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[54] **METHOD FOR PREPARING A DIMENSIONALLY STABLE WAXED POLYETHYLENE SHEET**

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[52] **U.S. Cl.** .... **117/7**, 117/138.8 E, 117/168, 264/134, 264/136, 264/288

[51] **Int. Cl.** **B05c 3/107**

[58] **Field of Search** .... 264/134–137, 210 R, 288–289, 95; 117/7, 6, 138.8 E, 168; 156/229, 244; 118/33

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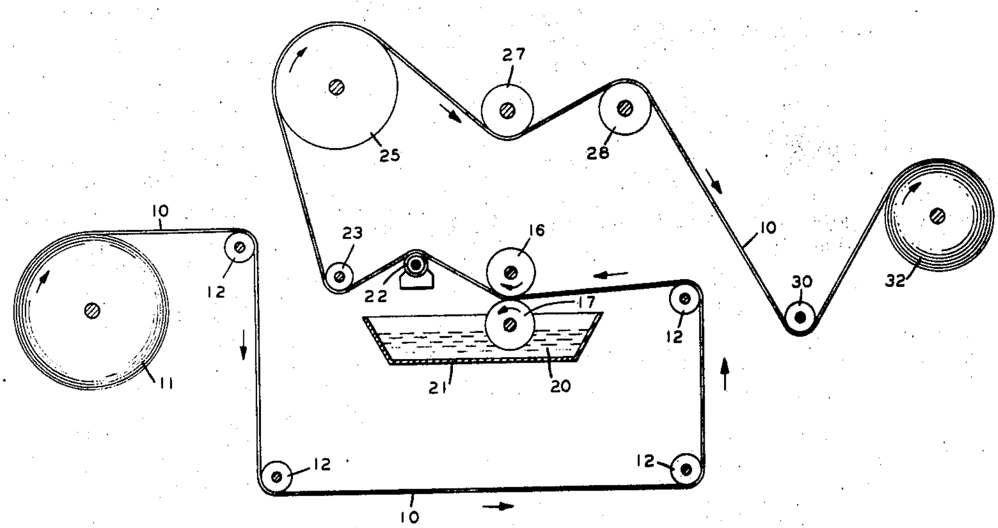
[57] **ABSTRACT**

This invention relates to a method for applying a wax coating to one or both sides of a polyethylene-based film which eliminates a hitherto troublesome problem of dimensional instability of the overwaxed film. More specifically, this invention provides a method for producing a waxed polyethylene film having a degree of dimensional stability heretofore not achieved.

7 Claims, 1 Drawing Figure



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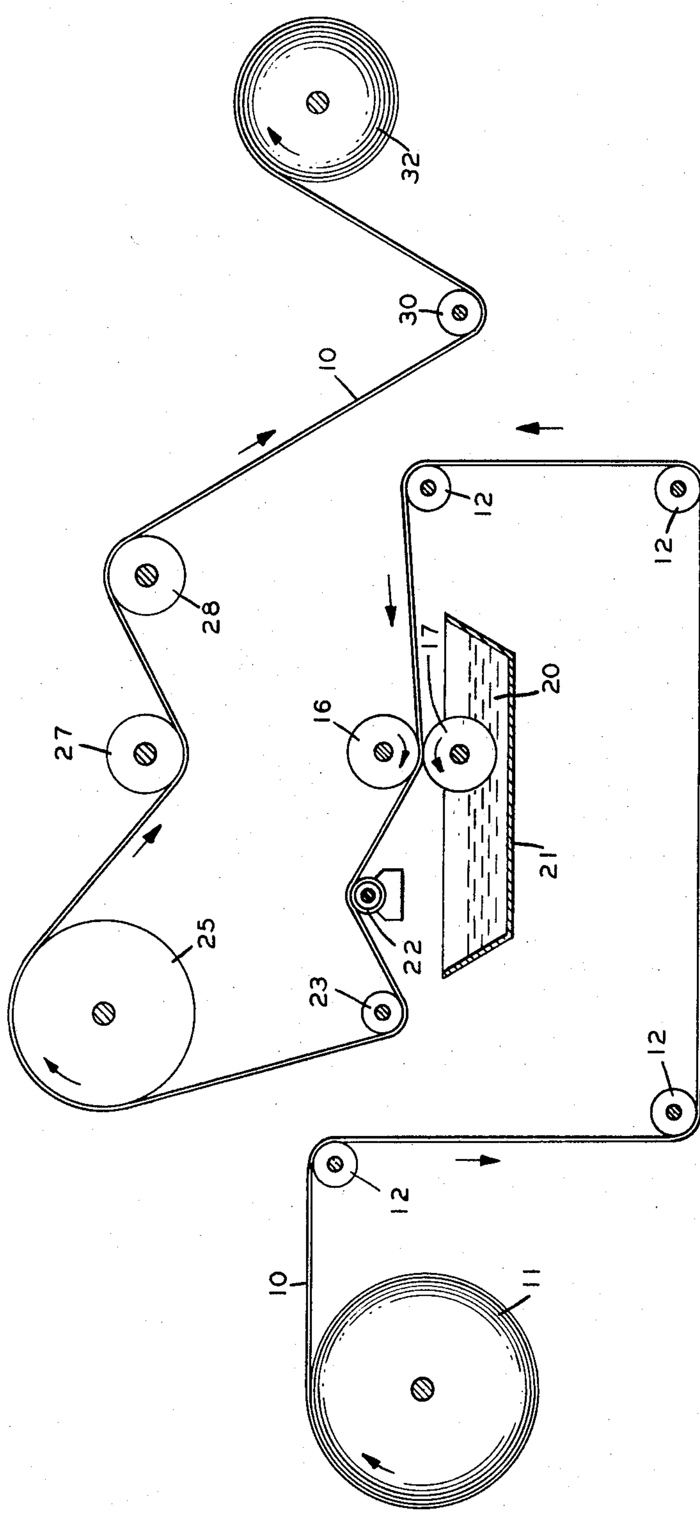

