 1.5 wt%, about 1.6 wt%, about 1.7 wt%, about 1.8 wt%, about 1.9 wt%, about 2 wt%, about 2.1 wt%, about 2.2 wt%, about 2.3 wt%, about 2.4 wt%, about 2.5 wt%, about 2.6 wt%, about 2.7 wt%, about 2.8 wt%, about 2.9 wt%, about 3 wt%, about 3.1 wt%, about 3.2 wt%, about 3.3 wt%, about 3.4 wt%, about 3.5 wt%, about 3.6 wt%, about 3.7 wt%, about 3.8 wt%, about 3.9 wt%, about 4 wt%, about 4.1 wt%, about 4.2 wt%, about 4.3 wt%, about 4.4 wt%, about 4.5 wt%, about 4.6 wt%, about 4.7 wt%, about 4.8 wt%, about 4.9 wt%, about 5 wt%, about 5.1 wt%, about 5.2 wt%, about 5.3 wt%, about 5.4 wt%, about 5.5 wt%, about 5.6 wt%, about 5.7 wt%, about 5.8 wt%, about 5.9 wt%, about 6 wt%, about 6.1 wt%, about 6.2 wt%, about 6.3 wt%, about 6.4 wt%, about 6.5 wt%, about 6.6 wt%, about 6.7 wt%, about 6.8 wt%, about 6.9 wt%, about 7 wt%, about 7.1 wt%, about 7.2 wt%, about 7.3 wt%, about 7.4 wt%, about 7.5 wt%, about 7.6 wt%, about 7.7 wt%, about 7.8 wt%, about 7.9 wt%, about 8 wt%, about 8.1 wt%, about 8.2 wt%, about 8.3 wt%, about 8.4 wt%, about 8.5 wt%, about 8.6 wt%, about 8.7 wt%, about 8.8 wt%, about 8.9 wt%, about 9 wt%, about

9.1 wt%, about 9.2 wt%, about 9.3 wt%, about 9.4 wt%, about 9.5 wt%, about 9.6 wt%, about 9.7 wt%, about 9.8 wt%, about 9.9 wt%, and about 10 wt%.

As used herein the term “perfume” is used to indicate any odoriferous material that is subsequently released from the composition as disclosed herein. A wide variety of chemicals are known for perfume uses, including materials such as aldehydes, ketones, alcohols, and esters. More commonly, naturally occurring plant and animal oils and exudates including complex mixtures of various chemical components are known for use as perfumes. The perfumes herein can be relatively simple in their compositions or can include highly sophisticated complex mixtures of natural and synthetic chemical components, all chosen to provide any desired odor. Typical perfumes can include, for example, woody/earthy bases containing exotic materials, such as sandalwood, civet and patchouli oil. The perfumes can be of a light floral fragrance (e.g. rose extract, violet extract, and lilac). The perfumes can also be formulated to provide desirable fruity odors, e.g. lime, lemon, and orange. The perfumes delivered in the compositions and articles of the present invention can be selected for an aromatherapy effect, such as providing a relaxing or invigorating mood. As such, any material that exudes a pleasant or otherwise desirable odor can be used as a perfume active in the compositions and articles of the present invention.

A pigment or dye can be inorganic, organic, or a combination thereof. Specific examples of pigments and dyes contemplated include pigment Yellow (C.I. 14), pigment Red (C.I. 48:3), pigment Blue (C.I. 15:4), pigment Black (C.I. 7), and combinations thereof. Specific contemplated dyes include water soluble ink colorants like direct dyes, acid dyes, base dyes, and various solvent soluble dyes. Examples include, but are not limited to, FD&C Blue 1 (C.I. 42090:2), D&C Red 6(C.I. 15850), D&C Red 7(C.I. 15850:1), D&C Red 9(C.I. 15855:1), D&C Red 21(C.I. 45380:2), D&C Red 22(C.I. 45380:3), D&C Red 27(C.I. 45410:1), D&C Red 28(C.I. 45410:2), D&C Red 30(C.I. 73360), D&C Red 33(C.I. 17200), D&C Red 34(C.I. 15880:1), and FD&C Yellow 5(C.I. 19140:1), FD&C Yellow 6(C.I. 15985:1), FD&C Yellow 10(C.I. 47005:1), D&C Orange 5(C.I. 45370:2), and combinations thereof.

Contemplated fillers include, but are not limited to inorganic fillers such as, for example, the oxides of magnesium, aluminum, silicon, and titanium. These materials can be added as inexpensive fillers or processing aides. Other inorganic materials that can function as fillers include hydrous magnesium silicate, titanium dioxide, calcium carbonate, clay, chalk, boron nitride, limestone, diatomaceous earth, mica glass quartz, and ceramics.

Additionally, inorganic salts, including alkali metal salts, alkaline earth metal salts, phosphate salts, can be used. Additionally, alkyd resins can also be added to the composition. Alkyd resins comprise a polyol, a polyacid or anhydride, and/or a fatty acid.

Contemplated surfactants include anionic surfactants, amphoteric surfactants, or a  
5 combination of anionic and amphoteric surfactants, and combinations thereof, such as surfactants disclosed, for example, in U.S. Patent Nos. 3,929,678 and 4,259,217 and in EP 414 549, WO93/08876 and WO93/08874.

Additional contemplated additives include nucleating and clarifying agents for the thermoplastic polymer. Specific examples, suitable for polypropylene, for example, are  
10 benzoic acid and derivatives (e.g. sodium benzoate and lithium benzoate), as well as kaolin, talc and zinc glycerolate. Dibenzlidene sorbitol (DBS) is an example of a clarifying agent that can be used. Other nucleating agents that can be used are organocarboxylic acid salts, sodium phosphate and metal salts (for example aluminum dibenzoate) The nucleating or clarifying agents can be added in ranges from 20 parts per million (20ppm) to 20,000ppm,  
15 more preferred range of 200ppm to 2000ppm and the most preferred range from 1000ppm to 1500ppm. The addition of the nucleating agent can be used to improve the tensile and impact properties of the finished admixture composition.

Contemplated surfactants include anionic surfactants, amphoteric surfactants, or a combination of anionic and amphoteric surfactants, and combinations thereof, such as  
20 surfactants disclosed, for example, in U.S. Patent Nos. 3,929,678 and 4,259,217 and in EP 414 549, WO93/08876 and WO93/08874.

Contemplated nanoparticles include metals, metal oxides, allotropes of carbon, clays, organically modified clays, sulfates, nitrides, hydroxides, oxy/hydroxides, particulate water-insoluble polymers, silicates, phosphates and carbonates. Examples include silicon dioxide,  
25 carbon black, graphite, graphene, fullerenes, expanded graphite, carbon nanotubes, talc, calcium carbonate, bentonite, montmorillonite, kaolin, zinc glycerolate, silica, aluminosilicates, boron nitride, aluminum nitride, barium sulfate, calcium sulfate, antimony oxide, feldspar, mica, nickel, copper, iron, cobalt, steel, gold, silver, platinum, aluminum, wollastonite, aluminum oxide, zirconium oxide, titanium dioxide, cerium oxide, zinc oxide,  
30 magnesium oxide, tin oxide, iron oxides ( $\text{Fe}_2\text{O}_3$ ,  $\text{Fe}_3\text{O}_4$ ) and mixtures thereof. Nanoparticles can increase the strength, thermal stability, and/or abrasion resistance of the compositions disclosed herein, and can give the compositions electric properties.

