

**METHOD FOR PRODUCING A POLYOLEFIN COMPOSITION
AND METHOD FOR PRODUCING A POLYOLEFIN FILM**

This patent application claims foreign
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5 filed September 13, 1994.

FIELD OF THE INVENTION

The present invention relates to a method
for producing a polyolefin composition and to a
method for producing a polyolefin film. More
10 particularly, the present invention relates to a
method for producing a polyolefin composition which
is comprised of polyolefin particles having an
average particle diameter of from 500 to 1300 μm and
having not more than 10% by weight of fine particles
15 with a diameter of 300 μm or less. The present
invention also relates to a method for producing a
polyolefin film from the polyolefin composition.
The resultant polyolefin film has an excellent
appearance, transparency, blocking resistance and
20 scratch resistance and, as such, is especially
useful as a food packaging film or wrap.

BACKGROUND OF THE INVENTION

Films made from polyolefin have a wide
variety of uses, such as, packaging materials for

various kind of articles including foods. More specifically, films made from polypropylene have been widely used for food packaging because of their excellent mechanical properties, optical properties, such as gloss, transparency and the like, and food sanitation properties, such as vapor barrier, odorlessness and the like.

However, films made from polyolefin have a distinct disadvantage in that the polyolefin film to the another film when the films are piled one on top of the other. This undesirable property of polyolefin films is referred to as blocking and deteriorates the work sufficiency, for example, in packaging.

Two main methods have developed to prevent blocking in polyolefin films. In one method, an antiblocking agent, such as an inorganic fine powder (e.g., silica) or an organic fine particles (e.g., cross-linked polymer), is mixed with the polyolefin particles used to prepare the polyolefin film. In the other method, an antiblocking agent in combination with a lubricant, such as a fatty acid amide, is mixed with the polyolefin particles used to prepare the polyolefin film.

It is well known that polyolefins can be obtained by polymerization of olefins in the

presence of a catalyst represented by a titanium trichloride-organoaluminum compound. However, the catalytic activity of such catalysts for polymerization is very low. The polyolefin particles obtained by such polymerization have a comparatively wide distribution of particles sizes and usually contain more than 20% by weight of fine particles having a diameter of not more than 300 μm . The average particle size is usually less than 500 μm . In addition, the dispersibility of antiblocking agents in such polyolefin particles is good.

Several improved catalysts having higher catalytic activity for the polymerization of olefins have been developed. Polyolefin particles obtained by using the improved catalysts usually contain less than 10% by weight of fine particles having particle diameters not more than 300 μm . In addition, such polyolefin particles have a narrow distribution of particle sizes and the average particle size is more than 500 μm .

Polyolefin particles obtained by using the improved catalyst have the advantages of ease of preparation and decreased danger of dust explosion. However, the dispersibility of antiblocking agents into such polyolefin particles is so poor that any polyolefin composition made from such particles will

have the antiblocking agents unhomogeneously dispersed throughout. Any polyolefin film produced from such a polyolefin composition will have a poor or inferior appearance.

5 Methods to improve the dispersion of antiblocking agents in polyolefin particles are described in JP-A-57-3840 and JP-A-58-225142. The term "A" as described herein means Unexamined Patent Publications.

10 In JP-57-3840, a method is disclosed in which polyolefin particles are mixed with an antiblocking agent, i.e., silica powder (trade name "syloid 244" or "Aerosil 380"). The mixture is subjected to a temperature between 50 °C and the
15 melting temperature of the polyolefin. An organic amide is then added to the mixture as a lubricant. According to this method, the antiblocking agent and the lubricant must be separately mixed with the polyolefin particles. The disclosed mixing steps
20 are complicated and undesirable for industrial processes.

 In JP-A-58-225142, a method is disclosed for preparing a composition in which propylene polymer particles are mixed with an antiblocking
25 agent, i.e., silica powder (trade name "syloid 244" or "Aerosil 200") and a magnesium and/or aluminum

salt of a specific fatty acid. However, the use of a metal salt of a specific fatty acid with the antiblocking agent makes the mixing step complicated and undesirable for industrial processes.

5

SUMMARY OF THE INVENTION

The object of the present invention is to provide a superior industrial process for producing a polyolefin composition comprising (I) polyolefin particles having a large average particle diameter and containing a small amount of fine particles
10 obtained by using a high activity catalyst for polymerization and (II) an antiblocking agent, without the need for the complicated mixing step or steps used in the conventional methods. The
15 resultant polyolefin composition would be used to produce a polyolefin film which has an excellent appearance, transparency, blocking resistance and scratch resistance.

