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METHOD OF COATING FIBROUS MATERIALS WITH WAX

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ABSTRACT OF THE DISCLOSURE

Fibrous sheet material is impregnated with paraffin wax and surface coated with microcrystalline wax by first surface coating the sheet material with a blend of paraffin and microcrystalline waxes having a difference in melting points of at least 15° F., and then heating the blend to a temperature intermediate the melting points to allow the paraffin to melt and soak into the sheet material while the microcrystalline wax remains on the surface.

This invention relates to the coating of cellulosic fibrous sheet material such as paper, cardboard, paper bond, regenerated cellulose, etc., with wax. The invention provides a means whereby in a single coating operation followed by an annealing or heat treating operation the paper or other sheet material is impregnated with paraffin wax and surface coated with a wax composition rich in microcrystalline wax.

Paper has been treated with wax for many years in order to make it more suitable for certain applications. Three types of treatments are known. One treatment involves impregnating the paper with wax. In this treatment the wax soaks into and saturates or at least partially saturates the fibers of the paper. This type of treatment results in, e.g., increased stiffness of the paper. A second treatment involves applying only a surface coating of wax to the paper without allowing any impregnation of the paper with the wax. This type of treatment also results in, e.g., increased stiffness, but also achieves certain benefits, e.g., improved gloss, not available with the first type of treatment. The third type of treatment is a combination of these two. The paper is treated with a relatively large amount of wax with the result that the paper is both impregnated and surface coated with wax. This third type of treatment achieves the benefits of both of the other treatments mentioned.

The type of wax used in treating paper depends upon a number of competing factors. Ideally, all wax treating of paper would be with paraffin wax since the latter is generally more economical than other waxes such as microcrystalline wax, hereinafter micro wax. Unfortunately paraffin waxes are often unsuitable for surface coating of paper for any of a number of reasons. The low melt point paraffin waxes (M.P. 110°–135° F.) have poor blocking properties and poor scuff resistance. For example, a paraffin wax melting at 125° F. might have a blocking temperature (ASTM D–1465) of about 93° F. whereas the minimum blocking temperature acceptable to the trade is about 115°–120° F. These blocking and scuff problems can be overcome to a large extent by the used of high-belt paraffins (M.P. 135°–165° F.). For example, paraffin waxes melting at 138° F. and 153° F. might have blocking temperatures of 116° F. and 130° F. respectively. However, the high-melt paraffins have disadvantages of their own, one being that they tend to be unduly brittle.

The disadvantages of the paraffin waxes are normally overcome by incorporating micro wax in them. Although

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the micro waxes are considerably more expensive than the paraffins they are harder, have higher melting points (usually 165°–210° F.), and as a result they have good blocking properties and good scuff resistance. Moreover, they are considerably more flexible than the paraffin waxes and hence can be used to reduce the brittleness of the latter. When micro wax is used to improve the blocking and scuff resistance characteristics of paraffin wax the amount used is normally such that the resulting blend contains 5–50%, preferably 15–40%, micro wax. Within these ranges the actual amount employed will depend mainly upon the properties of paraffin wax used and the intended application of the wax coated paper or other sheet material. As a general rule, the amount of micro wax used is inversely proportional to the paraffin wax melting point. In some cases, albeit relatively few, it is essential that maximum blocking properties be obtained regardless of cost. In such a situation the wax used may contain 50–100° micro wax. When micro wax is used to reduce the brittleness of paraffin wax the amount used is normally such that the resulting blend contains 1–10%, usually 2–7%, micro wax the actual amount again depending upon the factors mentioned above.

When paper or other fibrous sheet material is to be both impregnated and surface coated with wax and when a blend of paraffin and micro waxes is necessary so that the coated paper will have satisfactory blocking, brittleness, and scuff resistance properties, the usual procedure is to effect the wax treatment in a single step with the result that the wax blend is used for both impregnation and surface coating. Thus the wax which impregnates the paper has the same composition as the wax which coats the paper. This is obviously undesirable since the wax which impregnates the paper is not exposed and therefore its blocking, scuff resistance, and certain other properties are immaterial. It would be more desirable to impregnate the paper with a relatively cheap wax such as paraffin and only surface coat the paper with the more expensive paraffin-micro blend. Moreover, it would be desirable to do this by a technique involving only a single application of wax to the paper. We have now found such a technique.