It is contemplated to add waxes or that some amount of wax is present in the composition. The wax may be unrelated to the lipid present or can be a saturated version of the oil. Regardless of the nature of the wax, it's level should be less than 50 weight percent in relation to the amount of oil present. Non-limiting examples of waxes contemplated in the compositions disclosed herein include beef tallow, castor wax, coconut wax, coconut seed wax, corn germ wax, cottonseed wax, fish wax, linseed wax, olive wax, oiticica wax, palm kernel wax, palm wax, palm seed wax, peanut wax, rapeseed wax, safflower wax, soybean wax, sperm wax, sunflower seed wax, tall wax, tung wax, whale wax, and combinations thereof. Non-limiting examples of specific triglycerides include triglycerides such as, for example, tristearin, tripalmitin, 1,2-dipalmitoolein, 1,3-dipalmitoolein, 1-palmito-3-stearo-2-olein, 1-palmito-2-stearo-3-olein, 2-palmito-1-stearo-3-olein, 1,2-dipalmitolinolein, 1,2-distearo-olein, 1,3-distearo-olein, trimyristin, trilaurin and combinations thereof. Non-limiting examples of specific fatty acids contemplated include capric acid, caproic acid, caprylic acid, lauric acid, myristic acid, palmitic acid, stearic acid, and mixtures thereof. Other specific waxes contemplated include hydrogenated soy bean oil, partially hydrogenated soy bean oil, partially hydrogenated palm kernel oil, and combinations thereof. Inedible waxes from *Jatropha* and rapeseed oil can also be used. The wax can be selected from the group consisting of a hydrogenated plant oil, a partially hydrogenated plant oil, an epoxidized plant oil, a maleated plant oil. Specific examples of such plant oils include soy bean oil, corn oil, canola oil, and palm kernel oil. The amount of wax present can range from 0 weight percent to 40 weight percent of the composition, more preferably from 5 weight percent to 20 weight percent of the composition and most preferably from 8 weight percent to 15 weight percent of the composition.

Specific examples of mineral wax include paraffin (including petrolatum), Montan wax, as well as polyolefin waxes produced from cracking processes, preferentially polyethylene derived waxes. Mineral waxes and plant derived waxes can be combined together. Plant based waxes can be differentiated by their carbon-14 content.

Contemplated anti-static agents include fabric softeners which are known to provide antistatic benefits. For example those fabric softeners that have a fatty acyl group which has an iodine value of above 20, such as N,N-di(tallowoyl-oxy-ethyl)-N,N-dimethyl ammonium methylsulfate.

**Films**

A composition as disclosed herein can be formed into a film and can comprise one of many different configurations, depending on the film properties desired. The properties of the film can be manipulated by varying, for example, the thickness, or in the case of  
5 multilayered films, the number of layers, the chemistry of the layers, i.e., hydrophobic or hydrophilic, and the types of polymers used to form the polymeric layers. The films disclosed herein can have a thickness of less than 300  $\mu\text{m}$ , or can have a thickness of 300  $\mu\text{m}$  or greater. Typically, when films have a thickness of 300  $\mu\text{m}$  or greater, they are referred to as extruded sheets, but it is understood that the films disclosed herein embrace both films  
10 (e.g., with thicknesses less than 300  $\mu\text{m}$ ) and extruded sheets (e.g., with thicknesses of 300  $\mu\text{m}$  or greater).

The films disclosed herein can be multi-layer films. The film can have at least two layers (e.g., a first film layer and a second film layer). The first film layer and the second film layer can be layered adjacent to each other to form the multi-layer film. A multi-layer  
15 film can have at least three layers (e.g., a first film layer, a second film layer and a third film layer). The second film layer can at least partially overlie at least one of an upper surface or a lower surface of the first film layer. The third film layer can at least partially overlie the second film layer such that the second film layer forms a core layer. It is contemplated that multi-layer films can include additional layers (e.g., binding layers, non-permeable layers,  
20 etc.).

It will be appreciated that multi-layer films can comprise from about 2 layers to about 1000 layers; in certain embodiments from about 3 layers to about 200 layers; and in certain embodiments from about 5 layers to about 100 layers.

The films disclosed herein can have a thickness (e.g., caliper) from about 10 microns  
25 to about 200 microns; in certain embodiments a thickness from about 20 microns to about 100 microns; and in certain embodiments a thickness from about 40 microns to about 60 microns. For example, in the case of multi-layer films, each of the film layers can have a thickness less than about 100 microns less than about 50 microns; less than about 10 microns, or about 10 micron to about 300 micron. It will be appreciated that the respective film layers  
30 can have substantially the same or different thicknesses.

Thickness of the films can be evaluated using various techniques, including the methodology set forth in ISO 4593:1993, Plastics – Film and sheeting – Determination of



thickness by mechanical scanning. It will be appreciated that other suitable methods may be available to measure the thickness of the films described herein.

For multi-layer films, each respective layer can be formed from a composition described herein. The selection of compositions used to form the multi-layer film can have  
5 an impact on a number of physical parameters, and as such, can provide improved characteristics such as lower basis weights and higher tensile and seal strengths. Examples of commercial multi-layer films with improved characteristics are described in U.S. Patent No. 7,588,706.

A multi-layer film can include a 3-layer arrangement wherein a first film layer and a  
10 third film layer form the skin layers and a second film layer is formed between the first film layer and the third film layer to form a core layer. The third film layer can be the same or different from the first film layer, such that the third film layer can comprise a composition as described herein. It will be appreciated that similar film layers could be used to form multi-layer films having more than 3 layers. For multi-layer films, it is contemplated having  
15 different concentration of oil in different layers. One embodiment for using multi-layer films is to control the location of the oil. For example, in a 3 layer film, the core layer may contain the oil while the outer layer do not. Alternatively, the inner layer may not contain oil and the outer layers do contain oil.

If incompatible layers are to be adjacent in a multi-layer film, a tie layer is preferably  
20 positioned between them. The purpose of the tie layer is to provide a transition and adequate adhesion between incompatible materials. An adhesive or tie layer is typically used between layers of layers that exhibit delamination when stretched, distorted, or deformed. The delamination can be either microscopic separation or macroscopic separation. In either event, the performance of the film may be compromised by this delamination. Consequently, a tie  
25 layer that exhibits adequate adhesion between the layers is used to limit or eliminate this delamination.

A tie layer is generally useful between incompatible materials. For instance, when a polyolefin and a copoly(ester-ether) are the adjacent layers, a tie layer is generally useful.

The tie layer is chosen according to the nature of the adjacent materials, and is  
30 compatible with and/or identical to one material (e.g. nonpolar and hydrophobic layer) and a reactive group which is compatible or interacts with the second material (e.g. polar and hydrophilic layer).

Suitable backbones for the tie layer include polyethylene (low density - LDPE, linear low density - LLDPE, high density - HDPE, and very low density - VLDPE) and polypropylene.

The reactive group may be a grafting monomer that is grafted to this backbone, and is  
5 or contains at least one alpha- or beta- ethylenically unsaturated carboxylic acid or anhydrides, or a derivative thereof. Examples of such carboxylic acids and anhydrides, which maybe mono-, di-, or polycarboxylic acids, are acrylic acid, methacrylic acid, maleic acid, fumaric acid, itaconic acid, crotonic acid, itaconic anhydride, maleic anhydride, and substituted malic anhydride, e.g. dimethyl maleic anhydride. Examples of derivatives of the  
10 unsaturated acids are salts, amides, imides and esters e.g. mono- and disodium maleate, acrylamide, maleimide, and diethyl fumarate.

A particularly preferred tie layer is a low molecular weight polymer of ethylene with about 0.1 to about 30 weight percent of one or more unsaturated monomers which can be copolymerized with ethylene, e.g., maleic acid, fumaric acid, acrylic acid, methacrylic acid,  
15 vinyl acetate, acrylonitrile, methacrylonitrile, butadiene, carbon monoxide, etc. Preferred are acrylic esters, maleic anhydride, vinyl acetate, and methacrylic acid. Anhydrides are particularly preferred as grafting monomers with maleic anhydride being most preferred.

An exemplary class of materials suitable for use as a tie layer is a class of materials known as anhydride modified ethylene vinyl acetate sold by DuPont under the tradename  
20 Bynel®, e.g., Bynel® 3860. Another material suitable for use as a tie layer is an anhydride modified ethylene methyl acrylate also sold by DuPont under the tradename Bynel®, e.g., Bynel® 2169. Maleic anhydride graft polyolefin polymers suitable for use as tie layers are also available from Elf Atochem North America, Functional Polymers Division, of Philadelphia, PA as Orevac™.

25 Alternatively, a polymer suitable for use as a tie layer material can be incorporated into the composition of one or more of the layers of the films as disclosed herein. By such incorporation, the properties of the various layers are modified so as to improve their compatibility and reduce the risk of delamination.