The present inventors found that the
20 object could be attained by

a) mixing

i) polyolefin particles having a large average particle diameter and containing a small amount of fine particles, and

ii) a specified amount of an inorganic fine powder having a specific range of an average particle diameter and an apparent specific gravity, and

5 b) then melt-kneading the mixture.

More specifically, the present invention relates to a process for producing a polyolefin composition, comprising the steps of:

a) mixing

10 i) 100 parts by weight of polyolefin particles (I) having an average particle diameter of from 500 to 1300 μm and containing not more than 10% by weight of fine particles having particle diameters of 300 μm or less and

15 ii) 0.05 to 0.6 parts by weight of an inorganic fine powder (II) having an average particle diameter of from 1 to 4 μm and an apparent specific gravity of 0.2 to 0.5 g/cm^3 ; and

20 b) melt-kneading the mixture to form the composition.

The present invention also relates to a process for producing polyolefin film, comprising the steps of:

a) mixing

25 i) 100 parts by weight of polyolefin particles (I) having an average particle diameter of

from 500 to 1300 μm and containing not more than 10% by weight of fine particles having particle diameters of 300 μm or less and

5 ii) 0.05 to 0.6 parts by weight of an inorganic fine powder (II) having an average particle diameter of from 1 to 4 μm and an apparent specific gravity of 0.2 to 0.5 g/cm^3 ;

 b) melt-kneading the mixture to form a polyolefin composition; and

10 c) melt-extruding the polyolefin composition to form the polyolefin film.

DETAILED DESCRIPTION OF THE INVENTION

 The present invention will be explained in
15 further detail in the following Preparation section and Examples.

Preparation of polyolefin composition

 The process for producing a polyolefin composition comprises a step of mixing polyolefin
20 particles (I) having a specific range of an average particle diameter and containing an amount of fine particles and a specified amount of an inorganic fine powder (II) having a specific range of an

average particle diameter and an apparent specific gravity, and a step of melt-kneading the obtained mixture.

The polyolefin particles (I) used in the present invention are polyolefin particles, such as polypropylene and polyethylene, having an average particle diameter of from 500 to 1300 μm , preferably from 600 to 1200 μm . Furthermore, the polyolefin particles contain not more than 10% by weight of fine particles having an average particle diameter of 300 μm or less. When the average particle diameter of the polyolefin particles exceeds 1300 μm , the dispersion of the inorganic powder is insufficient to produce the desired polyolefin composition. The average particle diameter of the polyolefin particles is determined by measuring particle size distribution by laser diffraction.

As the polyolefin used in the present invention, polyolefins incorporating propylene as a monomer component are preferred, and a homopolymer of propylene and random copolymers of propylene-ethylene, propylene- α -olefin and propylene-ethylene- α -olefin are particularly preferred. As the α -olefin, those having 4 to 10 carbon atoms are preferred and, among them, butene-1 is more preferred.

The inorganic fine powder (II) used in the present invention is one having an average particle diameter of from 1 to 4 μm and an apparent specific gravity of from 0.2 to 0.5 g/cm^3 , preferably from 0.2 to 0.5 g/cm^3 . When the average particle diameter of the inorganic fine powder is less than 1 μm , the blocking resistance of the film obtained from the polyolefin composition comprising the polyolefin particles and the inorganic fine powder is insufficient. When the average particle diameter of the inorganic fine powder exceeds 4 μm , the appearance and transparency of the film obtained from the composition is decreased. When the apparent specific gravity of the inorganic fine powder is less than 0.2 g/cm^3 , the appearance of the film obtained from the composition decreases and when it exceeds 0.5 g/cm^3 , the scratch resistance of the film obtained from the composition deteriorates.

Examples of the inorganic fine powders are silica, magnesium silicate, aluminum silicate, calcium carbonate, magnesium oxide and the like. Among them, silica is suitably used.

The amount of the inorganic fine powder (II) used in the composition is between 0.05 to 0.6 parts by weight per 100 parts by weight of the polyolefin particles (I), more preferably 0.1 to 0.3

parts by weight to 100 parts by weight of the polyolefin particles (I). When the amount of the inorganic fine powder in the composition is less than 0.05 parts by weight, the blocking resistance of the film made from the composition is
5 insufficient. When the amount of the inorganic fine powder in the composition exceeds 0.6 parts by weight, the dispersibility of the inorganic fine powder in the composition decreases and results in
10 deterioration of the appearance and transparency of the resulting film.

