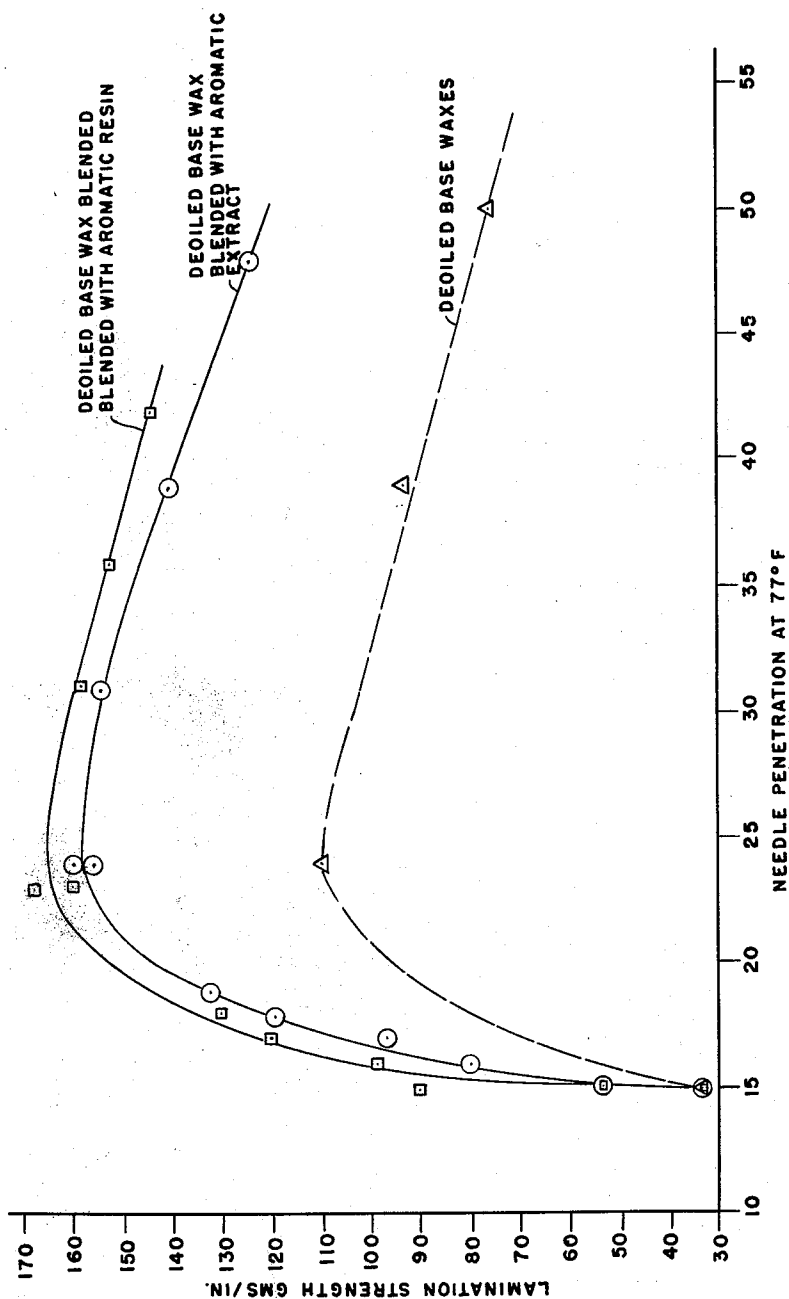


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COMPOSITION OF MATTER CONSISTING OF  
A MICROCRYSTALLINE WAX AND 5-25% OF  
AN AROMATIC PETROLEUM EXTRACT  
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2,954,337

**COMPOSITION OF MATTER CONSISTING OF A  
MICROCRYSTALLINE WAX AND 5-25% OF AN  
AROMATIC PETROLEUM EXTRACT**

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My invention relates to the production of novel microcrystalline wax compositions useful in the lamination of materials.

Microcrystalline waxes are used to a great extent in the paper industry for the lamination or bonding together of materials such as two or more sheets of glassine, cellophane, sulphite, kraft, and various metallic foils for the packaging of goods such as food and dairy products. The wax serves as an adhesive and, at the time time, acts as a barrier to the passage of moisture, oil and grease through the paper. Microcrystalline waxes are particularly suited for this purpose because of their properties of tackiness, film flexibility, grease resistance, water and water vapor resistance, and adhesiveness. Microcrystalline waxes are the solid hydrocarbon mixtures that are separated from heavy petroleum distillates and residues by conventional dewaxing processes. They may be deoiled to produce waxes of controlled melting points and different degrees of hardness.