Other intermediate layers besides tie layers can be used in the multi-layer film  
30 disclosed herein. For example, a layer of a polyolefin composition can be used between two outer layers of a hydrophilic resin to provide additional mechanical strength to the extruded web. Any number of intermediate layers may be used.

Examples of suitable thermoplastic materials for use in forming intermediate layers include polyethylene resins such as low density polyethylene (LDPE), linear low density polyethylene (LLDPE), ethylene vinyl acetate (EVA), ethylene methyl acrylate (EMA), polypropylene, and poly(vinyl chloride). Preferred polymeric layers of this type have  
5 mechanical properties that are substantially equivalent to those described above for the hydrophobic layer.

In addition to being formed from the compositions described herein, the films can further include additional additives. For example, opacifying agents can be added to one or more of the film layers. Such opacifying agents can include iron oxides, carbon black,  
10 aluminum, aluminum oxide, titanium dioxide, talc and combinations thereof. These opacifying agents can comprise about 0.1% to about 5% by weight of the film; and in certain embodiments, the opacifying agents can comprise about 0.3% to about 3% of the film. It will be appreciated that other suitable opacifying agents can be employed and in various concentrations. Examples of opacifying agents are described in U.S. Patent No. 6,653,523.

Furthermore, the films can comprise other additives, such as other polymers materials (e.g., a polypropylene, a polyethylene, a ethylene vinyl acetate, a polymethylpentene any combination thereof, or the like), a filler (e.g., glass, talc, calcium carbonate, or the like), a mold release agent, a flame retardant, an electrically conductive agent, an anti-static agent, a pigment, an antioxidant, an impact modifier, a stabilizer (e.g., a UV absorber), wetting  
20 agents, dyes, a film anti-static agent or any combination thereof. Film antistatic agents include cationic, anionic, and, preferably, nonionic agents. Cationic agents include ammonium, phosphonium and sulphonium cations, with alkyl group substitutions and an associated anion such as chloride, methosulphate, or nitrate. Anionic agents contemplated include alkylsulphonates. Nonionic agents include polyethylene glycols, organic stearates,  
25 organic amides, glycerol monostearate (GMS), alkyl di-ethanolamides, and ethoxylated amines.

### **Properties of the Films**

The films described herein can have enhanced properties, such as higher tensile strengths. The tensile strength of the film measured at 10% elongation can be about 8 N/mm<sup>2</sup>  
30 to about 24 N/mm<sup>2</sup>; or about 10 N/mm<sup>2</sup> to about 15 N/mm<sup>2</sup>. The tensile strength of the film measured at break can be about 20 N/mm<sup>2</sup> to about 60 N/mm<sup>2</sup>; or about 25 N/mm<sup>2</sup> to about 40 N/mm<sup>2</sup>. Such tensile strength measurements are provided in normalized states

Tensile strength can be measure in a variety of ways, including an evaluation of the tensile strength at either 10% elongation or at break. One standard to apply in measuring tensile strength is the methodology set forth in ISO 527-5:2009, Plastics – Determination of tensile properties. In order to apply the methodology of ISO 527-5:2009, a sample size of 25.4 mm (or 1 inch) of a film as disclosed herein is placed under pressure by a clamping mechanism, such that a grip distance of about 50 mm is established. Next, the sample is subject to a testing speed of about 500 mm/min such that sufficient force is placed on the sample to stretch it accordingly. Using various modeling techniques and measuring the displacement of the sample under pressure, a model can be developed calculating the tensile strength associated with the sample of the film. The results of the modeling can then be evaluated pursuant to the parameters set forth in the ISO 527-5:2009 permitting calculation of the tensile strength at both 10% elongation and at break. It will be appreciated that other suitable techniques may be available by which to measure tensile strength of a film.

The films can have a seal strength of about 0.10 N/m to about 2.0 N/m; or about 0.20 N/m to about 1.0 N/m. The seal strength can be measured using a variety of techniques, including the methodology set forth in ISO 527-5:2009. To apply the methodology of ISO 527-5:2009, a sample size of 25.4 mm (or 1 inch) of a film as disclosed herein is prepared, wherein the sample includes a seal extending along the mid-region of the sample. The “seal” can include any region where one edge of the film has been joined with another edge of the same (or different) film. It will be appreciated that this seal can be formed using a variety of suitable techniques (e.g., heat sealing). The sample can then be placed under pressure by a clamping mechanism, such that a grip distance of about 50 mm is established and the seal is placed between the grip distance. Next, the sample is subject to a testing speed pursuant to ISO 527-5:2009 such that sufficient force is placed on the sample to stretch it accordingly. Using various modeling techniques, the seal strength associated with the sample of the multi-layer film can be measured. The results of the modeling can then be evaluated pursuant to the parameters set forth in the ISO 527-5:2009. It will be appreciated that other suitable techniques may be available by which to measure seal strength of a film.

#### **Processes of Making the Compositions as Disclosed herein**

Melt mixing of the polymer and oil: The polymer and oil can be suitably mixed by melting the polymer in the presence of the oil. In the melt state, the polymer and oil are subjected to shear which enables a dispersion of the oil into the polymer. In the melt state, the oil and polymer are significantly more compatible with each other.

The melt mixing of the polymer and oil can be accomplished in a number of different processes, but processes with high shear are preferred to generate the preferred morphology of the composition. The processes can involve traditional thermoplastic polymer processing equipment. The general process order involves adding the polymer to the system, melting the polymer, and then adding the oil. However, the materials can be added in any order, depending on the nature of the specific mixing system.

Haake Batch Mixer: A Haake Batch mixer is a simple mixing system with low amount of shear and mixing. The unit is composed of two mixing screws contained within a heated, fixed volume chamber. The materials are added into the top of the unit as desired. The preferred order is to add the polymer, heat to 20°C to 120°C above the polymer's melting (or solidification) temperature into the chamber first. Once the polymer is melted, the oil can be added and mixed with the molten polymer. The mixture is then mixed in the melt with the two mixing screws for about 5 to about 15 minutes at screw RPM from about 60 to about 120. Once the composition is mixed, the front of the unit is removed and the mixed composition is removed in the molten state. By its design, this system leaves parts of the composition at elevated temperatures before crystallization starts for several minutes. This mixing process provides an intermediate quenching process, where the composition can take about 30 seconds to about 2 minutes to cool down and solidify. Mixture of polypropylene with soy bean oil in the Haake mixture showed that greater than 20wt% of oil lead to incomplete incorporation of the oil in the polypropylene mixture, indicating that higher shear rates can lead to better incorporation of oil and greater amounts of oil able to be incorporated.

Single Screw Extruder: A single screw extruder is a typical process unit used in most molten polymer extrusion. The single screw extruder typically includes a single shaft within a barrel, the shaft and barrel engineered with certain screw elements (e.g., shapes and clearances) to adjust the shearing profile. A typical RPM range for single screw extruder is about 10 to about 120. The single screw extruder design is composed of a feed section, compression section and metering section. In the feed section, using fairly high void volume flights, the polymer is heated and supplied into the compression section, where the melting is completed and the fully molten polymer is sheared. The compression section the void volume between the flights is reduced. In the metering section the polymer the polymer is subjected to its highest shearing amount using low void volume between the flights. For this work, general purpose single screw designs were used. In this unit, a continuous or steady state type of process is achieved where the composition components are introduced at desired

locations, and then subjected to temperatures and shear within target zones. The process can be considered to be a steady state process as the physical nature of the interaction at each location in the single screw process is constant as a function of time. This allows for optimization of the mixing process by enabling a zone-by-zone adjustment of the temperature and shear, where the shear can be changed through the screw elements and/or barrel design or screw speed.

The mixed composition exiting the single screw extruder can then be pelletized via extrusion of the melt into a liquid cooling medium, often water, and then the polymer strand can be cut into small pieces. There are two basic types of molten polymer pelletization process used in polymer processing: strand cutting and underwater pelletization. In strand cutting the composition is rapidly quenched (generally much less than 10 seconds) in the liquid medium then cut into small pieces. In the underwater pelletization process, the molten polymer is cut into small pieces then simultaneously or immediately thereafter placed in the presence of a low temperature liquid which rapidly quenches and crystallizes the polymer. These methods are commonly known and used within the polymer processing industry.