METHOD FOR PREPARING A DIMENSIONALLY STABLE WAXED POLYETHYLENE SHEET

BACKGROUND OF THE INVENTION

Polyethylene film bearing a superficial coating of a petroleum wax composition possesses excellent protective properties for a number of packaging applications, and is of particular value as an innerwrap sheet for the packaging of a variety of cereals and crackers. Unfortunately, the waxed polyethylene sheet material heretofore produced exhibited the unusual and vexatious property of growth or elongation in both directions of the sheet when stored under summer warehousing conditions for a period of time in roll form after the waxing operations were completed. The extent of elongation of the web was often sufficient to cause it to separate from the core of the stock roll and the resultant distortion of the roll stock made the web unsatisfactory for use on any automated food packing machinery. Growth or elongation of the waxed film often amounted to as much as 0.5 to 1.0 inches per foot of length and width of the web, or from about 4 percent to more than 8 percent.

The elongation which occurs upon storage of a waxed polyethylene film is relatively slow at room temperature (70°–75° F.) but the rate of elongation increases rapidly as the storage temperature is increased. Warehousing of the waxed film during summer months in which the temperature of normal storage areas may reach levels in the range of 100°–130° F. has hitherto been impossible due to the dimensional instability of such films when subjected to even moderately elevated temperatures of storage. It has been established that a waxed film which exhibits a dimensional change of more than 2–3 percent during storage will prove unsatisfactory for use on the high speed packaging machinery commonly used in a variety of packaging applications. It is therefore imperative for commercial acceptability that the elongation of the sheet be kept well within the above limits even when subjected to storage in warehouses which may reach summer temperatures in the range of 100°–130° F., and preferably the dimensional change should not exceed 1.5 percent under such conditions.

It is an object of this invention to make it possible to produce an overwaxed, polyethylene-based web which is dimensionally stable during storage and use in packaging applications.

It is a further object to produce an overwaxed polyethylene web of low water vapor permeability which is satisfactory for packaging of cereals, crackers and similar food items subject to degradation in the presence of atmospheric moisture, which web may be stored without substantial dimensional variation, even under conditions of moderately elevated temperatures, and utilized in the packaging of foodstuffs on high-speed packaging machinery.

Further objects will become apparent from a perusal of the following specification taken in connection with the accompanying drawing and attached claims.

