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STRESSING OF PETROLEUM WAX TO IMPROVE PHYSICAL PROPERTIES THEREOF

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This invention relates to a process for improving the physical properties of petroleum wax by subjecting the wax to a critical pressure, and also to an improved petroleum wax having an increased resistance to penetration. The invention also relates to an improved wax coated paper and a process for producing such paper by subjecting it to a critical pressure prior to use.

In the past, paraffin waxes of the type derived from petroleum have found application in such articles as coated paper, fibre board, or the like. In some cases these waxes have also been employed in the manufacture of candles. Although wax is generally considered to be a crystalline substance, it is well known that it has a tendency to coalesce with surfaces of the same material, even at room temperature. This property is usually referred to as the blocking tendency of the wax, and it is desired to minimize such tendency in order to produce wax coated paper which may be stored easily and which does not have a tendency to adhere to adjacent wax or other surfaces. This property is generally correlated to the hardness of the wax so that a wax of increased hardness would have a substantially reduced blocking tendency.

In the use of petroleum waxes in candle manufacturing it has been found that candles have a tendency to sag under their own weight and become deformed, even when stored at room temperature. This property is similar to the blocking tendency of the wax when used as a coating and is also directly related to the hardness of the wax.

It is an object of the present invention to provide a process for improving the hardness of petroleum wax and simultaneously reducing its blocking tendency when employed as a coating.

It is a further object of this invention to provide an improved hardened petroleum wax especially suitable as a coating for paper or the like as well as for use in candles and in other applications where a hard wax is useful.

It is a still further object of the invention to provide an improved wax-coated paper or fibre board having substantially reduced blocking tendency.

Other objects will be specifically pointed out or obvious from the ensuing description.

The objects of the invention are achieved by determining the pressure to which a particular petroleum wax may be subjected before it undergoes mechanical failure and then applying a uniaxial load to the wax and increasing it at a constant rate up to a predetermined pressure at which maximum hardening of the wax occurs. In this way the wax is permanently hardened and its blocking tendency substantially reduced. Pressure may be applied either to the wax when it is in the form of integral pieces or slabs or when it is in the form of a thin coating on paper or the like. In these latter cases the pressure may be applied by passing the coated paper between two rollers which exert the desired pressure.

In studying the behavior of petroleum wax under mechanical stress, a logical explanation has been formulated for the unexpected improvements in physical properties obtained by a stressing treatment. In applying a uniaxial load to a block of wax, three separate and distinct types of deformation have been observed. Initially, the wax is deformed elastically as a linear function of the total load applied. As the load is increased, plastic, rather than elastic, deformation sets in, followed by plastic flow of the wax with the accompanying softening thereof. This third phase, plastic flow, occurs to the point of wax failure. During the second or plastic deformation phase a hardening of the wax takes place. If wax is subjected to stress sufficient to cause plastic deformation, it will be permanently hardened and its blocking tendency substantially decreased. If, however, the wax is subjected to a load great enough to cause plastic flow, softening will set in and the wax will not be hardened.

It is believed that during the hardening period the individual particles of wax become coalesced into larger particles than those existing in the unstressed wax. The existence of these large individual wax particles probably enables the wax to change from a tacky to a rigid body.

The pressure required for maximum hardening may be calculated for any wax after carrying out a few tests to determine the relation of stress to strain and strain rate for the particular wax. The compressive stress A may be described by the following equation:

$$A=B(e(t+1))^b$$

where e is the strain rate, t is the time, and B and b are constants. At constant stress the product $e(t+1)$ is a constant and is equal to the parameter C which has the dimensions of strain. It has been found that the mechanical properties of petroleum waxes under compression are defined by their coefficients of plastic traction, which has the dimensions of viscosity and may be expressed by the following equation:

$$U=bA(t+1)/C=bA/e$$

where U is the coefficient of plastic traction. It has been found that this coefficient reaches a maximum at certain stresses, and it is at these stresses that maximum hardening is attained. This is the value referred to as the critical hardening pressure or the pressure at which maximum hardening occurs.

It has been found that the critical hardening pressure to which waxes must be subjected varies depending upon the melting point and composition of the wax. For example, a microcrystalline wax will require substantially less maximum hardening stress than a recrystallized or sweated paraffin wax. This difference is attributed to the greater oil content and more heterogeneous composition of the microcrystalline wax.