According to the invention, paper or other fibrous sheet material is coated by a procedure involving the following steps. In these steps and the discussion relating thereto it will be assumed that the fibrous sheet material is paper.

(1) The paper is surface coated with a blend of paraffin and micro wax. This coating is applied in a manner so as to avoid any substantial impregnation of the paper.

(2) The surface coated paper is then brought to a temperature which is sufficient to melt substantially only the paraffin wax component of the paraffin-micro blend. In other words the temperature is such that the paraffin wax component is molten and the micro wax component is solid.

(3) The paper is held at this temperature until some or all of the paraffin wax soaks into, i.e., impregnates, the paper.

(4) The paper is then cooled to a temperature below the melting point of each wax. This results in paper which is impregnated with solid paraffin wax and surface coated with a solid wax composition relatively rich in micro wax. The surface coating is relatively rich in micro wax because it has a higher micro content than the surface coating applied in (1).

Each of these four steps is described in more detail as follows:

The first step involves surface coating the paper with a blend of paraffin and micro wax. The actual amount of each type of wax is discussed more fully subsequently but will depend upon the amount of paraffin wax soaked into the paper in step (3), the percentage of micro wax desired in the surface coating on the finished paper, and the amount of surface coating on the finished paper.

The coating applied in this first step should be only a surface coating. In other words there should be no substantial impregnation of the paper. The reason for this is two-fold. One, if the paper is completely saturated with the initial wax blend it would obviously be impossible to soak additional wax into the paper in step (3) of the invention. Secondly, the purpose of the invention is to provide a means of impregnating the paper with paraffin wax and surface coating the paper with a wax containing a relatively large amount of micro wax. To the extent that the paper is impregnated with micro wax this purpose is defeated. In general, in this first step the paper should be less than 50% impregnated, i.e., the amount of impregnated wax should be less than 50% of the amount required to saturate the paper. Preferably the paper is less than 20% impregnated and the purpose of the invention is even more effectively achieved when the paper is impregnated less than 5–10%.

This first coating can be applied by any convenient means such as curtain coating or by reverse roll coating. These are only two of the means well known in the art for applying a surface coating of wax to paper without any or without substantial impregnation of the paper. In all of these known means impregnation of the paper is prevented by rapid cooling of the coating, i.e., by solidifying the coating before it has time to soak into the paper. Since these means are well known in the art and since they are not per se part of this invention they are not further described. A coating procedure generally unsuitable for the present purpose is dip coating. As is well known this coating procedure usually results in complete impregnation of the paper.

The second step of the invention involves bringing the surface coating formed in step (1) to a temperature sufficient to melt substantially only the paraffin wax component thereof. This temperature will not be the same for every paraffin-micro blend but will vary depending almost entirely upon the melting points of the component waxes. For example, with a blend containing 80% of a paraffin wax melting at 126° F. and 20% of a micro wax melting at 192° F. a temperature of 150° F. is effective to melt essentially all of the paraffin and essentially none of the micro. A temperature of 140° F. is also effective with the same blend but this results in a higher viscosity of the molten paraffin wax and the time required for the paraffin wax to soak into the paper in step (3) is thereby increased. With a blend containing equal parts (all percentages and parts herein are by weight) of a paraffin wax melting at 155° F. and a micro wax melting at 200° F. a temperature of 170° F. is sufficient to melt essentially all of the paraffin and essentially none of the micro.

Although no single temperature can be specified which will be suitable for all paraffin-micro wax blends, a suitable temperature for a given blend will always be between the melting points of the paraffin wax and micro wax in the blend. Furthermore, a suitable temperature can be readily determined by those skilled in the art. One method of determining a suitable temperature involves heating the blend until it is completely molten. Next the blend is slowly cooled. This results eventually in precipitation of the micro wax followed by precipitation of the paraffin wax. As wax precipitates it is periodically (e.g., every 4° F. during the cooling) separated and analyzed to determine the micro content thereof. Such analysis can be by, e.g., refractive index. The temperature at which the paraffin wax begins to precipitate is thus readily determined, for it is known that the refractive index (R.I.) of a wax blend varies linearly with the refractive index and amount of each wax in the blend.