The polymer strands that come from the extruder are rapidly placed into a water bath, most often having a temperature range of 1°C to 50°C (e.g., normally is about room temperature, which is 25°C). An alternate end use for the mixed composition is further processing into the desired structure, for example fiber spinning or injection molding. The single screw extrusion process can provide for a high level of mixing and high quench rate. A single screw extruder also can be used to further process a pelletized composition into fibers and injection molded articles. For example, the fiber single screw extruder can be a 37 mm system with a standard general purpose screw profile and a 30:1 length to diameter ratio.

Twin Screw Extruder: A twin screw extruder is the typical unit used in most molten polymer extrusion, where high intensity mixing is required. The twin screw extruder includes two shafts and an outer barrel. A typical RPM range for twin screw extruder is about 10 to about 1200. The two shafts can be co-rotating or counter rotating and allow for close tolerance, high intensity mixing. In this type of unit, a continuous or steady state type of process is achieved where the composition components are introduced at desired locations along the screws, and subjected to high temperatures and shear within target zones. The process can be considered to be a steady state process as the physical nature of the interaction at each location in the single screw process is constant as a function of time. This allows for

optimization of the mixing process by enabling a zone-by-zone adjustment of the temperature and shear, where the shear can be changed through the screw elements and/or barrel design.

The mixed composition at the end of the twin screw extruder can then be pelletized via extrusion of the melt into a liquid cooling medium, often water, and then the polymer strand is cut into small pieces. There are two basic types of molten polymer pelletization process, strand cutting and underwater pelletization, used in polymer processing. In strand cutting the composition is rapidly quenched (generally much less than 10s) in the liquid medium then cut into small pieces. In the underwater pelletization process, the molten polymer is cut into small pieces then simultaneously or immediately thereafter placed in the presence of a low temperature liquid which rapidly quenches and crystallizes the polymer. An alternate end use for the mixed composition is further processing into the desired structure, for example fiber spinning or injection molding.

Three different screw profiles can be employed using a Baker Perkins CT-25 25mm corotating 40:1 length to diameter ratio system. This specific CT-25 is composed of nine zones where the temperature can be controlled, as well as the die temperature. Four liquid injection sites as also possible, located between zone 1 and 2 (location A), zone 2 and 3 (location B), zone 4 and 5 (location C), and zone 6 and 7 (location D).

The liquid injection location are not directly heated, but indirectly through the adjacent zone temperatures. Locations A, B, C and D can be used to inject the additive. Zone 6 can contain a side feeder for adding additional solids or used for venting. Zone 8 contains a vacuum for removing any residual vapor, as needed.

Two types of regions, conveyance and mixing, are used in the CT-25. In the conveyance region, the materials are heated (including through melting which is done in Zone 1 into Zone 2 if needed) and conveyed along the length of the barrel, under low to moderate shear. The mixing section contains special elements that dramatically increase shear and mixing. The length and location of the mixing sections can be changed as needed to increase and decrease shear as needed.

Two primary types of mixing elements are used for shearing and mixing. The first are kneading blocks and the second are thermal mechanical energy elements. The simple mixing screw has 10.6% of the total screw length using mixing elements composed of kneading blocks in a single set followed by a reversing element. The kneading elements are RKB 45/5/12 (right handed forward kneading block with 45° offset and five lobes at 12mm total

element length), followed by two RKB 45/5/36 (right handed forward kneading block with 45° offset and five lobes at 36mm total element length), that is followed by two RKB 45/5/12 and reversing element 24/12 LH (left handed reversing element 24mm pitch at 12mm total element length).

5           The Simple mixing screw mixing elements are located in zone 7. The Intensive screw is composed of additional mixing sections, four in total. The first section is single set of kneading blocks is a single element of RKB45/5/36 (located in zone 2) followed by conveyance elements into zone 3 where the second mixing zone is located. In the second mixing zone, two RKB 45/5/36 elements are directly followed by four TME 22.5/12  
10 (thermomechanical element with 22.5 teeth per revolution and total element length of 12mm) then two conveyance elements into the third mixing area. The third mixing area, located at the end of zone 4 into zone 5, is composed of three RKB 45/5/36 and a KB45/5/12 LH (left handed forward reversing block with 45° offset and five lobes at 12mm total element length). The material is conveyed through zone 6 into the final mixing area comprising two TME  
15 22.5/12, seven RKB 45/5/12, followed by SE 24/12 LH. The SE 24/12 LH is a reversing element that enables the last mixing zone to be completely filled with polymer and additive, where the intensive mixing takes place. The reversing elements can control the residence time in a given mixing area and are a key contributor to the level of mixing.

          The High Intensity mixing screw is composed of three mixing sections. The first  
20 mixing section is located in zone 3 and is two RKB45/5/36 followed by three TME 22.5/12 and then conveyance into the second mixing section. Prior to the second mixing section three RSE 16/16 (right handed conveyance element with 16mm pitch and 16mm total element length) elements are used to increase pumping into the second mixing region. The second mixing region, located in zone 5, is composed of three RKB 45/5/36 followed by a KB  
25 45/5/12 LH and then a full reversing element SE 24/12 LH. The combination of the SE 16/16 elements in front of the mixing zone and two reversing elements greatly increases the shear and mixing. The third mixing zone is located in zone 7 and is composed of three RKB 45/5/12, followed by two TME 22.5.12 and then three more RKB45/5/12. The third mixing zone is completed with a reversing element SE 24/12 LH.

30           An additional screw element type is a reversing element, which can increase the filling level in that part of the screw and provide better mixing. Twin screw compounding is a mature field. One skilled in the art can consult books for proper mixing and dispersion. These types of screw extruders are well understood in the art and a general description can be



found in: Twin Screw Extrusion 2E: Technology and Principles by James White from Hansen Publications. Although specific examples are given for mixing, many different combination are possible using various element configurations to achieve the needed level of mixing.

### **Properties of Compositions**

5           The compositions as disclosed herein can have one or more of the following properties that provide an advantage over known thermoplastic compositions. These benefits can be present alone or in a combination.

          Shear Viscosity Reduction: Addition of an oil, e.g., SBO, to a thermoplastic polymer, e.g., Basell PH-835, reduces the viscosity of the thermoplastic polymer (here,  
10 polypropylene). Viscosity reduction is a process improvement as it can allow for effectively higher polymer flow rates by having a reduced process pressure (lower shear viscosity), or can allow for an increase in polymer molecular weight, which improves the material strength. Without the presence of the oil, it may not be possible to process the polymer with a high polymer flow rate at existing process conditions in a suitable way.

15           Sustainable Content: Inclusion of sustainable materials into the existing polymeric system is a strongly desired property. Materials that can be replaced every year through natural growth cycles contribute to overall lower environmental impact and are desired.

          Pigmentation: Adding pigments to polymers often involves using expensive inorganic compounds that are particles within the polymer matrix. These particles are often  
20 large and can interfere in the processing of the composition. Using an oil as disclosed herein, because of the fine dispersion (as measured by droplet size) and uniform distribution throughout the thermoplastic polymer allows for coloration, such as via traditional ink compounds. Soy ink is widely used in paper publication) that does not impact processability.

          Fragrance: Because the oils, for example SBO, can contain perfumes much more  
25 preferentially than the base thermoplastic polymer, the present composition can be used to contain scents that are beneficial for end-use. Many scented candles are made using SBO based or paraffin based materials, so incorporation of these into the polymer for the final composition is useful.

          Morphology: The benefits are delivered via the morphology produced in production  
30 of the compositions. The morphology is produced by a combination of intensive mixing and rapid crystallization. The intensive mixing comes from the compounding process used and

rapid crystallization comes from the cooling process used. High intensity mixing is desired and rapid crystallization is used to preserve the fine pore size and relatively uniform pore size distribution.

