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WAXED PAPER

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This invention relates to a new and improved waxed paper, to be used for wrapping foods and other products.

One of the objects of the invention is to coat or impregnate paper with a wax of improved sealing strength.

Another object of the invention is to provide paper which has been coated or impregnated with improved petroleum wax.

Other objects of my invention will be set forth in the following description which illustrates preferred embodiments thereof, it being understood that the above statement of the objects of my invention is intended generally to explain the same without limiting it in any manner.

This application is a continuation in part of application Serial No. 186,978 filed by me in the United States Patent Office on January 26, 1938, and of my application Serial No. 207,378 filed on May 11, 1938. Reference is made to the disclosures of said previously filed applications, with the same force and effect as though the disclosures thereof had been specifically repeated herein.

Petroleum wax has a high resistance to water vapor, and paper which has been coated or impregnated with such wax, is highly effective in excluding or retaining moisture from or in the product being wrapped. A good example is waxed bread wrapper, consisting of a sulphite sheet of 20 to 23 lbs. weight to the ream which has been waxed up to 33 to 37 lbs. to the ream. In the manufacture, part of the wax soaks into the paper and part remains on the surface. Hence this sheet is typical of what is known as the "wet-waxed" sheet and it possesses self-sealing properties under the application of heat which melts or softens the wax, and subsequent cooling which solidifies the wax.

The self-sealing is carried out in well-known continuous machines, when the paper is used for wrapping loaves of bread.

The waxes suitable for sealing must have sufficient coherence, toughness and tensile strength, so that the seal remains intact when the loaves are handled and transported. Fully refined petroleum wax made by sweating, having a tensile strength of two hundred to four hundred pounds to the square inch at 70° F., and a melting point of 127° F. to 140° F. by the English method (same as ASTM D-87-22) is used almost exclusively for this purpose.

The sealing effectiveness, even for the petroleum waxes having a tensile strength of four hundred pounds to the square inch, never has been

entirely satisfactory, especially for such paper as is used for wrapping soft products. Hence it is important that the sealing strength be increased above that of the fully refined petroleum wax or waxes.

I have found that the incorporation of small amounts of selected "I. G." waxes into the usual fully refined petroleum wax or waxes increases both the tensile and sealing strength, but the latter in much greater percentage than the former.

One of the "I. G." waxes which I prefer to use has a melting point of 176°-181° F., an acid value of 15-20, an ester value of 140-155, and a light yellow color. It contains only about 8-10% of unsaponifiable matter. It is readily saponified by dissolving soap and either caustic soda or caustic potash jointly in water, and adding the melted "I. G." wax to the boiling solution, with vigorous stirring. Said "I. G." wax can be melted in a solution of soap and caustic alkali in water, and additional boiling water can be added slowly. Smooth emulsions can thus be formed. The method of making the "I. G." types of wax is disclosed in British Patent No. 368,425. Some of the "I. G." waxes which I prefer to use are known in the market as "I. G. Wax E" and "I. G. Wax B" and they are described in Bulletin G-47-1 issued by General Dyestuff Corporation of 230 Fifth Avenue, New York, N. Y.

Other "I. G." waxes are not useful for increasing the sealing power of the wax composition. Some "I. G." waxes are unsuitable because they are not compatible with the fully refined petroleum wax and hence cannot be incorporated uniformly into petroleum wax, while the preferred "I. G." waxes can be thus uniformly incorporated. When the improved composition is repeatedly melted and solidified, the preferred and compatible "I. G." waxes do not separate from the petroleum wax or waxes. The composition is either a true and uniform and stable solution of the "I. G." wax in the petroleum wax, or a uniform and stable dispersion. The "I. G. Wax E" and "I. G. Wax B" when used in percentages less than 0.3%, impart a slight odor to the wax, but the odor is not detectable in the final waxed paper. The "I. G. Wax S" can also be incorporated uniformly into paraffin wax so as to form a heat-stable solution or dispersion which is uniform, but it has too much odor for some purposes. Said "S" wax has a melting point of 180°-183° F., an acid value of 142-152, an ester value of 25-35, and a very pale yellow color.

These products are used preferably in combination with the fully-refined petroleum wax.