I have found that deoiling a microcrystalline wax fraction to a hardness corresponding to a needle penetration at 77° F. of about 24 to 25 produces a wax of optimum lamination strength. I have further found that the addition of aromatic petroleum extracts to a microcrystalline wax fraction which has been deoiled to a hardness corresponding to a needle penetration at 77° F. of less than about 24 to 25 unexpectedly and greatly improves the lamination strength over that obtained by deoiling the wax. Although improvement in lamination strength at any penetration value is shown, surprisingly improved lamination strength is developed at the penetration hardness corresponding to the optimum strength of the deoiled waxes. As little as about 5 weight percent based on the composition of an aromatic petroleum extract produces a wax composition of increased lamination strength over that obtainable by deoiling the wax to its optimum strength. The flexibility of the wax composition is also improved. Thus, the wax compositions provide materials of greatly improved lamination strength for the lamination or bonding together of various materials used for the packaging of various articles. In use, the wax compositions are applied to a material to be laminated such as paper or foil to form a sheet or film and constitute a component part of the material. They serve as an adhesive and as a barrier to the passage of moisture, oil and grease when the material is used for packaging.

By the term aromatic petroleum extracts, I mean the aromatic petroleum products obtained by conventional solvent extraction of petroleum oils, such as residual lubricating oil stocks, which are characterized by their tackiness, high aromaticity, high viscosity and low viscosity index. The term includes aromatic extracts obtained from the solvent treating of residual lubricating oil stocks from Mid-Continent and paraffin base crudes, for example with phenol, furfural, nitrobenzene and the like, as well as the extracted aromatic residues known conventionally as petroleum resins which are the high molecular weight naturally contained viscous materials

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obtained by the solvent treating, for example, with propane, of heavy residual lubricating oil fractions from a Mid-Continent or naphthenic base crude. These materials are readily available and relatively inexpensive. By contrast, I have found that the addition to the deoiled microcrystalline wax fraction of paraffinic petroleum materials such as the corresponding raffinate from the solvent treating of lubricating oil stocks, i.e. a solvent refined Mid-Continent lubricating oil, or high viscosity index petroleum resins from Pennsylvania crude do not produce any improvement in lamination strength beyond that obtained by deoiling alone.

The compositions are prepared by first deoiling a microcrystalline wax fraction, e.g. petrolatum, to a hardness corresponding to a needle penetration at 77° F. of less than about 24 to 25. For commercial operation, deoiling to a penetration of about 15 to 20 is satisfactory and additional expense need not be incurred by further deoiling. The base wax must be deoiled, however, to a penetration lower than the optimum penetration of about 24 to 25 before the aromatic petroleum extract is added in order to achieve the improved results. Addition of the extract to a wax which has not been deoiled to below this penetration value does not improve the lamination strength but on the contrary has an adverse effect. An aromatic petroleum extract is then added to the deoiled wax and the components are intimately admixed. The amount of aromatic extract added may vary from about 1 to 25 percent based on the weight of the final composition. At least about 5 weight percent is required, however, to produce a composition of greater lamination strength than that obtainable by deoiling the base wax. Amounts of about 5 to 20 weight percent are preferred as these give the largest increase in lamination strength of the compositions. A proportion of about 10 to 12 percent produces an optimum laminating strength at about the same penetration hardness corresponding to the optimum lamination strength of the deoiled waxes. The improvement obtained with these aromatic materials appears to be independent of their viscosity.

The preparation and properties of the wax compositions of my invention will be further illustrated by the following example and by reference to Figure 1 which illustrates graphically the properties of the compositions.

Microcrystalline wax fractions of varying degrees of hardness were prepared by deoiling a Mid-Continent petrolatum and the products tested for lamination strength, following which aromatic petroleum extracts were added to a deoiled wax in varying proportions and the products tested for lamination strength.