### **Method of Making Films**

5           The film as disclosed herein can be processed using conventional procedures for producing films on conventional coextruded film-making equipment. In general, polymers can be melt processed into films using either cast or blown film extrusion methods both of which are described in *Plastics Extrusion Technology*—2nd Ed., by Allan A. Griff (Van Nostrand Reinhold—1976).

10           Cast film is extruded through a linear slot die. Generally, the flat web is cooled on a large moving polished metal roll (chill roll). It quickly cools, and peels off the first roll, passes over one or more auxiliary rolls, then through a set of rubber-coated pull or "haul-off" rolls, and finally to a winder.

          In blown film extrusion, the melt is extruded upward through a thin annular die  
15   opening. This process is also referred to as tubular film extrusion. Air is introduced through the center of the die to inflate the tube and causes it to expand. A moving bubble is thus formed which is held at constant size by simultaneous control of internal air pressure, extrusion rate, and haul-off speed. The tube of film is cooled by air blown through one or more chill rings surrounding the tube. The tube is next collapsed by drawing it into a  
20   flattened frame through a pair of pull rolls and into a winder.

          A coextrusion process requires more than one extruder and either a coextrusion feedblock or a multi-manifold die system or combination of the two to achieve a multilayer film structure. U.S. Patent Nos. 4,152,387 and 4,197,069, incorporated herein by reference, disclose the feedblock and multi-manifold die principle of coextrusion. Multiple extruders are  
25   connected to the feedblock which can employ moveable flow dividers to proportionally change the geometry of each individual flow channel in direct relation to the volume of polymer passing through the flow channels. The flow channels are designed such that, at their point of confluence, the materials flow together at the same velocities and pressure, minimizing interfacial stress and flow instabilities. Once the materials are joined in the  
30   feedblock, they flow into a single manifold die as a composite structure. Other examples of feedblock and die systems are disclosed in *Extrusion Dies for Plastics and Rubber*, W. Michaeli, Hanser, New York, 2nd Ed., 1992, hereby incorporated herein by reference. It may

be important in such processes that the melt viscosities, normal stress differences, and melt temperatures of the material do not differ too greatly. Otherwise, layer encapsulation or flow instabilities may result in the die leading to poor control of layer thickness distribution and defects from non-planar interfaces (e.g. fish eye) in the multilayer film.

5           An alternative to feedblock coextrusion is a multi-manifold or vane die as disclosed in U.S. Patent Nos. 4,152,387, 4,197,069, and 4,533,308, incorporated herein by reference.

Whereas in the feedblock system melt streams are brought together outside and prior to entering the die body, in a multi-manifold or vane die each melt stream has its own manifold in the die where the polymers spread independently in their respective manifolds. The melt  
10 streams are married near the die exit with each melt stream at full die width. Moveable vanes provide adjustability of the exit of each flow channel in direct proportion to the volume of material flowing through it, allowing the melts to flow together at the same velocity, pressure, and desired width.

Since the melt flow properties and melt temperatures of polymers vary widely, use of  
15 a vane die has several advantages. The die lends itself toward thermal isolation characteristics wherein polymers of greatly differing melt temperatures, for example up to 175° F (80° C), can be processed together.

Each manifold in a vane die can be designed and tailored to a specific polymer. Thus the flow of each polymer is influenced only by the design of its manifold, and not forces  
20 imposed by other polymers. This allows materials with greatly differing melt viscosities to be coextruded into multilayer films. In addition, the vane die also provides the ability to tailor the width of individual manifolds, such that an internal layer can be completely surrounded by the outer layer leaving no exposed edges. The feedblock systems and vane dies can be used to achieve more complex multilayer structures.

25           One of skill in the art will recognize that the size of an extruder used to produce the films as disclosed herein depends on the desired production rate and that several sizes of extruders may be used. Suitable examples include extruders having a 1 inch (2.5 cm) to 1.5 inch (3.7 cm) diameter with a length/diameter ratio of 24 or 30. If required by greater production demands, the extruder diameter can range upwards. For example, extruders  
30 having a diameter between about 2.5 inches (6.4 cm) and about 4 inches (10 cm) can be used to produce the films of the present invention. A general purpose screw may be used. A suitable feedblock is a single temperature zone, fixed plate block. The distribution plate is

machined to provide specific layer thicknesses. For example, for a three layer film, the plate provides layers in an 80/10/10 thickness arrangement, a suitable die is a single temperature zone flat die with "flex-lip" die gap adjustment. The die gap is typically adjusted to be less than 0.020 inches (0.5 mm) and each segment is adjusted to provide for uniform thickness  
5 across the web. Any size die may be used as production needs may require, however, 10–14 inch (25–35 cm) dies have been found to be suitable. The chill roll is typically water-cooled. Edge pinning is generally used and occasionally an air knife may be employed.

For some coextruded films, the placement of a tacky hydrophilic material onto the chill roll may be necessary. When the arrangement places the tacky material onto the chill  
10 roll, release paper may be fed between the die and the chill roll to minimize contact of the tacky material with the rolls. However, a preferred arrangement is to extrude the tacky material on the side away from the chill roll. This arrangement generally avoids sticking material onto the chill roll. An extra stripping roll placed above the chill roll may also assist the removal of tacky material and also can provide for additional residence time on the chill  
15 roll to assist cooling the film.

Occasionally, tacky material may stick to downstream rolls. This problem may be minimized by either placing a low surface energy (e.g. Teflon®) sleeve on the affected rolls, wrapping Teflon® tape on the effected rolls, or by feeding release paper in front of the effected rolls. Finally, if it appears that the tacky material may block to itself on the wound  
20 roll, release paper may be added immediately prior to winding. This is a standard method of preventing blocking of film during storage on wound rolls. Processing aids, release agents or contaminants should be minimized. In some cases, these additives can bloom to the surface and reduce the surface energy (raise the contact angle) of the hydrophilic surface.

An alternative method of making the multi-layer films as disclosed herein is to  
25 extrude a web comprising a material suitable for one of the individual layers. Extrusion methods as known to the art for forming flat films are suitable. Such webs may then be laminated to form a multi-layer film suitable for formation into a fluid pervious web using the methods discussed below. As will be recognized, a suitable material, such as a hot melt adhesive, can be used to join the webs to form the multi-layer film. A preferred adhesive is a  
30 pressure sensitive hot melt adhesive such as a linear styrene isoprene styrene ("SIS") hotmelt adhesive, but it is anticipated that other adhesives, such as polyester or polyamide powdered adhesives, hotmelt adhesives with a compatibilizer such as polyester, polyamide or low

residual monomer polyurethanes, other hotmelt adhesives, or other pressure sensitive adhesives could be utilized in making the multi-layer films of the present invention.

In another alternative method of making the films as disclosed herein, a base or carrier web can be separately extruded and one or more layers can be extruded thereon using an extrusion coating process to form a film. Preferably, the carrier web passes under an extrusion die at a speed that is coordinated with the extruder speed so as to form a very thin film having a thickness of less than about 25 microns. The molten polymer and the carrier web are brought into intimate contact as the molten polymer cools and bonds with the carrier web.

As noted above, a tie layer may enhance bonding between the layers. Contact and bonding are also normally enhanced by passing the layers through a nip formed between two rolls. The bonding may be further enhanced by subjecting the surface of the carrier web that is to contact the film to surface treatment, such as corona treatment, as is known in the art and described in Modern Plastics Encyclopedia Handbook, p. 236 (1994).

If a monolayer film layer is produced via tubular film (i.e., blown film techniques) or flat die (i.e., cast film) as described by K. R. Osborn and W. A. Jenkins in "Plastic Films, Technology and Packaging Applications" (Technomic Publishing Co., Inc. (1992)), then the film can go through an additional post-extrusion step of adhesive or extrusion lamination to other packaging material layers to form a multi-layer film. If the film is a coextrusion of two or more layers, the film can still be laminated to additional layers of packaging materials, depending on the other physical requirements of the final film. "Laminations Vs. Coextrusion" by D. Dumbleton (Converting Magazine (September 1992), also discusses lamination versus coextrusion. The films contemplated herein can also go through other post extrusion techniques, such as a biaxial orientation process.