The "I. G." waxes which may be used for improving self-sealing, and for decreasing blocking, in addition to "I. G. Wax E" and "I. G. Wax B," are "I. G. Wax S" (melting point 180°-183° F.), "I. G. Wax V" (melting point 122° F.). However, the types which are preferred are the "I. G. Wax E," "I. G. Wax FP" and "I. G. Wax KP." The "B" wax has a melting point of 167° F.-171° F.

I have found it particularly advantageous to use the wax known as "I. G. Wax KP" when this "KP" wax is added to the fully refined petroleum wax having a melting point of 125-150° F. (English), so that the mixture contains from $\frac{1}{16}$ % to $\frac{1}{8}$ % of the "KP" wax. Superior sealing properties and gloss are secured. I have made the surprising discovery that the sealing strength of the fully refined wax is increased 50% and even more by the use of as little as $\frac{1}{16}$ % of the "KP" wax. Likewise, the mixture with the "KP" wax can be used advantageously in large scale production.

This "KP" wax can be identified by the following properties:

Melting point-----°F--	175-180
Acid number-----	25- 35
Ester number-----	100-120
Saponification number-----	130-145
Unsaponifiable matter-----per cent--	12- 14
Specific gravity at 70° F-----	1.02 to 1.03

The "I. G. Wax FP" is an esterified montan wax and it has the following properties: melting point 175° F. to 180° F., and number 20 to 30, ester number 115-125, saponification number 135 to 155, unsaponifiable matter 10% to 30%, and specific gravity 1.0 to 1.01 at a temperature of about 70° F.

When sulphite sheet paper is wet waxed with a fully refined petroleum wax whose melting point is 132° F. (English), the sealing strength was seven grams, according to the method of testing hereinafter stated. When the same sulphite sheet was wet waxed with a composition which had 99.8% of said fully refined wax, and $\frac{1}{16}$ % of the "KP" wax, the sealing strength was twelve grams, showing an increase of the original sealing strength of 70%.

In incorporating the compatible "I. G." wax with the petroleum wax which is ordinarily used, the latter is melted in a tank by means of steam heat to a temperature of 150° to 180° F. The "I. G." wax is melted in a separate container and it is added slowly to the molten petroleum wax while the latter is stirred mechanically or while it is stirred by being blown with air. Since the "I. G." wax is compatible, it goes into uniform solution or into such a state of uniform dispersion that it does not settle out upon standing and solidifying. The mixture is maintained molten at 150° F.-180° F. while said solution or dispersion is being made.

Specific examples may be given as follows: The petroleum wax used was a fully refined product having a melting point of 132° F. (English) and a tensile strength of 252 pounds to the square inch at 70° F. About 0.5% "I. G. Wax E" dissolved in the petroleum wax increased the tensile strength at 320 pounds, or about 27%, while the sealing strength, as tested by the method to be described below, increased 68%. About 0.3% of the "I. G. Wax E" increased the sealing strength 39%, while 1.0% of the "I. G.

Wax E" increased the sealing strength 92%. These proportions of the "I. G. Wax E" are by weight.

The method of making the sealing strength tests was as follows: Sections of a uniform sulphite sheet were waxed respectively with the petroleum wax alone and with the various mixtures above stated, so that each section had the same amount of wax per unit of area. Then strips of said sections one inch in width were doubled and each doubled strip was pressed against a beaker containing hot water at a temperature of 190° F. At this temperature, the coating of each doubled strip melted, and the two halves of each strip adhered to each other by means of the coating. Then the doubled strips were removed from the surface of the beaker and permitted to cool in the air. After ageing at room temperature of 77° F. for two hours, the doubled strips were tested as follows: The ends of each strip were separated for an inch, one of the separated ends was then inserted into a clamp and the other separated end was attached to a thread which extended horizontally over a metal bar and then vertically to a small cup to which the thread was attached. Bird shot then was poured into the cup slowly until the connected parts of the strip started to separate or peel apart at a slow rate. The weight of the cup and its contents which was necessary to separate the sealed parts of the strips, was recorded as the sealing strength.

Another example of the effect of the "I. G. Wax E" on the sealing strength of petroleum wax is as follows: The petroleum wax had a tensile strength of 377 pounds to the square inch at 70° F., and a sealing strength of 11.4 gms. When 0.5% of "I. G. Wax E" was incorporated in solution with the petroleum wax, the tensile strength was 460 pounds, an increase of 22%, while the sealing strength was 19 grams, an increase of 66%.