The inorganic fine powder (II) preferred in the present invention is silica having an average particle diameter of from 1.5 to 4 μm and an
15 apparent specific gravity of from 0.2 to 0.4 g/cm^3 . Furthermore, it is preferred that the amount of the silica in the composition be between 0.1 to 0.3 parts by weight per 100 parts by weight of the polyolefin particles (I).

20 Conventional mixers, such as a tumbler mixer, super mixer, Henschel mixer, screw blender and ribbon blender, can be used for mixing the polyolefin particles (I) and the inorganic fine powder (II) in the method of the present invention.
25 Mixers equipped with rotors are preferred in the present method because of the higher dispersion

efficiency of the inorganic fine powder (II) in the composition. When mixers equipped with rotors (for example, super mixer and Henschel mixer) are used in the present method, the peripheral speed of the edge
5 of the rotors is preferably more than 5 m/sec in view of the dispersibility of the inorganic fine powder (II).

In the melt-kneading step, a temperature equal to or greater than the melting temperature of
10 the polyolefin particles can be used. This temperature can be obtained, for example, by using a melt-extruder, Banbury mixer.

In addition to the polyolefin particles (I) and the inorganic fine powder (II), additives
15 such as antioxidants, neutralizers, lubricants, antistatic agents and the like can be mixed with the polyolefin composition of the present invention as the need arises. The amount of additives to be used is not critical. For example, the amount of
20 lubricants to be used can vary from 0.5 to 0.05 parts by weight per 100 parts by weight of the polyolefin composition, preferably from 0.2 to 0.05 parts by weight and more preferably from 0.1 to 0.05 parts by weight.

25 The polyolefin composition obtained from the above-described process can be used for

producing melt-extruded articles, especially for producing polyolefin films of about 10 to 100 μm in thickness.

In the method for producing polyolefin
5 film, any conventional extrusion method (e.g., a T-die film-forming method, a tubular film-forming method and the like) can be used. A T-die film-forming method wherein high-speed film formation can be accomplished by using a large scale film-forming
10 machine is preferred.

The conditions for film formation depend on the kind of polyolefin and the extrusion method used. For example, when a T-die film-forming method is used, polypropylene film can be obtained by the
15 following four step process. First, the polypropylene composition obtained by the above-disclosed method is melted between 180 and 300 °C. Second, the resultant molten composition is melt-extruded between 180 and 280 °C. Third, the extruded
20 molten composition is cooled with cooling rolls between 10 and 70 °C. Finally, the cooled composition is subjected to winding in a film winder in order to obtain the polyolefin film. The thickness of the film can be controlled by adjusting
25 the amount of the extruded molten composition and the winding speed.

According to the present invention, a polyolefin composition providing a film having an excellent balance of a transparency, blocking resistance and scratch resistance can be obtained by
5 using polyolefin particles having a large average particle diameter and low content of fine particles without applying the conventional method in which the complicated mixing step is considered essential.

The following Examples will illustrate the
10 present invention. However, it is to be understood that the present invention is not limited to these Examples.

The properties mentioned in the DETAILED DESCRIPTION OF THE INVENTION and the Examples were
15 measured in the following manner.

(1) Melt flow rate (MFR)

Melt flow rate was determined according to the method described in JIS K7210, condition 14.

20 (2) The contents of ethylene and butene-1

Ethylene content was determined by the IR spectrum method described at page 256 in "(i) Random copolymer" of KOBUNSHI BUNSEKI Handbook (published by Asakura Shoten in 1985).

Butene-1 content was determined by the IR spectrum method from the following equation:

$$\text{Butene-1 content (\% by weight)} = 1.208K'_{767}$$

wherein K'_{767} is absorbance at 767 cm^{-1} .

5 (3) Average particle diameter of the polyolefin particles

The average particle diameter of the polyolefin particles was determined based on the weight-cumulative particle size distribution of the powder. The weight-cumulative particle size distribution of the powder was measured with an apparatus (HELOS-E/LA; manufactured by Nihon Densi Company Ltd.) for measuring particle size distribution by laser diffraction and was expressed
10
15 in terms of median diameter.