SUMMARY OF THE INVENTION

It has now been found that growth or elongation of a waxed, polyethylene-base film while stored in roll form at temperatures up to 120°–130° F. may be eliminated or reduced to a negligible level by a unique waxing procedure whereby the polyethylene web is submitted to tension stresses during and immediately after the application of molten wax, sufficient to stretch the film by a controlled amount in the range of 5–20 percent of its original length. The required degree of stretch is impressed on the sheet immediately subsequent to the actual application of molten wax, the coated sheet is cooled to solidify the wax while the sheet is maintained in the stretched state and the coated web is formed into a roll and stored for use in packaging operations. When prepared in accordance with the process of this invention, the waxed sheet is dimensionally stable within the limit of ±2 percent even after extended periods of storage and exhibits very desirable protective properties as a packaging film. Preferably, the basic polyethylene film which is to be overcoated with a petroleum wax is first subjected to a surface treatment of the oxidative type which so modifies the surface layer of the film that it is more readily wet by the molten wax, which then spreads more evenly on the film surface to form a smooth overall coating of uniform thickness. Any of the conventional polyolefin surface treatments may be utilized, including corona discharge, flame treatment or an oxidative chemical treatment, all being carried out in known manner and not being a part of the present invention. The protective properties of the overwaxed film are, in large measure, dependent on an even distribution of the wax composition coating over the surface of the polymer film, which is generally enhanced by the surface treatment of the base film. For example, the moisture vapor transmission rate of a 1.5 mil (1 mil = 0.001 inch) high density polyethylene film is in the range of 5 –10 gm. per square meter per 24 hours. If this film is corona discharge treated and overwaxed with 4 lb. per ream (3,000 sq. ft.) of a 135° F. melting point paraffin wax, the moisture vapor transmission rate (MVTR) is reduced to between 0.5 and 1.0 gm/M^2/24 hrs., whereas a similar film overwaxed without prior corona discharge treatment will have a MVTR of between 2 and 4 gm/M^2/24 hrs. For this reason, the data herein presented are all based on films which have been subjected to an oxidative surface treatment prior to the overwaxing operation.

DESCRIPTION OF THE DRAWINGS

In the preferred embodiment of this invention, the stretching and waxing operation is carried out on apparatus such as that shown schematically in the accompanying drawing wherein a web of polyethylene **10** is unwound from stock roll **11**, led over idler rolls **12** and through a nip formed between a back-up roll **16** and an application roll **17**, which latter roll is rotatably mounted with the lower portion of the roll dipping in a bath of molten wax **20** maintained at the desired waxing temperature in a wax pan **21.** The applicator roll preferably has an etched, engraved or knurled surface which carries sufficient molten wax from the pan **21** up into contact with the web **10** to maintain a flooded nip where the sheet passes between the two rolls **16** and **17.** If desired, the rolls **16** and **17** may be so arranged that the web **10** dips in the wax bath so that both sides of the web receive a coating of the wax composition. After passing through the nip, the coated side (or coated sides) of the sheet may be passed over a heated metering rod or roll **22,** suitably a conventional wire-wound rod or other comparable device to remove any excess of wax and to smooth the wax film to a coating of even thickness on the web surface. If both sides of the sheet are coated, each side will be subjected to a metering operation, of course. The coated sheet after passing idler roll **23** is then successively passed into contact with a tempering drum **25** maintained at a few degrees below the melting point of the wax, and then to the surface of cooling drums **27** and **28** which complete the solidification and hardening of the wax coating and finally, after passage over idler roller **30**, the wax coated sheet is wound into roll form on a wind-up roll **32**.

In order to impart a controlled degree of stretch to the polymer web during the critical portion of its travel through the waxing operation, the applicator and back-up rolls **17** and **16** are provided with a first variable speed driving means, not shown, and the tempering drum **25**, the cooling drums **27** and **28** and wind-up roll **32** are provided with a second variable speed driving means. With such provision, the tempering and cooling drums and the wind-up roll may be run, as desired, at variably higher speeds than the applicator and back-up roll, thus imparting a preselected degree of stretch to the waxed film in the critical area between the wax-applying nip and the tempering and cooling drums. It has been found that, in this particular phase of the operation, the film will attain a temperature above the melting point of the wax in the wax pan and it is important to the dimensional stability of the final, wax-coated film that the desired degree of stretch is applied to the film while it is heated in a temperature range between the wax melting point (usually at least 130° F.) and a maximum temperature which approaches the softening point of the polyethylene. The softening temperature of polyethylene varies depending on the molecular weight and density of the film, low density polyethylene generally softening in the vicinity of about 220°–230° F. while the softening point of high density polyethylene may range as high as 260° F. or slightly above. It is therefore important that the temperature of the waxing bath, the applicator roll, back-up roll and the metering roll be controlled to, first, raise the temperature of the film into the desired temperature range and then to maintain it within this range during the waxing and stretching operations. Generally, the heated, molten wax provides sufficient heat to achieve the above-described desired result, but it is to be understood that the film may be partially preheated before its delivery to the waxing nip as, for example, by utilizing one or more of the idler rolls **12** as a preheating roll for the film.