In a wax paper plant, paper was waxed on the large machines, first using straight petroleum wax, then using a mixture consisting of 99.5% petroleum wax and 0.5% "I. G. Wax E." The rolls of paper were then taken to a bakery where loaves of bread were wrapped and sealed on the commercial machines, using both the paper waxed with the straight petroleum wax and that waxed with said mixture of petroleum wax and "I. G. Wax E." Comparing the two products, it was plainly evident that the mixture containing the "I. G. Wax E" was much stronger than the straight wax.

A mixture of 0.3% wax "E," 0.5% amorphous petroleum wax (whose melting point was 160° F.-165° F. by the drop method) and of fully refined paraffin wax (petroleum wax) having a melting point of 132° F. by the English method, was used to wax paper. This gave even better sealing results than the previously mentioned composition consisting only of fully refined paraffin wax and "E" wax.

The amorphous petroleum wax can be present in the composition in the proportion of 0.1% to 10%. Whenever I specify a minimum proportion as "substantially," I can use less than said minimum proportion.

In some cases it is desirable to soften the wax for special sheets, in which case the incorporation of 0.1% to 10% of amorphous wax has the desired effect.

Instead of using amorphous petroleum wax or amorphous paraffin wax for any of the purposes

specified, I can use from 0.1% to 5% of the product known as "Vistanex" (No. 6 or No. 7) instead of using from 0.1% to 10% of said amorphous wax. Said "Vistanex" is described in certain patents and publications as follows:

French Patent No. 740,407; U. S. Patent No. 2,054,115; Rubber Age (New York Edition), vol. 41, p. 102 (1937). This product is prepared by polymerizing iso-olefines, and particularly isobutylene. The molecular weight of "Vistanex" varies, depending upon the method of preparation.

The No. 6 and No. 7 "Vistanex" are substantially colorless, and their viscosity is close to that of unvulcanized crepe rubber at a room temperature of 70° F.

The paraffin wax or petroleum wax is melted and kept at a temperature of about 200° F. while the "Vistanex" is added in small pieces with vigorous stirring, until the "Vistanex" has been uniformly dissolved in or dispersed in the molten wax. The composition is then allowed to cool and solidify. It is applied in the molten condition to the paper, at a temperature of 150° F.-200° F.

Hence, whenever I refer to the use of amorphous petroleum wax, I include the use of said "Vistanex" as an equivalent.

These "I. G." waxes are prepared by esterifying or saponifying waxes, and particularly montan wax (preferably bleached and oxidized) with alcohols. These alcohols may be higher alcohols, such as montanyl alcohol. Some of these waxes are described in British Patent No. 368,425 which discloses the use of lower alcohols, such as anhydrous ethyl alcohol. They can be prepared so as to simulate beeswax and other waxes in certain physical properties. They are described in vol. 2, pp. 690 and 691, of the Supplement to Thorpe's "Dictionary of Applied Chemistry," published in 1935 (which refers to various technical publications which describe said "I. G." waxes), and also in vol. I, p. 668, of said supplement.

When I refer to petroleum wax, I include said material if derived from sources other than petroleum, and analogous waxes.

Likewise, said British Patent No. 368,425 and the other patents referred to therein, disclose products made from carnauba wax and other waxes, which are equivalent to those made from montan wax. Hence, whenever I refer to montan wax in the claims, I include such equivalent products.

The so-called amorphous wax which has previously been referred to and which has been referred to in my applications Serial No. 186,978 and No. 207,378, is not truly amorphous, because it contains crystalline wax. This amorphous wax can be defined as a plastic, waxy, hydrocarbon material which is secured from petroleum. When small solid blocks of amorphous wax are squeezed together at various temperatures, the specimens flatten without fracturing if the temperature is 40° or higher. The fully refined waxes made by sweating and whose melting point is 120° F. or higher, compress without fracturing only when their temperature is above 80° F.

The "Vistanex" can be used as a softener for the composition which contains the "KP" wax. Other softeners which I can use are the so-called amorphous wax, and resins, such as ester gum or "Santo," which is a polymerized petroleum hydrocarbon. These resins or resin substitutes

can be incorporated up to 20% by weight. The resins or resin substitutes improve the transparency of the composition.

