This invention relates to the recovery of the hard wax found in the filter residues which are obtained in extracting sugar cane and aims to improve a method for recovering from such residues high yields of a hard wax which is an acceptable substitute for eucalyptus wax.

In my Patent No. 2,391,683 of January 1, 1946, I have disclosed a rapid and economical one-step method for recovering such hard wax from filter press residues of sugar cane, by extracting the residues with suitable solvents at elevated temperatures, in a ratio of not less than four parts of solvents to one part of residue (the ratio based on the dry weight of the residue) and separating from the extract, by means of chilling, that portion of extracted matter which consists of the hard wax, while the simultaneously extracted fatty constituents of the filter residue remain in solution.

The object of the present invention is an improvement in the process of extracting the said hard wax and this improvement is based on the discovery that the amount of solvent, necessary for the extraction of a given quantity of filter residue, can be materially reduced if the latter, prior to the extraction, is compressed and dried so as to form coherent masses which substantially retain their coherency throughout the subsequent procedures. I found that, after compressing and drying the filter residue in this manner, whereby its apparent density (compared in the dry state) is approximately doubled, only about one-fourth of the previously required amount of solvent is needed, without impairing the efficiency of the extraction and that the amount of solvent remaining absorbed in the extracted residue is similarly reduced. In my preferred procedure, the residue is compressed into rods of a diameter of about one-quarter of one inch. Upon leaving the extrusion press, these rods may break into segments of various lengths.

I wish to emphasize, however, that it is the step of compressing along which permits the subsequent saving on solvent, irrespective of the form and, within certain limits, irrespective of the size which the particles may attain thereby. The results will be the same if the material is compressed into particles having hexagonal cross sections or into bars having a width of, say, up to one-half of one inch.

It is the more surprising that the increased density does not impair the extraction since it is ordinarily thought that a more finely divided material is extracted more easily; an assumption which I found not to be true in this case.

Several important procedural changes become possible in consequence of this discovery. The filter residue or mud (as it is frequently called), leaves the filter presses at the cane sugar mills with a moisture content of about 75 per cent and is rather bulky for this reason. Inasmuch as it contains, besides other growth supporting substances, small amounts of sugar, it constitutes an effectual medium for bacterial propagation and, for this reason, cannot be stored for any length of time without deteriorating. But if the mud is compressed into coherent particulate form and dried thereafter, its weight and bulk are not only reduced to less than half but it may now safely be stored for any length of time.

Whenever wax is to be extracted from moist filter residue, in view of the rapid deterioration and putrefaction of the undried material, the extraction must be carried out concurrently with the cane sugar campaign of the sugar mills from which the mud is obtained. It is significant that the drying of the mud renders the extraction process independent of the time of harvesting and crushing of the sugar cane and that it enables a continuous operation of the extraction facilities, provided a sufficient quantity of the dried residue has been accumulated and stored.

Aside from the social factor of procuring steady employment if the extraction of wax from the dry mud is extended over the larger part of the year with, perhaps, the only interruption of switching the operators to the drying of the mud during the relatively short cane sugar campaign, the economic gains in the wake of the aforementioned discovery are most noteworthy. Since the capacity required of the extraction equipment, for the production of a given quantity of wax, will change in an inverse ratio with the length of time during which the equipment can be utilized, the possibility of spreading the production over the entire year reduces the required unit size to less than one-fourth if the originally planned production volume is to be maintained, or, conversely, increases more than four fold the capacity of a unit which had been designed for seasonal operation. It is important that this reduction of the relative costs of installation permits a considerably more profitable use of capital expenditures while a continuous operation tends to reduce the costs of depreciation. In view of the fact that the margin between gain and loss in the extraction of sugar cane wax is necessarily a narrow one, largely depending upon the degree of efficiency of operation and inasmuch as solvent losses represent a major item in the pro-
duction costs, the economical import of my discovery is self-evident. Referring to the drawing, in practicing my invention a plant for the extraction of sugar cane wax (which should if possible be centrally situated relative to a number of sugar mills supplying the filter press residue), consists of two main parts: a seasonally run drying installation and a continuously operated extraction assembly. At the sugar mills the cane is shredded and crushed between rollers to extract the juice which, at this stage, consists of a dark, opaque liquid, carrying, besides other solids, part of the wax from the canes in suspension. In order to remove these suspended solids and to precipitate colloidal matter which is present, the juice is limed, heated, and, as a rule, clarified by sedimentation in tanks from where the clear supernatant liquid is drawn off. The final sediments are concentrated in filter presses. These residues leave the presses with a moisture content of about 60 to 70 per cent and are reduced, preferably at the mills, to a moisture content of about 40 per cent (which may easily be accomplished by means of a dryer [12]); not only in order to reduce the mud in weight and volume prior to its transfer to the extraction plant, but mainly because it was found that a moisture content of roughly 40 per cent is required for compressing the mud into coherent particles which are essential for the subsequently claimed saving on solvent. A moisture content within the range of, say, 35 to 45 per cent will generally be found optimal for this purpose, although a proper compressing will depend upon a number of factors, such as the relative amount of sugar, gums, and other constituents of the mud which act as binders, upon the type of equipment, the temperature, pressure, etc. The operation may be performed by means of a suitable extrusion press [13]. The resulting mud particles are then dried to a moisture content of about two per cent, which may be done either by means of a vacuum drier [14], or with a blower type drier at atmospheric pressure, whereby, however, temperatures above 185°F should be avoided in order not to impair the wax and its subsequent extracting. The material is placed, after drying, in storing facilities [15] for storage of the mud and from where adequate amounts are removed as needed for the extraction.