DESCRIPTION OF A PREFERRED EMBODIMENT OF THE INVENTION

To demonstrate, on the above-described apparatus, the effect of stretching a high density polyethylene film to varying degrees during the waxing operation, a 1.5 mil film of polyethylene blown from Chemplex 6108 high density polyethylene resin (available from Chemplex, Rolling Meadows, Ill.) was waxed with 4.8 lbs. of a 135°–140° F. melting point paraffin wax maintained at a temperature of 210° F. The film was fed to the applicator nip at 50 feet per minute, the chill roll and wind-up rolls being operated at speeds sufficient to give the various degrees of stretch in the waxing area indicated in the following table. The resulting wax coated polyethylene films were then stored at 120° F. for eight days and tested for dimensional stability after one day, four days and eight days of such storage. The test data are recorded in the following Table I.

TABLE I

% ELONGATION OF WAXED POLYETHYLENE STORED AT 120° F.

% Stretch of PE During Waxing	% Elongation After Storage 1 day	4 days	8 days
0% (Control)	6 %	7 %	7.7%
8%	2.4%	3.4%	3.9%
12%	0	0.4%	0.8%
14%	0.7%	1.4%	1.8%
23%	−2.1%	−1.4%	−0.8%

The data in the above Table I clearly indicate that high density polyethylene which, during waxing at a speed of 50 feet per minute at a wax temperature of 210° F., is stretched to an extent of between 10 percent and 20 percent, yields a waxed polymer film which is well within the limits of satisfactory dimensional stability (maximum dimensional change of less than about 2 percent) for commercial utilization on high speed packaging machinery, even when the coated film is stored under rather extreme summertime warehouse temperatures. It is also quite evident that the film which was waxed without stretching (control) did not exhibit the required degree of dimensional stability.

It has previously been stated that the application of a thin coating of wax on a polyethylene film greatly reduces the water vapor transmission rate of the film and that the resulting waxed polymer film therefore has great utility in the packaging of items susceptible to the deteriorative effects of moisture.

The amount of wax coating required will depend on the particular protective requirements of the specific packaging situation and will also vary somewhat depending on the type and caliper of the polyethylene base film and on the particular wax composition utilized. It is to be understood that a wide variety of wax compositions based primarily on petroleum wax may be utilized. Both paraffin wax and microcrystalline petroleum wax may be used, as well as blends of either or both of these materials with such other known wax composition components as polyolefins, ethylene-vinyl acetate co-polymers, natural and synthetic resins and the like. Typical wax compositions suitable for the present invention would include paraffin wax containing from 1 percent to 50 percent of ethylene vinyl acetate co-polymer, blends of paraffin wax with minor percentages of microcrystalline wax and/or polyethylene, polyisobutylene, rosin, natural and synthetic rubber, hydrogenated rosin esters and the like. Generally, between 1 lb. of wax composition and about 10 lbs. of wax composition per ream (3,000 sq. ft.) of polyethylene applied to either one or both surfaces of the web is quite adequate to provide the degree of protection desired in the packaging film. Greater amounts of the coating could be applied but are less economical and lesser amounts are generally ineffective. A preferred range of coating weight is from 3 to 6 lbs. per ream of the base web.

The following table presents data comparing the water vapor transmission rate of an unwaxed high density polyehtylene film (1.5 mil in caliper) made from Chemplex 6,108 polyethylene resin with the MVTR of a similar film overwaxed with 4.8 lbs./ream of 135–140° F. melting point paraffin and with a similar waxed film which had been stretched to the extent of 12 percent during the waxing operation in accord with the process of this invention as hereinbefore described.

Moisture vapor transmission rate was determined on all samples according to the procedure set forth in TAPPI standard method number T-464-*m*-45.

The waxed films were conditioned for eight hours at 120° F. prior to conducting the MVTR tests. Values of MVTR are reported in grams per square meter per 24 hours.

TABLE II

MVTR OF WAXED AND UNWAXED POLYETHYLENE

Film: 1.5 mil caliper High Density Polyethylene

Coating: 4.8 lbs./ream of Paraffin Wax (135-40° F.M.P.)