The improved wet-waxing composition consists substantially of a major proportion of fully refined petroleum wax, with which .05%-5% of a selected wax is intimately mixed. This selected wax is an esterified product of montan wax. The selected waxes have melting points between 175°-180° F., acid numbers between 25-30, ester numbers between 100-120, saponification numbers of substantially 130-155, and unsaponifiable matter substantially in the proportion of 12%-14%. These figures are used for identifying the range of selected "I. G." waxes.

Therefore, the compositions which can be made according to the invention include a composition which comprises petroleum wax and an additional wax which is uniformly distributed in said petroleum wax and which has a higher melting point than the petroleum wax, said additional wax being an esterified product of montan wax and having a melting point of at least substantially 167° F., said composition containing said additional wax in a proportion of at least substantially 0.05% to 2%. Said composition also contains a polymerized iso-butylene product which is dispersible in the petroleum wax, in the proportion of at least substantially 0.1% to 5%, and a resin in a proportion of up to 20%.

I claim:

1. A wet-waxing composition consisting substantially of a major proportion of fully refined petroleum wax and an additional wax, the weight of the additional wax in said composition being substantially .05% to 2% of the weight of the entire composition, said additional wax being an esterified product of montan wax and having a melting point higher than the melting point of said petroleum wax, said additional wax being uniformly distributed in the composition, said additional wax being selected from a class which has acid numbers of substantially 25 to 30, ester numbers of substantially 100 to 120, and saponification numbers of substantially 130 to 155.

2. A wet-waxing composition consisting substantially of a major proportion of fully refined petroleum wax and an additional wax, the weight of the additional wax in said composition being substantially .05% to 2% of the weight of the entire composition, said additional wax being an esterified product of montan wax and having a melting point higher than the melting point of said petroleum wax, said additional wax being uniformly distributed in the composition, said additional wax being selected from a class which has acid numbers of substantially 25 to 30, ester numbers of substantially 100 to 120, saponification numbers of substantially 130 to 155, unsaponifiable matter substantially in the proportion of 12% to 14%.

3. A wet-waxing composition consisting substantially of a major proportion of fully refined petroleum wax and an additional wax, the weight of the additional wax in said composition being substantially .05% to 2% of the weight of the entire composition, said additional wax being an esterified product of montan wax and having a melting point higher than the melting point of said petroleum wax, said additional wax being uniformly distributed in the composition, said additional wax being selected from a class which has acid numbers of substantially 25 to 30, ester numbers of substantially 100 to 120, and saponi-

fication numbers of substantially 130 to 155, said composition also containing uniformly distributed amorphous petroleum wax in the proportion of substantially 0.1% to 10% of the weight of the composition.

4. A wet-waxing composition consisting substantially of a major proportion of fully refined petroleum wax and an additional wax which is uniformly distributed in the composition, the weight of the additional wax in said composition being substantially .05% to 2% of the weight of the entire composition, said additional wax being an esterified product of montan wax and being selected from a class of waxes as follows:

	Melting point	Acid No.	Ester No.
a	176° F.-181° F.	15-20	140-155
b	180° F.-183° F.	142-152	25-35
c	167° F.-171° F.		
d	122° F.		
e	180° F.-183° F.		
f	175° F.-180° F.	25-35	100-120

5. A wet-waxing composition consisting substantially of a major proportion of fully refined petroleum wax, uniformly distributed amorphous petroleum wax in the proportion of substantial-

ly .10% to 10% of the weight of the composition, an additional wax which is uniformly distributed in the composition, the weight of the additional wax in said composition being substantially .05% to 2% of the weight of the entire composition, said additional wax being an esterified product of montan wax and being selected from a class of waxes as follows:

	Melting point	Acid No.	Ester No.
a	176° F.-181° F.	15- 20	140-155
b	180° F.-183° F.	142-152	25-35
c	167° F.-171° F.		
d	122° F.		
e	180° F.-183° F.		
f	175° F.-180° F.	25- 35	100-120

6. A composition according to claim 4 which also contains substantially 0.1% to 5% of a polymerized iso-butylene product which is dispersible in the petroleum wax.

7. A composition according to claim 4 which also contains substantially 0.1% to 5% of a polymerized iso-butylene product which is dispersible in the petroleum wax, said composition also containing a resin in a proportion of up to 20%.

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