Having charged an extractor [17] with a given quantity of dry mud particles, about one and one-half times its weight of a suitable solvent is drawn from a solvent storage tank [16] and the mud is digested, at a temperature preferably approximating or approaching the boiling point of the solvent, for a sufficient period of time to insure complete solution of the waxy and fatty constituents of the mud. The solution is then separated, by means of a filter [18] or any other suitable means, and the mud transferred to an evaporator [19], where it is freed, as completely as possible, from the absorbed solvent, whereby the vapors, after having been reduced to the liquid stage in a condenser [20], are recycled to the solvent storage tank [16]. The remaining exhausted mud, collected in storage bin [21], may be used for any suitable purpose, such as fuel, fertilizer, or as a filler.

The solvent, containing the waxy and fatty matter, has been transferred, in the meantime, to a cooling tank [22]. There it is chilled to about 15°C, whereby the so-called hard wax precipitates in granular form while the extracted fatty material remains in solution. The precipitated wax is separated from the solution by means of a filter [23] and the solution is transferred to an evaporator [24] in order to recover therefrom the fatty constituents, which are collected in storage tank [25]. Subsequently, the so-called soft wax, containing about 30 to 45 per cent of the totally extracted matter and since it contains in the main of glycerides of fatty acids, such as palmitic, stearic and oleic acid, of various sterols of the phytosterol class, i.e., free and esterified cholesterol, the soft fraction provides a valuable by-product. The solvent which is evaporated in this operation, after passing a condenser [26], is sent to the solvent storage tank [16]. Having freed, in the meantime, in drier [27] the hard wax from the remaining solvent (whereby the vapors of the latter are precipitated in condenser [28] and are also recycled to storage tank [10]), it is recovered, depending upon the manner of drying, either in form of the little granules at which it has been precipitated in cooling tank [22], or in form of a melted product if dryer [21] is operated at a sufficiently high temperature. A typical product, taken from storage [29], analyzes as follows: methyl iso-butyl ketone, methyl amyl ketone, diisopropyl ketone, methyl, ethyl, propyl, butyl and amyl acetates and propionates. Of the solvents, I prefer to use those having boiling points not in excess of 120°C, since I find that the solvents having a higher boiling point are somewhat more difficult to recover from the wax, and that discoloration of the wax may occur with them unless great care is taken in the recovery operation.

For such subject matter as is common to my co-pending applications, I claim the benefit of that date. I claim:

1. In the extraction of wax from sugar cane mud by an organic wax solvent, the improved step of preparing the mud for extraction which comprises compressing mud, dewatered to a moisture content of 36 to 42 per cent by weight, into rod-like, coherent masses, and drying said masses at a temperature below 185°F, whereby the wax may be extracted with a fraction of the amount of solvent necessary for the extraction of dried mud not compressed in the said manner.

2. In the extraction of wax from sugar cane mud by an organic wax solvent, the improved step of preparing the mud for extraction which comprises dewatering the wet mud to a moisture content between 36 to 42 per cent by weight, compressing the mud into coherent masses long in one dimension and of a cross section in the short dimension not above one-half of one inch, and drying said masses at a temperature below 150°F, to a moisture content of about two per cent, whereby the wax may be extracted with less than 40 per cent of the amount of solvent necessary for the extraction of dried mud not compressed in the said manner.

3. In the extraction of wax from sugar cane mud by an organic wax solvent, the improved step of preparing the mud for extraction which
comprises compressing mud, dewatered to a moisture content of 36 to 42 per cent by weight into coherent masses long in one dimension and of a cross section in the short dimension not above one-half of one inch, and drying said masses at a temperature below 150° F. to a moisture content of about two per cent, whereby the wax may be extracted with less than 40 per cent of the amount of solvent necessary for the extraction of dried mud not compressed in the said manner.

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