The data in the above table confirm the fact that overwaxing of polyethylene film produces a substantial reduction in MVTR and also demonstrate that the film waxed in accord with the process of this invention also exhibits a substantially lower MVTR than unwaxed polyethylene. It has previously been demonstrated by data in Table I that the stretched waxed film has a satisfactory dimensional stability, while the waxed, unstretched film does not.

The phenomenon of growth or elongation after waxing is common to both high density and low density polyethylene film and is also demonstrated, in a far lesser degree, by polypropylene film. The following table illustrates the elongation which results when unstretched polyolefin films from a variety of sources are overwaxed on one side with 4.0 to 4.5 lbs./ream of a paraffin wax of 135°–140° F. melting point at a temperature of 170° F. and stored for periods of up to three days at temperatures from 100° F. to 120° F.

In the following table, HDPE refers to high density polyethylene, LDPE refers to low density polyethylene and PP refers to polypropylene.

TABLE III

ELONGATION OF WAXED POLYOLEFINS STORED AT ELEVATED TEMPERATURE

	Polyolefin Film			*Wax* Weight	Storage		Percent Elongation
Sample	Film	Type	Caliper Caliper	lbs./ream	Time	Temp. ° F.	During Storage Percent
A	HDPE	Cast	2	4.0	3 days	100	4.5
B	HDPE	Cast	1.5	4.0	3 days	100	4.8
C	HDPE	Cast		4.0	3 days	100	6.2
D	HDPE	Blown	1.5	4.0	60 hours	100	5.4
					24 hours	110	5.6
					16 hours	120	7.7
E	HDPE	Cast	2	4.0	24 hours	120	5.4
F	HDPE	Cast	1.5	4.0	24 hours	120	3.3
G	HDPE	Cast	1.5	4.0	24 hours	120	5.7
H	LDPE	Cast	1.5	4.5	3 days	110	4.3
I	PP	Cast	2	4.0	3 days	100	0.3

Sample A is a high density polyethylene film designated as Philjo 606W.

Sample B is a high density polyethylene film designated as Philjo 670W.

Sample I is a polypropylene film designated as Philjo 410.

Samples A, B and I are obtainable from Phillips Joanna, Division of Joanna Western Mill Company of Ladd, Illinois.

Sample C is high density polyethylene film obtainable from Allied Chemical Corporation, Plastic Film Department, Chicago, Ill.

Sample D is a blown high density polyethylene film prepared from pelletized resin designated Chemplex 6,108 and obtainable from Chemplex, Rolling Meadows, Ill.

Samples E and F are high density cast polyethylene films prepared from pelletized resins designated by the numbers 7,140 and 7,150, respectively, which are available from E. I. duPont de Nemours & Co., Electrochemicals Department, Chicago, Ill.

Sample G, a cast high density polyethylene film, was prepared from pelletized resin designated Amoco 650-B4, which is obtainable from Amoco Chemical Corporation, Chicago, Ill.

Sample H, a cast low density polyethylene film, was prepared from pelletized low density polyethylene, designated as USI., N.A. 222, obtained from U. S. Industrial Chemicals Company, Oak Brook, Ill.

The data in the above table clearly indicate that unstretched waxed polyethylene film, prepared either as a blown film from a tubing type extruder or as a cast film from a slot die extruder, is subject to a substantial elongation as a result of storage at moderately elevated temperatures. Both low density polyethylene film and high density polyethylene film exhibit this peculiar characteristic in a very substantial degree, whereas polypropylene film shows only minimal elongation after waxing. Data for Sample D, above, indicate that storage temperature severely affects both the rate and extent of the elongation or growth of the waxed film, since 16 hours of storage at 120° F. results in a greater growth than 60 hours of storage at 100° F. Further tests have demonstrated that at 120° F. storage temperature, the waxed film elongates by 5 percent in about an hour, whereas at 110° F., a 5 percent elongation requires about 10 hours and at 100° F., an equivalent elongation results in about 30 hours. At normal room temperature of 73° F., the growth rate is very slow, 200 hours of storage resulting in only 0.5 percent elongation of the film. The rate of elongation of the waxed film in storage under summer warehousing conditions (90° F. to 130° F.) is most rapid in the freshly waxed film, between 60 percent and 80 percent of the ultimate growth occuring in the first 10–20 hours. Growth tests have been carried to 500 hours at 100° F., 110° F. and 120° F., final figures on the percentage elongation of the waxed film at the end of the test period being 8 percent, 8.5 percent and 9.6 percent, respectively.