For any given wax blend there will normally be a range of temperatures suitable for use in step (2). Thus in the example mentioned above temperatures of 140°–

150° F. are suitable. However, for the viscosity reason also mentioned above it is preferable to use the maximum temperature at which there is no substantial melting of the micro wax.

The second step of the invention is described as melting substantially only the paraffin wax. The term substantially is used because there will be some micro wax dissolved in the molten paraffin wax, i.e., there will be some micro wax not in solid form. This amount will vary with the actual temperature but will generally be less than 1% based on the weight of paraffin wax and will always be less than 5–10%. In fact, another means of describing the second step of the invention is that the surface coating applied in step (1) is brought to a temperature at which the paraffin wax is molten and at which the solubility of the micro wax therein is less than 5–10%, preferably less than 1%.

In order to melt the paraffin wax without melting any substantial amount of micro wax it is necessary that the paraffin and micro waxes have a difference in melting point of at least 15° F. For example, if the paraffin wax melts at 155° F. and the micro wax at 158° F. practice of the invention would be impossible for several reasons. One, a temperature sufficient to melt the paraffin wax would be so close to the micro wax melting point that the solubility of the latter in the former would be excessive. Secondly, the melting points are so close that extremely precise temperature control, often not obtainable in commercial equipment, would be required. Preferably the difference in the melting points of the waxes is at least 25° F.

The surface coating applied in step (1) can be brought to the temperature specified in step (2) in either of two ways. In one technique the coating applied in step (1) is cooled to completely solidify the coating, e.g., to room temperature, and the coating is subsequently reheated to the temperature specified in step (2). In cooling to room temperature the coating will, obviously, pass through a temperature sufficient for step (2) of the invention. However, in conventional methods of cooling a wax coating applied to paper or other material the cooling is so fast, and intentionally so, that the time for which the coating is at such a temperature is insignificant. This complete solidification followed by reheating is the preferred technique. The second technique is to cool the coating applied in step (1) directly to the temperature specified in step (2).

After preferentialy melting out the paraffin wax in step (2) the coating is held at the temperature specified in step (2) until some or all of the the molten paraffin wax soaks into the paper. The time required for this impregnation will depend mainly upon the absorptivity of the paper, the viscosity of the molten paraffin wax, and the amount of wax desired to be impregnated but will generally be 5–60 minutes, usually 10–60 minutes. The amount of wax which soaks into the paper is discussed more fully hereinafter and can be determined by known techniques.

After the paraffin wax has soaked into the paper the latter is cooled to below the melting point of each wax. This results in paper impregnated with solid paraffin wax and surface coated with a solid wax composition relatively rich in micro wax. In most cases the surface coating will contain 1–50% micro wax, more frequently 2–40% micro wax, but will in some cases contain higher amounts, i.e., 50–100%, micro wax. In most cases the amount of micro wax in the final surface coating will be at least 50% higher, more frequently at least 100% higher, than the amount of micro wax in the coating applied in step (1).

The amount and composition of the blend applied to the paper in step (1) will depend upon the amount thereof which is soaked into the paper in step (3) and upon the desired amount and composition of the surface coating in the finished paper obtained in step (4). From these variables the amount and composition of the blend applied in step (1) will normally be back calculated. For example, if it is desired to saturate a glassine paper which can be completely saturated with 2 lbs. wax per ream (1 ream=3000 sq. ft.) and then coat the paper with 3 lbs. per ream of a blend of 70% paraffin and 30% micro the initial coating in step (1) would be 5 lbs. per ream of a blend containing 82% paraffin and 18% micro, calculated as follows where all lbs. are lbs. per ream.

Surface coat: 3 lbs. x 70% paraffin	Lbs. paraffin	2.1
Impregnated wax	Lbs. paraffin	2.0
Total paraffin	Lbs. paraffin	4.1
Surface coat: 3 lbs. x 30% micro	Lbs. micro	0.9
Total wax	Lbs. wax	5.0

Micro=18%
Paraffin=82%

In a similar manner it can be calculated that if cardboard is to be coated with 20 lbs. per ream of a blend containing equal amounts of paraffin and micro and the cardboard can be saturated with 200 lbs. per ream but it is only desired to soak in 80 lbs. per ream the coating in step (1) should contain 90% paraffin and 10% micro and should be applied in the amount of 100 lbs. per ream.