### **Fluid Pervious Webs**

The films as disclosed herein can be formed into fluid pervious webs suitable for use as a topsheet in an absorbent article. As is described below, the fluid pervious web is preferably formed by macroscopically expanding a film as disclosed herein. The fluid pervious web contains a plurality of macroapertures, microapertures or both. Macroapertures and/or microapertures give the fluid pervious web a more consumer-preferred fiber-like or cloth-like appearance than webs apertured by methods such as embossing or perforation (e.g. using a roll with a multiplicity of pins) as are known to the art. One of skill in the art will

recognize that such methods of providing apertures to a film are also useful for providing apertures to the films as disclosed herein. Although the fluid pervious web is described herein as a topsheet for use in an absorbent article, one having ordinary skill in the art will appreciate these webs have other uses, such as bandages, agricultural coverings, and similar  
5 uses where it is desirable to manage fluid flow through a surface.

The macro and microapertures are formed by applying a high pressure fluid jet comprised of water or the like against one surface of the film, preferably while applying a vacuum adjacent the opposite surface of the film. In general, the film is supported on one surface of a forming structure having opposed surfaces. The forming structure is provided  
10 with a multiplicity of apertures therethrough which place the opposed surfaces in fluid communication with one another. While the forming structure may be stationary or moving, a preferred embodiment uses the forming structure as part of a continuous process where the film has a direction of travel and the forming structure carries the film in the direction of travel while supporting the film. The fluid jet and, preferably, the vacuum cooperate to  
15 provide a fluid pressure differential across the thickness of the film causing the film to be urged into conformity with the forming structure and to rupture in areas that coincide with the apertures in the forming structure.

The film passes over two forming structures in sequence. The first forming structure being provided with a multiplicity of fine scale apertures which, on exposure to the  
20 aforementioned fluid pressure differential, cause formation of microapertures in the web of film. The second forming structure exhibits a macroscopic, three-dimensional cross section defined by a multiplicity of macroscopic cross section apertures. On exposure to a second fluid pressure differential the film substantially conforms to the second forming structure while substantially maintaining the integrity of the fine scale apertures.

25 Such methods of aperturing are known as "hydroformation" and are described in greater detail in U.S. Patent Nos. 4,609,518; 4,629,643; 4,637,819; 4,681,793; 4,695,422; 4,778,644; 4,839,216; and 4,846,821, the disclosures of each being incorporated herein by reference.

30 The apertured web can also be formed by methods such as vacuum formation and using mechanical methods such as punching. Vacuum formation is disclosed in U.S. Patent No. 4,463,045, the disclosure of which is incorporated herein by reference. Examples of

mechanical methods are disclosed in U.S. Patent Nos. 4,798,604; 4,780,352; and 3,566,726, the disclosures of which are incorporated herein by reference.

### EXAMPLES

Polymers: The primary polymers used in this work are polypropylene (PP) and polyethylene (PE), but other polymers can be used (see, e.g., U.S. Patent No. 6,783,854, which provides a comprehensive list of polymers that are possible, although not all have been tested). Specific polymers evaluated were:

- Basell Profax PH-835: Produced by Lyondell-Basell as nominally a 35 melt flow rate Ziegler-Natta isotactic polypropylene.

- Basell Metocene MF-650W: Produced by Lyondell-Basell as nominally a 500 melt flow rate metallocene isotactic polypropylene.

- Polybond 3200: Produced by Crompton as a nominally 250 melt flow rate maleic anhydride copolymer.

- Exxon Achieve 3854: Produced by Exxon-Mobil Chemical as nominally a 25 melt flow rate metallocene isotactic polypropylene.

- Mosten NB425: Produced by Unipetrol as nominally a 25 melt flow rate Ziegler-Natta isotactic polypropylene.

- Danimer 27510: a polyhydroxyalkanoate copolymer from Danimer Scientific LLC.

- Dow Aspun 6811A: Produced by Dow Chemical as a 27 melt index polyethylene copolymer.

- Eastman 9921: Produced by Eastman Chemical as a polyester terephthalic homopolymer with a nominally 0.81 intrinsic viscosity.

Oils: Specific examples used were: Soy Bean Oil (SBO); Epoxidized soy bean oil (ESBO); Corn Oil (CO); Cottonseed Oil (CSO); and Canola Oil (CNO).

Compositions were made using a Baker Perkins CT-25 Screw, with the process conditions as noted in the below table:

Table

|    | Polymer      | Oil  | Ratio |    | Twin-Screw Temperature Profile (°C) |     |     |     |     |     |     |     |     |     | Poly Temp (°C) | Oil Temp (°C) | Screw RPM | Screw Type | Torque (%) |
|----|--------------|------|-------|----|-------------------------------------|-----|-----|-----|-----|-----|-----|-----|-----|-----|----------------|---------------|-----------|------------|------------|
|    |              |      |       |    | Z1                                  | Z2  | Z3  | Z4  | Z5  | Z6  | Z7  | Z8  | Z9  | Die |                |               |           |            |            |
| 1  | 835/650W     | SBO  | 90    | 10 | 40                                  | 160 | 180 | 200 | 200 | 200 | 210 | 210 | 210 | 170 | 216            | 80            | 500       | Intensive  | 29         |
| 2  | PH-835       | SBO  | 90    | 10 | 40                                  | 160 | 180 | 200 | 200 | 200 | 210 | 210 | 210 | 170 | 214            | 80            | 500       | Intensive  | 81         |
| 3  | PH-835       | SBO  | 80    | 20 | 40                                  | 160 | 180 | 200 | 200 | 200 | 210 | 210 | 210 | 170 | 214            | 80            | 500       | Intensive  | 56         |
| 4  | PH-835       | SBO  | 70    | 30 | 40                                  | 160 | 180 | 200 | 200 | 200 | 210 | 210 | 210 | 170 | 217            | 80            | 500       | Intensive  | 41         |
| 5  | PH-835       | SBO  | 65    | 35 | 40                                  | 160 | 180 | 200 | 200 | 200 | 210 | 210 | 210 | 170 | NR             | 80            | 500       | Intensive  | NR         |
| 6  | Achieve 3854 | SBO  | 90    | 10 | 40                                  | 160 | 180 | 200 | 200 | 200 | 210 | 210 | 210 | 170 | 220            | 80            | 500       | Intensive  | 64         |
| 7  | Achieve 3854 | SBO  | 80    | 20 | 40                                  | 160 | 180 | 200 | 200 | 200 | 210 | 210 | 210 | 170 | NR             | 80            | 500       | Intensive  | NR         |
| 8  | Mosten NB425 | SBO  | 80    | 20 | 40                                  | 160 | 180 | 200 | 200 | 200 | 210 | 210 | 210 | 170 | 220            | 80            | 500       | Intensive  | 44         |
| 9  | Mosten NB425 | SBO  | 70    | 30 | 40                                  | 160 | 180 | 200 | 200 | 200 | 210 | 210 | 210 | 170 | 213            | 80            | 500       | Intensive  | 37         |
| 10 | Mosten NB425 | SBO  | 65    | 35 | 40                                  | 160 | 180 | 200 | 200 | 200 | 210 | 210 | 210 | 170 | NR             | 80            | 500       | Intensive  | NR         |
| 11 | 835/PB32 00  | SBO  | 90    | 10 | 40                                  | 160 | 180 | 200 | 200 | 200 | 210 | 210 | 210 | 170 | 216            | 80            | 500       | Intensive  | 46         |
| 12 | 835/PB32 00  | SBO  | 80    | 20 | 40                                  | 160 | 180 | 200 | 200 | 200 | 210 | 210 | 210 | 170 | NR             | 80            | 500       | Intensive  | NR         |
| 13 | PH-835       | ESBO | 90    | 10 | 40                                  | 160 | 180 | 200 | 200 | 200 | 210 | 210 | 210 | 170 | 213            | 80            | 500       | Intensive  | 47         |
| 14 | PH-835       | ESBO | 80    | 20 | 40                                  | 160 | 180 | 200 | 200 | 200 | 210 | 210 | 210 | 170 | NR             | 80            | 500       | Intensive  | NR         |
| 15 | Achieve 3854 | ESBO | 90    | 10 | 40                                  | 160 | 180 | 200 | 200 | 200 | 210 | 210 | 210 | 170 | 216            | 80            | 500       | Intensive  | 46         |
| 16 | Achieve 3854 | ESBO | 80    | 20 | 40                                  | 160 | 180 | 200 | 200 | 200 | 210 | 210 | 210 | 170 | NR             | 80            | 500       | Intensive  | NR         |
| 17 | PH-835       | CO   | 90    | 10 | 40                                  | 160 | 180 | 200 | 200 | 200 | 210 | 210 | 210 | 170 | 197            | 80            | 400       | High       | 63         |
| 18 | PH-835       | CO   | 80    | 20 | 40                                  | 160 | 180 | 200 | 200 | 200 | 210 | 210 | 210 | 170 | 197            | 80            | 400       | High       | 50         |
| 19 | PH-835       | CO   | 70    | 30 | 40                                  | 160 | 180 | 200 | 200 | 200 | 210 | 210 | 210 | 170 | 210            | 80            | 400       | High       | 39         |