The degree to which a given polyethylene film must be stretched during the waxing operation in order to achieve the maximum degree of dimensional stability during storage and use of the waxed film is dependent on a number of factors, including the type and degree of crystallinity of the film, the temperature which the film attains during the waxing and stretching operation and the length of time of exposure of the film to the molten wax before the waxed film is cooled sufficiently to solidify the wax.

It has been determined, for example, that if a polyethylene is subjected to a waxing and stretching operation in which the film fails to reach a temperature of at least 130° F. during the waxing operation, the waxed film will show cold stretch marks and will have the desired degree of dimensional stability only if, during the waxing operation, the film is stretched by an amount in excess of 12 percent and preferably is stretched by about 20 percent. It is generally undesirable to stretch the film longitudinally to a degree approaching 20 percent, because "necking in" or a reduction of the width of the film tends to occur under these conditions, and this is undesirable.

It is greatly preferable to make sure that the film is heated to a temperature above 130° F., in which case the degree of stretch which must be imparted to the film during waxing to achieve satisfactory dimensional stability will seldom exceed 15 percent, and may even be as little as 6–8 percent, as will be discussed hereinafter. Variation of the temperature to which the film is heated between the limits of 130° F. and about 210° F. has a relatively small effect on the amount of stretching required for dimensional stability of the waxed film. It has been found, however, that the length of time of contact of molten wax with the film during the waxing and stretching operations has a profound effect on the degree of stretch which must be imparted to the polyethylene film to achieve optimum results. For example, if the polyethylene web is coated with a film of molten wax which is maintained in liquid state for as much as a full minute before chilling the sheet to set the wax, the waxed web need be stretched only by between 5 percent and 9 percent while the wax is molten in order to achieve optimum dimensional stability, so that the overwaxed sheet will change its dimensions by less than 2 percent even under extended storage under summer warehousing conditions.

By contrast, if polyethylene is waxed at commercially desirable speeds on high-speed waxing equipment such as that shown in the accompanying drawing, the time required for the flooding of the polyolefin web with molten wax, the metering off of excess wax, the stretching of the waxed film and finally the chilling of the molten wax into the solid state may be only a fraction of a second. Under these conditions, it has been found that the polyolefin web must be stretched as much as 10 to 18 percent during the waxing operation in order to achieve a dimensional stability of the product within the desired ranges. The effect of linear speed of the waxing equipment (inversely proportional to time of contact of the polyolefin web with molten wax) is shown in the following Table IV, wherein are shown data on dimensional stability of waxed polyethylene

webs which were subjected to varying degrees of stretching during the waxing operation. In each case, the wax was a 135°–140° F. melting paraffin, the bath was maintained at 220° F., the polyethylene web was a high density film of 1.5 mils in caliper, and the waxed sheet was stored for 10 days at 120° F. before the dimensional changes were obtained. Dimensional changes are measured in the direction of machine travel.

TABLE IV

EFFECT OF WAXER SPEED ON DIMENSIONAL STABILITY OF WAXED FILM

Web Speed in feet/min.	% Stretch imparted to film during waxing	% Change in waxed film length after storage 10 days at 120° F.
5	8%	0.2 decrease
50	8%	4.0 increase
400	8%	7.4 increase
100	10%	2.8 increase
200	10%	5.8 increase
300	10%	6.4 increase
400	10%	7.2 increase
50	15%	0.4 increase
400	15%	0.9 increase