The actual amount of impregnated wax and the amount and composition of the final surface coating will depend mainly upon the type of fibrous sheet material being treated and the intended application thereof. The amount of wax which will soak into the sheet material will vary rather widely depending upon the absorptivity of the paper. Normally it wil be 0.5–250 lbs. per ream. For example, with glassine paper only about 0.5–5 lbs. wax per ream will soak into the paper but with a highly absorptive material such as cardboard 100–250 lbs. per ream can be soaked in without difficulty. In most cases the amount of impregnated wax will be 1–50 lbs. per ream and will, in addition, be at least 50%, more frequently at least 80%, of the amount required to saturate the fibrous sheet material.

The amount of surface coating on the finished sheet material will also vary. The minimum amount is about 1–2 lbs. per ream and would be used in a material such as glassine paper. Normally at least 3 lbs. per ream is used. The maximum amount will be about 35–40 lbs. per ream and would be used in the case of a material such as cardboard. Usually not more than 30 lbs. per ream will be used. The amount of micro wax in the final surface coating has been discussed previously.

The following example specifically illustrates the invention.

A blend containing 82% of a paraffin wax having a melting point of 129° F. and an R.I. of 1.4223 and 18% of a micro wax having a melting point of 192° F. and an R.I. of 1.4363 is prepared by heating a mixture of the two waxes to 200° F. with stirring. The R.I. of the blend is 1.4248.

The blend is charged at 200° F. to a reverse roll coater and is then applied to 14 point milk carton stock at a weight of 40 lbs. per ream with essentially no impregnation of the stock. The surface coating is removed (by scraping) from a sample of the stock and the R.I. thereof is determined to be 1.4248. Another sheet of the stock is saturated by dip coating and it is found that 21.7 lbs. wax per ream are required to completely saturate the stock.

One portion of the stock surface coated in the manner described is set aside for later use and another portion is placed in an oven at 150° F. for one hour. It is then removed, cooled to room temperature, and the surface coating is then removed from a small portion thereof. The R.I. of the coating is 1.4278 from which it can be calculated that the composition of the surface coating is 60.7% paraffin wax and 39.3% micro wax. Thus the amount of micro wax in the final surface coating is slightly over 100% higher than in the initial surface coating. It can also be calculated that the amount of paraffin wax which soaked into the stock is 21.7 lbs. per ream.

The blocking temperature and scuff resistance properties of the surface coated stock not subjected to the heat treatment at 150° F. (referred to as the untreated stock) and the coated stock heat treated at 150° F. (referred to as the treated stock) are then determined. The blocking temperature is determined by ASTM D–1465. Scuff resistance is determined by dragging a weighted razor blade over each stock 3 times and weighing the scrapings. The razor blade is weighed with 500 g. and is dragged at an angle of 52° with the horizontal with the blade inclined in the direction of travel.

The treated stock has a blocking temperature of 124° F. whereas the untreated stock has a blocking temperature of 115° F. In addition, the scrapings from the treated stock weigh 39% less than the scrapings from the untreated stock. These results show the advantages obtained from the heat treating steps.

The invention claimed is:

1. Method of impregnating and coating fibrous sheet material with wax which comprises (1) applying a surface coating of a mixture of paraffin and microcrystalline waxes having a difference in melting points of at least 15° F. to said sheet material without any substantial impregnation of the latter by either of said waxes, (2) bringing the surface coating to a temperature intermediate to the melting points of said waxes sufficient to melt substantially only the paraffin wax component of said coating, (3) holding the surface coating at said temperature until the resulting molten paraffin wax impregnates said fibrous sheet material, and (4) cooling the waxes to below the melting point of both of said waxes, whereby there is obtained fibrous sheet material impregnated with solid paraffin wax and surface coated with a solid wax composition which is relatively rich in microcrystalline wax.

2. Method according to claim **1** wherein the amount of surface coating on the fibrous sheet material obtained in (4) is 1–40 lbs. per ream of fibrous sheet material.

3. Method according to claim **2** wherein the surface coating on the fibrous sheet material obtained in (4) contains 1–50% microcrystalline wax.

4. Method according to claim **1** wherein the amount of microcrystalline wax in the surface coating on the fibrous sheet material obtained in (4) is at least 50% higher than the amount of microcrystalline wax in the surface coating applied in (1).

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