|    | Polymer       | Oil | Ratio    |     | Twin-Screw Temperature Profile (°C) |     |     |     |     |     |     |     |     |     | Poly Temp (°C) | Oil Temp (°C) | Screw RPM | Screw Type | Torque (%) |
|----|---------------|-----|----------|-----|-------------------------------------|-----|-----|-----|-----|-----|-----|-----|-----|-----|----------------|---------------|-----------|------------|------------|
|    |               |     | Poly-mer | Oil | Z1                                  | Z2  | Z3  | Z4  | Z5  | Z6  | Z7  | Z8  | Z9  | Die |                |               |           |            |            |
| 20 | Achieve 3854  | CO  | 90       | 10  | 40                                  | 160 | 180 | 200 | 200 | 200 | 200 | 210 | 210 | 210 | 204            | 80            | 400       | High       | 63         |
| 21 | Achieve 3854  | CO  | 80       | 20  | 40                                  | 160 | 180 | 200 | 200 | 200 | 200 | 210 | 210 | 210 | 200            | 80            | 400       | High       | 52         |
| 22 | Achieve 3854  | CO  | 70       | 30  | 40                                  | 160 | 180 | 200 | 200 | 200 | 200 | 210 | 210 | 210 | 202            | 80            | 400       | High       | 40         |
|    |               |     |          |     |                                     |     |     |     |     |     |     |     |     |     |                |               |           |            |            |
| 23 | PH-835        | CNO | 90       | 10  | 40                                  | 160 | 180 | 200 | 200 | 200 | 200 | 210 | 210 | 210 | 201            | 80            | 400       | High       | 60         |
| 24 | PH-835        | CNO | 80       | 20  | 40                                  | 160 | 180 | 200 | 200 | 200 | 200 | 210 | 210 | 210 | 201            | 80            | 400       | High       | 50         |
| 25 | PH-835        | CNO | 70       | 30  | 40                                  | 160 | 180 | 200 | 200 | 200 | 200 | 210 | 210 | 210 | 204            | 80            | 400       | High       | 39         |
| 26 | Achieve 3854  | CNO | 90       | 10  | 40                                  | 160 | 180 | 200 | 200 | 200 | 200 | 210 | 210 | 210 | 206            | 80            | 400       | High       | 62         |
| 27 | Achieve 3854  | CNO | 80       | 20  | 40                                  | 160 | 180 | 200 | 200 | 200 | 200 | 210 | 210 | 210 | 207            | 80            | 400       | High       | 51         |
| 28 | Achieve 3854  | CNO | 70       | 30  | 40                                  | 160 | 180 | 200 | 200 | 200 | 200 | 210 | 210 | 210 | 204            | 80            | 400       | High       | 41         |
|    |               |     |          |     |                                     |     |     |     |     |     |     |     |     |     |                |               |           |            |            |
| 29 | PH-835        | CSO | 90       | 10  | 40                                  | 160 | 180 | 200 | 200 | 200 | 200 | 210 | 210 | 210 | 197            | 80            | 400       | High       | 60         |
| 30 | PH-835        | CSO | 80       | 20  | 40                                  | 160 | 180 | 200 | 200 | 200 | 200 | 210 | 210 | 210 | 196            | 80            | 400       | High       | 51         |
| 31 | PH-835        | CSO | 70       | 30  | 40                                  | 160 | 180 | 200 | 200 | 200 | 200 | 210 | 210 | 210 | 196            | 80            | 400       | High       | 39         |
| 32 | Achieve 3854  | CSO | 90       | 10  | 40                                  | 160 | 180 | 200 | 200 | 200 | 200 | 210 | 210 | 210 | 199            | 80            | 400       | High       | 62         |
| 33 | Achieve 3854  | CSO | 80       | 20  | 40                                  | 160 | 180 | 200 | 200 | 200 | 200 | 210 | 210 | 210 | 193            | 80            | 400       | High       | 51         |
| 34 | Achieve 3854  | CSO | 70       | 30  | 40                                  | 160 | 180 | 200 | 200 | 200 | 200 | 210 | 210 | 210 | 194            | 80            | 400       | High       | 40         |
| 35 | Danimer 27510 | SBO | 95       | 5   | 40                                  | 170 | 180 | 180 | 180 | 180 | 180 | 180 | 180 | 180 | 177            | 80            | 500       | High       | 40         |
| 36 | Danimer 27510 | SBO | 93       | 7   | 40                                  | 170 | 180 | 180 | 180 | 180 | 180 | 180 | 180 | 180 | 171            | 80            | 500       | High       | 32         |
| 37 | Danimer 27510 | SBO | 90       | 10  | 40                                  | 170 | 180 | 180 | 180 | 180 | 180 | 180 | 180 | 180 | 169            | 80            | 500       | High       | 22         |

|    | Polymer      | Oil | Ratio    |     | Twin-Screw Temperature Profile (°C) |     |     |     |     |     |     |     |     |     | Poly Temp (°C) | Oil Temp (°C) | Screw RPM | Screw Type | Torque (%) |
|----|--------------|-----|----------|-----|-------------------------------------|-----|-----|-----|-----|-----|-----|-----|-----|-----|----------------|---------------|-----------|------------|------------|
|    |              |     | Poly-mer | Oil | Z1                                  | Z2  | Z3  | Z4  | Z5  | Z6  | Z7  | Z8  | Z9  | Die |                |               |           |            |            |
| 38 | Aspun 6811A  | SBO | 90       | 10  | 40                                  | 160 | 180 | 190 | 190 | 190 | 190 | 190 | 190 | 170 | 176            | 80            | 500       | High       | 50         |
| 39 | Aspun 6811A  | SBO | 80       | 20  | 40                                  | 160 | 180 | 190 | 190 | 190 | 190 | 190 | 190 | 170 | 179            | 80            | 500       | High       | 41         |
| 40 | Aspun 6811A  | SBO | 70       | 30  | 40                                  | 160 | 180 | 190 | 190 | 190 | 190 | 190 | 190 | 170 | 168            | 80            | 500       | High       | 28         |
| 41 | Eastman 9921 | SBO | 85       | 15  | 40                                  | 220 | 260 | 270 | 290 | 290 | 290 | 290 | 280 | 250 | 262            | 80            | 600       | High       | 43         |
| 42 | Eastman 9921 | SBO | 80       | 20  | 40                                  | 220 | 260 | 270 | 290 | 290 | 290 | 290 | 280 | 250 | NR             | 80            | 500       | High       | NR         |

For examples 5, 7, 10, 12, 16, and 42, it was noted that the SBO was surging at the end of the CT-25 extruder. Examples 5, 7, 10, 12, 16, 39, and 41 failed to properly pelletize. Example 41 produced brittle strands.

The shear viscosity influence of adding soy bean oil to Lyondell Basell Profax PH-835 at 10, 20 and 30wt% was measured using a capillary rheometer according to ASTM D3835 at 230°C using a 30:1 capillary. Adding 30wt% soy bean oil to PH-835 results in a 50% reduction in shear viscosity at 1000 s<sup>-1</sup>, which results in lower flow forces and process pressures.