(4) Average particle diameter of the inorganic fine powder

The average particle diameter of the inorganic fine powder was determined based on the weight-cumulative particle size distribution of the powder measured with a Coulter counter/multi-sizer and was expressed in terms of median diameter.
20

(5) Apparent specific gravity of the inorganic fine powder

The apparent specific gravity of the inorganic fine powder was determined according to the testing method described in item 6.8 in JIS K6220.

Film properties.

(6) Appearance.

The number of fish eyes (FE) in the film having a diameter of 200 μm or more were visually counted. The appearance of the film was judged as "good" when the number of FE was about 10 or less per 1,000 cm^2 of the film and as "poor" when it was 20 or more. When the number of FE was in the range of 10 to 20, the appearance was judged as "nearly good", if FE having a diameter of about 100 μm could be observed.

(7) Haze

Haze was determined according to the method described in JIS K7105.

(8) Blocking resistance

Blocking resistance was determined by the following method. Two films of 225 mm by 50 mm were

placed one upon the other and an overlapped area of 100 mm by 50 mm of the films was conditioned under a load of 40 g/cm² at 60 °C for 3 hours. Thereafter, the conditioned overlapped films were allowed to stand in an atmosphere having 50% humidity at 23 °C for at least 30 minutes. Then the strength required for peeling the stored films was determined at a loading velocity of 20 g/min using a blocking tester manufactured by Shimazu Corp. The lower the value obtained by above measurement, the greater the blocking resistance.

(9) Scratch resistance

Scratch resistance was determined by the following method. Two films laid at 23 °C for 1 day after the preparation of the films were used. One of the films was set on a soft material to prevent scratching of the film and fixed on a shaker (film A). The other film was fixed on a metal pipe and pressed onto film A by a bar laminated with silicone rubber. The films were rubbed against each other for 1 minute under the conditions such that the swinging width was 40 mm and the swinging speed was 120 cycles/min. The haze values of film A before and after the rubbing were determined according to method described in JIS K7105. The difference

between the two values was termed Δ haze and is used to evaluate the scratch resistance of the film.

Δ haze = haze after the rubbing - haze before the rubbing

5 The lower the value obtained by the above measurement method, i.e., the lower the Δ haze, the greater the scratch resistance.

Example 1

10 In this Example, polypropylene particles (melt flow rate: 8.2 g/10 min) having an average particle diameter of 620 μ m and containing 3% by weight of a fine powder of 300 μ m or less in diameter were used as the polyolefin particles. 100
15 parts by weight of these polypropylene particles, 0.05 parts by weight of calcium stearate, 0.1 parts by weight of Sumilizer BHT (manufactured by Sumitomo Chemical Company Ltd.), 0.1 parts by weight of Irganox 1010 (manufactured by Ciba-Geigy Ltd.), 0.1
20 parts by weight of erucic amide, 0.15 parts by weight of Syloid 55 as the inorganic fine powder (manufactured by Fuji Davison Ltd., silica powder; average particle diameter of 2.7 μ m and an apparent specific gravity of 0.33 g/cm³) were mixed with a 20
25 L super mixer. The peripheral speed of the edge of a rotor was 8 m/sec. The resulting mixture was

melt-kneaded by a 40 mmφ T-die extruder at a die temperature of 250 °C and then cooled with a cooling roll of 30 °C at surface temperature to obtain a single layer film of 30 μm in thickness. The properties of the film are given in Table 3. The appearance, haze, blocking resistance and scratch resistance were excellent.

Example 2

The same procedure as described in Example 1 was repeated in Example 2, except that propylene-ethylene-butene-1 copolymer particles (hereinafter abbreviated as PP-1; ethylene content of 2.1% by weight, butene-1 content of 5.5% by weight and melt flow rate of 4.8 g/10 min) having an average particle diameter of 700 μm and containing 2% by weight of a fine powder of 300 μm or less in diameter was used as the polyolefin particles. In addition, the amount of Syloid 55 used as the inorganic fine powder was 0.10 parts by weight. The properties of the film thus obtained are given in Table 3.

Example 3

The same procedure as described in Example 1 was repeated in Example 3, except that propylene-

ethylene-butene-1 copolymer particles (ethylene content of 4.3% by weight, butene-1 content of 2.0% by weight and melt flow rate of 4.9 g/10 min) having an average particle diameter of 1170 μm and
5 containing 6% by weight of a fine powder of 300 μm or less in diameter was used as the polyolefin particles. In addition, the amount of Syloid 55 used as the inorganic fine powder was 0.15 parts by weight. The properties of the film thus obtained
10 are given in Table 3.