The pressure employed in hardening the wax will broadly range between 5 and 100 p. s. i. g. with the higher pressures being necessary for recrystallized waxes of higher purity. The pressure may be applied uniaxially for a period of from about 1 to 5 hours in any suitable manner, depending upon the form of the wax being treated. For example, when treating a wax coated paper it is convenient to apply the pressure by passing the wax between rollers.

Examples of the operation of the present invention are set forth below.

EXAMPLE I

A sweated paraffin wax having a melting point of 136° F. was subjected to a compressive stress of 80 p. s. i. g. for one hour at 77° F. This sample and an unstressed sample of the same wax were then tested for hardness by determining the penetration into a slab of the wax of a metal cylinder of 0.125 inch diameter under

a load of 558 grams in sixty seconds. The following results were obtained:

	Unstressed Wax	Stressed Wax
Penetration in decimillimeters @ 77° F.	2.75	1.0
Penetration in decimillimeters @ 100° F.	68.5	30.0

It will be seen from the above results that the stressed wax produced by the process of the present invention was more than twice as hard when viewed with respect to its resistance to penetration as the ordinary unstressed wax.

EXAMPLE II

A block of sweated wax having a melting point of 129° F. and of the dimensions of 5 x 2.5 x 9/16 inches was stressed under 12 p. s. i. g. for two hours at a temperature of about 65° F. This stressed block and an unstressed block of the same size made from the same wax were then freely suspended at their short edges and allowed to sag under their own weight for 72 hours at room temperature. At the end of this time it was found that the unstressed wax deflected 0.4 inch during this period, while the stressed wax experienced immeasurably small elastic deflection which recovered almost immediately upon reversing its position. On the other hand, the unstressed wax remained permanently deformed. These results clearly illustrate the advantages to be gained from using a prestressed wax in the manufacture of candles, which are subject to such deflection during storage.

Although it is preferably to subject the wax being treated to a stress just below the point at which plastic flow will begin, improvements may also be obtained by the application of pressures below this maximum critical value. For example, the critical pressure of the sweated wax described in Example II above was substantially more than 12 p. s. i. g., namely, about 50 p. s. i. g. Nevertheless, a substantial improvement in hardness was imparted by such treatment. In some cases it may be impractical or undesirable to subject the wax to a pressure close to the maximum, especially where the maximum is close to 100 p. s. i. g., which is about the highest critical pressure required for any petroleum wax. Table I below gives the melting points and maximum stresses of three typical petroleum waxes.

Table I

	Melting Point, ° F.	Stress for Maximum Hardness, p. s. i. g.
Microcrystalline wax	148	9.1
Sweated paraffin wax	138	63.7
Recrystallized paraffin wax	139	85.7

In general, recrystallized waxes of more homogeneous character and lower oil content require greater stresses to produce maximum hardening. The particular pressure required for a particular wax can be determined simply by loading the sample until failure occurs and then applying a pressure somewhat below this. In general, it has been found that the maximum hardening pressure will be about 30–50% of the pressure at which failure occurs.

While this invention has been described with respect to certain specific embodiments thereof, it will be understood that it embraces various equivalents regarding the methods of applying pressure and the forms in which the wax may be stressed. The invention is not to be limited, therefore, except by the scope of the appended claims.

What is claimed is:

1. A process for increasing the hardness of a petroleum wax which comprises plastically deforming said wax by the application thereto of uniaxial pressure, said pressure being discontinued before plastic flow of said wax occurs.

2. A process for increasing the hardness of a petroleum wax which comprises plastically deforming said wax by the application of uniaxial pressure thereto, said pressure being maintained below the pressure at which plastic flow of said wax occurs.

3. A process for increasing the hardness of a petroleum wax which comprises plastically deforming said wax by the application of uniaxial pressure thereto, increasing the pressure applied to said wax to a value 30% to 50% of the pressure at which mechanical failure of said wax occurs, and continuing the application of said pressure for a period of from 1 to 5 hours.

4. A process for increasing the hardness of petroleum wax on a waxed paper which comprises plastically deforming said wax by passing said paper between pressure rolls, the pressure exerted by said rolls being of insufficient duration to cause plastic flow of said wax.

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