Examples 1-42 show the polymer plus additive tested in a stable range and to the limit. As used herein, stable refers to the ability of the composition to be extruded and to be pelletized. What was observed was that during the stable composition, strands from the B&P 25mm system could be extruded, quenched in a water bath at 5°C and cut via a pelletizer without interruption. The twin-screw extrudate was immediately dropped into the water bath. During stable extrusion, no significant amount of oil separated from the formulation strand (>99wt% made it through the pelletizer). The composition became unstable when it was clear that the polymer and oil were separating from each other at the end of the twin-screw and the composition strands could not be maintained. Without being bound by theory, the polymer at this point is considered fully saturated. The saturation point can change based on the oil and polymer combination, along with the process conditions. The practical utility is that the oil and polymer remain admixed and do not separate, which is a function of the mixing level and quench rate for proper dispersion of the additive. Specific Examples where the extrusion became unstable from high oil inclusion are Example 5, 7, 10, 12, 16 and 42.

Films can be produced from a composition of any one of Examples 1-42.

All documents cited in the Detailed Description of the Invention are, in relevant part, incorporated herein by reference; the citation of any document is not to be construed as an admission that it is prior art with respect to the present invention. To the extent that any meaning or definition of a term in this document conflicts with any meaning or definition of the same term in a document incorporated by reference, the meaning or definition assigned to that term in this document shall govern.

The dimensions and values disclosed herein are not to be understood as being strictly limited to the exact numerical values recited. Instead, unless otherwise specified, each such dimension is intended to mean both the recited value and a functionally equivalent range

surrounding that value. For example, a dimension disclosed as “40 mm” is intended to mean “about 40 mm”.

While particular embodiments of the present invention have been illustrated and described, it would be obvious to those skilled in the art that various other changes and  
5 modifications can be made without departing from the spirit and scope of the invention. It is therefore intended to cover in the appended claims all such changes and modifications that are within the scope of this invention.

## CLAIMS

What is Claimed is:

1. A film comprising at least one layer of a composition comprising an intimate admixture of
  - (a) a thermoplastic polymer; and
  - (b) 5 wt% to 40 wt% of an oil, based upon the total weight of the composition, the oil having a melting point of 25°C or less and a boiling point greater than 160°C.
2. The film of claim 1, wherein the thermoplastic polymer comprises a polyolefin, a polyester, a polyamide, copolymers thereof, or combinations thereof.
3. The film of claim 2, wherein the thermoplastic polymer is selected from the group consisting of polypropylene, polyethylene, polypropylene co-polymer, polyethylene co-polymer, polyethylene terephthalate, polybutylene terephthalate, polylactic acid, polyhydroxyalkanoates, polyamide-6, polyamide-6,6, and combinations thereof.
4. The film of any one of claims 1 to 3, wherein the thermoplastic polymer comprises polypropylene.
5. The film of any one of claims 1 to 4, comprising 8 wt% to 30 wt% of the oil, based upon the total weight of the composition.
6. The film of any one of claims 1 to 5, wherein the oil comprises a lipid.
7. The film of claim 6, wherein the lipid comprises a monoglyceride, diglyceride, triglyceride, fatty acid, fatty alcohol, esterified fatty acid, epoxidized lipid, maleated lipid, hydrogenated lipid, alkyd resin derived from a lipid, sucrose polyester, or combinations thereof.
8. The film of any one of claims 1 to 5, wherein the oil is selected from the group consisting of soy bean oil, epoxidized soy bean oil, maleated soy bean oil, corn oil, cottonseed oil, canola oil, castor oil, coconut oil, coconut seed oil, corn germ oil, linseed oil, olive oil, oiticica oil, palm kernel oil, palm oil, palm seed oil, peanut oil, cottonseed oil, hempseed oil, rapeseed oil, safflower oil, sperm oil, sunflower seed oil, tall oil, tung oil, whale oil, triolein, trilinolein, 1-stearo- dilinolein, 1,2-diacetopalmitin, and combinations thereof.

9. The film of any one of claims 1 to 8, wherein the oil is dispersed within the thermoplastic polymer such that the oil has a droplet size of less than 10  $\mu\text{m}$  within the thermoplastic polymer.
10. The film of claim 9, wherein the droplet size is less than 1  $\mu\text{m}$ .

## AMENDED CLAIMS

received by the International Bureau on 28 september 2012 (28.09.2012)

## CLAIMS

What is Claimed:

1. A film comprising at least one layer of a composition comprising an intimate admixture of
  - (a) a thermoplastic polymer; and
  - (b) 5 wt% to 40 wt% of an oil, based upon the total weight of the composition, the oil having a melting point of 25°C or less and a boiling point greater than 160°C, said oil being dispersed within said thermoplastic polymer such that the oil has a droplet size of less than 10 µm within the thermoplastic polymer.
2. The film of claim 1, wherein the thermoplastic polymer comprises a polyolefin, a polyester, a polyamide, copolymers thereof, or combinations thereof.
3. The film of claim 2, wherein the thermoplastic polymer is selected from the group consisting of polypropylene, polyethylene, polypropylene co-polymer, polyethylene co-polymer, polyethylene terephthalate, polybutylene terephthalate, polylactic acid, polyhydroxyalkanoates, polyamide-6, polyamide-6,6, and combinations thereof.
4. The film of any one of claims 1 to 3, wherein the thermoplastic polymer comprises polypropylene.
5. The film of any one of claims 1 to 4, comprising 8 wt% to 30 wt% of the oil, based upon the total weight of the composition.
6. The film of any one of claims 1 to 5, wherein the oil comprises a lipid.
7. The film of claim 6, wherein the lipid comprises a monoglyceride, diglyceride, triglyceride, fatty acid, fatty alcohol, esterified fatty acid, epoxidized lipid, maleated lipid, hydrogenated lipid, alkyd resin derived from a lipid, sucrose polyester, or combinations thereof.
8. The film of any one of claims 1 to 5, wherein the oil is selected from the group consisting of soy bean oil, epoxidized soy bean oil, maleated soy bean oil, corn oil, cottonseed oil, canola oil, castor oil, coconut oil, coconut seed oil, corn germ oil, linseed oil, olive oil, oiticica oil, palm kernel oil, palm oil, palm seed oil, peanut oil, cottonseed oil, hempseed oil, rapeseed oil, safflower oil, sperm oil, sunflower seed oil, tall oil, tung oil, whale oil, triolein, trilinolein, 1-stearo- dilinolein, 1,2-diacetopalmitin, and combinations thereof.

9. The film of any one of claims 1 to 8, wherein the droplet size of said oil is less than 1  $\mu\text{m}$ .



# INTERNATIONAL SEARCH REPORT

International application No

PCT/US2012/038537

## A. CLASSIFICATION OF SUBJECT MATTER

INV. C08K5/01 C08J5/18  
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)

C08J C08K

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. |
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| X         | US 2006/057409 A1 (KLIESCH HOLGER [DE] ET AL) 16 March 2006 (2006-03-16)<br>claims 1,8,9,11,12<br>-----                                                                            | 1-5,8                 |
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| X         | WO 2007/044803 A1 (PROCTER & GAMBLE [US]; NODA ISAO [US]; SATKOWSKI MICHAEL M [US]; ALLEN) 19 April 2007 (2007-04-19)<br>examples 1,3,4,5,6,20,26,28-20<br>claims 1,3,5,9<br>----- | 1-5,8                 |



Further documents are listed in the continuation of Box C.



See patent family annex.

### \* Special categories of cited documents :

"A" document defining the general state of the art which is not considered to be of particular relevance

"E" earlier application or patent but published on or after the international filing date

"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art

"&" document member of the same patent family

Date of the actual completion of the international search

30 July 2012

Date of mailing of the international search report

06/08/2012

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# INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No

PCT/US2012/038537

| Patent document<br>cited in search report |    | Publication<br>date | Patent family<br>member(s)                                                                                                                                                                                                                                                                                                                                                                                                                                                                                           | Publication<br>date                                                                                                                                                                                                                                                                                                                                                                                  |
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