Comparative Example 1

The same procedure as described in Example 1 was repeated in Comparative Example 1, except that propylene-ethylene-butene-1 copolymer particles
15 (ethylene content of 4.3% by weight, butene-1 content of 4.1% by weight and melt flow rate of 5.7 g/10 min) having an average particle diameter of 1510 μm and containing 3% by weight of a fine powder of 300 μm or less in diameter was used as the
20 polyolefin particles. In addition, the amount of Syloid 55 used as the inorganic fine powder was 0.15 parts by weight. The properties of the film thus obtained are given in Table 3.

Examples 4 to 6

The same procedure as described in Example 1 was repeated in Examples 4 to 6, except that PP-1 was used as the polyolefin particles and types and amounts (parts by weight) of the inorganic fine powder were as indicated in Table 1. The properties of the films thus obtained are given in Table 4.

Table 1

Example No.	inorganic fine powder	mixing amount
4	Syloid 57 (silica powder manufactured by Fuji Devison Ltd.	0.15
5	Syloid 74 (silica powder manufactured by Fuji Devison Ltd.	0.15
6	Syloid 55 (silica powder manufactured by Fuji Devison Ltd.	0.25

Example 7

The same procedure as described in Example 1 was repeated in Example 7, except that PP-1 used as the polyolefin particles and the mixing amount of the inorganic fine powder was 0.50 parts by weight. In addition, a 20 mmφ twin-screw extruder was used

for melt-kneading. The properties of the film thus obtained are given in Table 4.

Reference Example 1

The same procedure as described in Example 1 was repeated in Reference Example 1, except that propylene-ethylene-butenen-1 copolymer particles (ethylene content of 2.0 % by weight, butene-1 content of 5.7 % by weight and melt flow rate of 6.0 g/10 min) having an average particle diameter of 320 μm and containing 37 % by weight of a fine powder of 300 μm or less in diameter was used as the polyolefin particles. In addition, Syloid 244 (manufactured by Fuji Devison Ltd., silica powder; average particle diameter of 1.8 μm and an apparent specific gravity of 0.13 g/cm³) was used as the inorganic fine powder at 0.15 parts by weight. The properties of the film thus obtained are given in Table 3.

In the present Reference Example, polymer particles were used which had an average particle diameter (320 μm) less than that employed in the present method (500 to 1300 μm) and which had a content of fine particles (37% of fine powder of 300 μm or less) greater than that employed in the present method (not more than 10% of fine powder of

300 μm or less). In addition, the apparent specific gravity of the inorganic fine powder (0.13 g/cm^3) was less than that used in the present method (from 0.2 to 0.5 g/cm^3). The resultant film had a good
5 appearance and scratch resistance but increased haze and blocking resistance.

Comparative Examples 2 to 7

The same procedure as described in Example 1 was repeated in Comparative Examples 2 to 7,
10 except that PP-1 was used as the polyolefin particles. In addition, the types and mixing amounts (parts by weight) of the inorganic fine powder were used as shown in Table 2. The properties of the films thus obtained are given in
15 Table 4.

In summary, when any item among the average particle diameter of the polyolefin particles (I), the apparent specific gravity of the inorganic fine powder (II) and the mixing amount of
20 the inorganic fine powder (II) is outside the ranges of those of the present invention, the appearance or properties of the film worsens.

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Table 2

Comparative Example No.	inorganis fine powder	mixing amount
2	-	0
3	SYLTON AMT (aluminium silicate powder manufactured by Mizusawa Chemical Co.)	0.15
4	Syloid 161 (silica powder manufactured by Fuji Devison Co.)	0.20
5	Syloid 244 (silica powder maufactured by Fuji Devison Co.)	0.15
6	SYLTON AMT 20 (aluminium silicate powder manufactured by Mizusawa Chemical Co.)	0.15
7	Syloid 55 (silica powder manufactured by Fuji Devison Co.)	1.0