In the example, the following test procedure was used for measuring the lamination strength of the compositions. Two sheets of glassine paper, 25 pounds per ream, were coated on one side with 4.5 to 5.5 pounds per ream of the wax composition to be tested. The sheets were trimmed and laminated by pressing them between two rolls. The bottom roll was heated to 210° F. with steam. The upper roll was a rubber roll weighted so that the bearing pressure between the rolls was 0.75 pound per inch. The laminated specimen was aged under fixed conditions and then delaminated at a uniform rate using a delamination tester. The amount of force in grams per inch to delaminate the test specimen was designated as the lamination strength.

The needle penetration was determined by ASTM D5-25 procedure modified in the following manner. The wax to be tested is completely melted in an oven and heated to 180° F. After stirring it is permitted to stand for fifteen minutes at 180° F. to allow any water to separate. A one inch section cut from a standard two inch copper tube is used as a mold and is placed on a sheet of aluminum foil on a stainless steel plate. The sample is

carefully poured into the mold, allowed to stand at 70° F.±2° F. for one hour, and aged in a water bath at 77° F.±1° F. for two hours. It is then placed in a transfer dish of such capacity as to insure complete immersion of the wax mold and to maintain a temperature of 77° F.±1° F. during the test. The needle penetration is determined in accordance with ASTM D5-25 method using the 100 gram load and five seconds penetration with the test points selected on the cast surface as nearly as possible at one half the distance from the center of the sample to the side of the mold.

The physical properties of the deoiled microcrystalline wax fractions, including the Mid-Continent petrolatum, and the lamination strength test results are tabulated in Table I.

aniline point of 48.3° C., an iodine number of 63.8 and a molecular weight of 516-519.

Another aromatic petroleum extract of the type known heavy residual lubricating oil stocks was also added in varying proportions to the 15 needle penetration deoiled as petroleum resin obtained by the solvent treating of wax and the resulting products tested for needle penetration and lamination strength. The aromatic resin had an API gravity of 9.9, a flash point of 540° F., a fire point of 625° F., a viscosity in SUS of 550,000 at 100° F., 1100 at 210° F., and 185.3 at 275° F., a viscosity index of -130, a pour point of 80° F., an O.D. color of 1939, a carbon residue of 7.738, an ASTM ash of 0.027, an aniline point of 46.0° C., an iodine number of 83.0 and a molecular weight of 558.

The results are tabulated below in Table II.

Table II

|                                          | Wt. Percent Additive <sup>1</sup> |    |      |     |     |          |     |     |     |
|------------------------------------------|-----------------------------------|----|------|-----|-----|----------|-----|-----|-----|
|                                          | 1                                 | 2  | 3    | 4   | 5   | 10       | 15  | 20  | 25  |
| Extract:                                 |                                   |    |      |     |     |          |     |     |     |
| Penetration at 77° F. <sup>2</sup> ----- | 15                                | 16 | 17   | 18  | 19  | 24       | 31  | 39  | 48  |
| Lamination Strength, gms./in.-----       | 53.3                              | 80 | 96   | 120 | 132 | 156, 160 | 154 | 140 | 124 |
| Resin:                                   |                                   |    |      |     |     |          |     |     |     |
| Penetration at 77° F. <sup>2</sup> ----- | 15                                | 15 | 16   | 17  | 18  | 23       | 31  | 36  | 42  |
| Lamination Strength, gms./in.-----       | 53.3                              | 90 | 98.6 | 120 | 130 | 160, 168 | 158 | 152 | 144 |

<sup>1</sup> Based on the final composition.

<sup>2</sup> Modified ASTM D5-25 procedure.

Table I

| Penetration, at 77° F.<br>(N).....            | Microcrystalline Base Waxes |       |       |       | Petrolatum |
|-----------------------------------------------|-----------------------------|-------|-------|-------|------------|
|                                               | 15                          | 24    | 32    | 39    | 66         |
| Gravity, ° API.....                           | 35.2                        | 34.5  | 34.5  | 34.1  | 33.6       |
| Viscosity, SUS/210° F.....                    | 74.0                        | 75.9  | 77.7  | 80.8  | 77.6       |
| Petrolatum, M.P., ° F.....                    | 160.0                       | 154.5 | 155.0 | 158.0 | 151.5      |
| Color, NPA.....                               | 7-(dilute)                  | 8     | 8     | 8     | 6          |
| Percent Oil (D721-51T).....                   | 0.13                        | 0.01  | 0.19  | 0.37  | 2.72       |
| Penetration, at 77° F.<br>(C).....            |                             |       |       |       | 33         |
| Penetration, at 110° F.<br>Abrahams Hard., at | 46                          |       |       |       |            |
| 110° F.....                                   | 17.0                        |       |       |       |            |
| Lamination Strength,<br>gms./in.....          | 34                          | 110   | 96    | 93    | 60         |