From the above data it is evident that the polyethylene web need be subjected to a higher degree of stretching to achieve dimensional stability of the waxed sheet if the rate of waxer speed is increased or, conversely, if the time of contact of the polyethylene with molten wax is decreased. As previously stated, the minimum degree of stretching of polyethylene during the waxing operation which will result in a satisfactorily dimensionally stable sheet is in the range of 5–8 percent. Since this minimum stretch can be utilized only when the wax coating is maintained in a molten state for a period of time ranging upwards of 30 seconds or so, production of waxed polyethylene sheet material on a commercial scale under these conditions would be subject to severe economic disadvantage. In actual practice, then, the operation is preferably carried out on waxing equipment which coats the polyethylene web at several hundred feet per minute. Since this effectively reduces the dwell time of the web in contact with molten wax to a fraction of a second, it is necessary to increase the degree of stretching of the polyethylene web to compensate for the short dwell time. It has been found that, at waxing speeds up to and above 500 feet per minute, if the polyethylene web is stretched during the waxing operation to the extent of 10–18 percent, the resulting waxed sheet will show a dimensional stability well within the desired range of no more than 2% change under summer warehousing conditions. These results have been found to apply to polyethylene sheets of calipers ranging from about 0.0005 in. to about 0.005 in. For most packaging operations carried out on high-speed automatic packaging equipment, films of from about 0.001 to 0.0025 in. thickness are preferred. An optimum set of operating conditions for waxing 1.5 mil film of high density polyethylene film with a crystalline petroleum wax of 135°–140° F. melting point include maintaining the wax bath temperature at 215–220° F., flooding the web with wax at 400–450 feet per minute, metering off the excess wax with a conventional wire wound rod to leave 4.5 lbs. of wax on the web, and stretching the web during the waxing operation as hereinbefore described by the amount of 12–15 percent. The resulting sheet has excellent dimensional stability and very low water vapor permeability. It is a superior sheet for use as a cereal inner wrap.

The invention having now been disclosed and described in detail, it is apparent that various modifications thereof are possible without departing from the spirit thereof. Therefore, no limitations on the invention are intended except as specifically set forth in the appended claims.

We claim:

1. A method for obtaining a substantially dimensionally stable, waxed polyethylene sheet which comprises:

a. passing a polyethylene film having a thickness of between 0.0005 and 0.005 inches into contact with a molten petroleum wax composition maintained at a temperature sufficiently above 130° F. to allow said film to reach a temperature of between 130° F. and the softening point of said film,

b. metering any excess wax from said film to leave on at least one surface of said film between 1 lb. and 10 lbs. of wax composition evenly distributed on each ream of 3,000 sq. ft. of said film,

c. stretching said film in the machine direction while in contact with said molten wax composition by an amount between 5 and 20 percent of the original length of said film, and

d. cooling said waxed and stretched film to solidify said wax composition coating.

2. A method according to claim **1** wherein said contacting, metering, stretching and cooling steps are carried out at a speed between 200 and 500 feet per minute and said stretching is carried out to the extent of between 10 and 18 percent of the original film length.

3. A method according to claim **2** wherein the weight of said wax composition after said metering step is between 3 and 6 lbs. per ream.

4. A method according to claim **3** wherein said polyethylene film is between 0.001 in. and 0.0025 in. in thickness.

5. A method according to claim **4** wherein said polyethylene film has a surface which is treated to be receptive to fluid coatings.

6. A method for obtaining a dimensionally stable wax-coated polyethylene web suitable for use in high-speed packaging operations which comprises applying a molten petroleum wax composition to at least one surface of a polyethylene web having a thickness of between 0.0005 in. and 0.005 in., maintaining said wax coating in a molten state and said polyethylene web at a temperature between about 130° F. and the softening temperature of the polyethylene while mechanically stretching said coated polyethylene web in a machine direction by an amount between about 5 percent and 20 percent, and subsequently chilling said coated and stretched web to complete solidification of said wax composition coating.

7. A method according to claim **6** wherein said wax composition is applied to a surface treated polyethylene web traveling at between about 200 and 500 feet per minute, said coated polyethylene web being stretched by an amount between 12 percent and 15 percent.

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UNITED STATES PATENT OFFICE
CERTIFICATE OF CORRECTION

Patent No. 3,751,281 Dated August 7, 1973

Inventor(s) James Thomas Peterson and Mitsuzo Shida

It is certified that error appears in the above-identified patent and that said Letters Patent are hereby corrected as shown below:

In Column 5, in Table II, between line 14, which reads "F.M.P.)" and line 17, which starts "The data in the above table ...", insert the following tabular data:

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	MVTR in $Gm/M^2/24$ Hours
Uncoated Polyethylene Film	6.0
Waxed, Unstretched Polyethylene Film	0.62-1.0
Waxed Polyethylene Film, Stretched 12% During Waxing	0.50-1.5 --

In Column 5, in Table III, in the fourth column of the table, delete one instance of the word "Caliper" from the column heading and add -- mil -- after each of the data in the column.

Signed and sealed this 27th day of August 1974.

(SEAL)
Attest:

McCOY M. GIBSON, JR.
Attesting Officer

C. MARSHALL DANN
Commissioner of Patents