Table 3

Examples and Comparative Examples	Polyolefin particles (I)		Inorganic fine powder (II)			Film property			
	Average particle diameter μm	Content of fine powder (300 μm or less) weight %	Average particle diameter μm	Apparent specific gravity g/cm^3	Mixing amount parts by weight	Appearance	Haze %	Blocking resistance $\text{g}/100\text{cm}^2$	Scratch resistance (Δ Haze) %
Example 1	620	3	2.7	0.33	0.15	good	2.0	11	2.3
Example 2	700	2	2.7	0.33	0.10	good	1.0	45	2.2
Example 3	1170	6	2.7	0.33	0.15	good	1.6	33	1.6
Comparative Example 1	1510	3	2.7	0.33	0.15	bad	2.1	50	2.0
Reference Example 1	320	37	1.8	0.13	0.15	good	3.0	63	0.5

Table 4

Examples and Comparative Examples	Polyolefin particles (I)		Inorganic fine powder (II)			Film property			
	Average particle diameter μm	Content of fine powder (300 μm or less) weight %	Average particle diameter μm	Apparent specific gravity g/cm^3	Mixing amount parts by weight	Appearance	Haze %	Blocking resistance $\text{g}/100\text{cm}^2$	Scratch resistance (Δ Haze) %
Example 4	700	2	1.9	0.33	0.15	good	1.1	49	1.5
Example 5	700	2	3.5	0.33	0.15	good	1.8	14	0.8
Example 6	700	2	2.7	0.33	0.25	good	2.6	25	1.5
Example 7	700	2	2.7	0.33	0.50	nearly good	3.5	11	1.8
Comparative Example 2	700	2			0	good	0.3	77	0
Comparative Example 3	700	2	0.8	0.40	0.15	good	0.7	65	0.6
Comparative Example 4	700	2	4.5	0.29	0.30	bad	2.3	21	0.1
Comparative Example 5	700	2	1.8	0.13	0.20	bad	3.0	17	0.3
Comparative Example 6	700	2	2.0	0.31	0.15	good	1.1	37	15
Comparative Example 7	700	2	2.7	0.33	1.0	bad	8.6	0	2.2

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It will be apparent to those of ordinary skill in the art that various changes and modifications can be made in the present invention without departing from the spirit and scope therefrom. Furthermore, all patents, including Japanese Patent Application 06-219119, and scientific publications cited throughout this application are hereby incorporated by reference and relied upon.



CLAIMS

WHAT IS CLAIMED IS:

1. A method for producing a polyolefin composition, comprising the step of:
 - a) mixing
 - i) 100 parts by weight of polyolefin particles (I) having an average particle diameter of from 500 to 1300 μm and containing not more than 10% by weight of fine particles having a particle diameter of 300 μm or less and
 - ii) from 0.05 to 0.6 parts by weight of inorganic fine powder (II) having an average particle diameter of from 1 to 4 μm and an apparent specific gravity of from 0.2 to 0.5 g/cm^3 ; and
 - b) melt-kneading the mixture.
2. The method of claim 1, wherein the polyolefin particles (I) are polypropylene.
3. The method of claim 1, wherein the inorganic fine powder (II) is silica.
4. The method of claim 3, wherein the inorganic fine powder (II) has an average particle



diameter of from 1.5 to 4 μm and an apparent specific gravity of from 0.2 to 0.4 g/cm^3 .

5. The method of claim 4, wherein the amount of the inorganic fine powder (II) is from 0.1 to 0.3 parts by weight.

6. A method for producing a polyolefin film comprising the steps of:

a) mixing

i) 100 parts by weight of polyolefin particles (I) having an average particle diameter of from 500 to 1300 μm and containing not more than 10% by weight of fine particles having a particle diameter of 300 μm or less and

ii) 0.05 to 0.6 parts by weight of inorganic fine powder (II) having an average particle diameter of 1 to 4 μm and an apparent specific gravity of from 0.2 to 0.5 g/cm^3 ;

b) melt-kneading the mixture to produce a polyolefin composition; and

c) melt-extruding the polyolefin composition to form the polyolefin film.

7. The method of claim 6, wherein the polyolefin particles (I) are polypropylene.



8. The method of claim 6, wherein the inorganic fine powder (II) is silica.

9. The method of claim 8, wherein the inorganic fine powder (II) has an average particle diameter of from 1.5 to 4 μm and an apparent specific gravity of from 0.2 to 0.4 g/cm^3 .

10. The method of claim 9, wherein the amount of the inorganic fine powder (II) is from 0.1 to 0.3 parts by weight.