The results show that the optimum lamination strength of the waxes that can be obtained by a conventional solvent deoiling of petrolatum occurs at a needle penetration at 77° F. of 24. By reference to Figure 1, the curve resulting from the plotting of penetration against lamination strength of the deoiled waxes shows a gradual rise in lamination strength, as the wax is deoiled to a lower penetration value, to a peak optimum strength at a needle penetration at 77° F. of about 24 to 25, followed by a sharp decline.

If, however, aromatic extracts are added to the wax which has been deoiled to a hardness corresponding to a needle penetration at 77° F. of less than about 24 to 25 greatly improved lamination strength is obtained which is as much as about 50 percent more than the optimum lamination of the solvent deoiled waxes. For example, an aromatic petroleum extract obtained by the solvent treating of a lubricating oil stock was added in varying proportions to the 15 needle penetration deoiled wax of Table I and the resulting products tested for needle penetration and lamination strength. The aromatic extract oil had an API gravity of 11.5, a flash point of 510° F., a fire point of 595° F., a viscosity in SUS of 60,000 at 100° F., 9,604 at 130° F. and 405 at 210° F., a viscosity index of -72, a pour point of 55° F., an O.D. Color of 943, a carbon residue of 4.176, ASTM ash of 0.003, an

The results show that the optimum lamination strength of the wax compositions containing aromatic extracts occurs at a needle penetration at 77° F. of about 24 as does the optimum strength of the deoiled microcrystalline waxes of Table I. The lamination strength of the wax compositions, however, is unexpectedly and greatly increased at this needle penetration to as high as 168 grams per inch as compared to 110 for the deoiled waxes. The surprising improvement is shown graphically in Figure 1 in which the curves resulting from the plotting of penetration against lamination strength of the wax compositions show a marked increase in lamination strength over the deoiled waxes at any penetration value and particularly at the penetration value corresponding to the optimum lamination strength of the deoiled waxes.

Thus, microcrystalline wax compositions of greatly improved lamination strength can be produced by the addition of readily available and relatively inexpensive materials to a deoiled microcrystalline wax thereby providing materials of improved utility in the lamination of materials such as paper and foils for the packaging of goods.

I claim:

1. A microcrystalline wax composition of improved lamination strength which essentially comprises a microcrystalline wax fraction base which has been deoiled to a hardness in terms of needle penetration at 77° F. of less than about 24 to 25 and about 5 to 25 weight percent based on the composition of an aromatic petroleum extract.

2. A microcrystalline wax composition of improved lamination strength which essentially comprises a microcrystalline wax fraction base which has been deoiled to a hardness in terms of needle penetration at 77° F. of about 15 to 20 and about 5 to 20 weight percent based on the composition of an aromatic petroleum extract.

3. The method of preparing a microcrystalline wax composition of improved lamination strength which comprises deoiling a microcrystalline wax fraction to a hardness corresponding to a needle penetration at 77° F. of less than about 24 to 25 and admixing therewith about 5 to 25 weight percent based on the composition of an aromatic petroleum extract.

4. The method of preparing a microcrystalline wax composition of improved lamination strength which comprises deoiling a microcrystalline wax fraction to a hardness corresponding to a needle penetration at 77° F. of about 15 to 20 and admixing therewith about 5 to 20 weight percent based on the composition of an aromatic petroleum extract.

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UNITED STATES PATENT OFFICE  
CERTIFICATE OF CORRECTION

Patent No. 2,954,337

September 27, 1960

Edward J. Martin

It is hereby certified that error appears in the printed specification of the above numbered patent requiring correction and that the said Letters Patent should read as corrected below.

Column 4, line 6, strike out "as petroleum resin obtained by the solvent treating of" and insert the same after "known" in line 3, same column 4; column 6, line 4, list of references cited, under "UNITED STATES PATENTS" for "Bennett et al." read -- Hill et al. --.

Signed and sealed this 11th day of April 1961.

(SEAL)

Attest:

ERNEST W. SWIDER

Attesting Officer

ARTHUR W. CROCKER  
Acting Commissioner of Patents