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METHODS OF REGENERATING VULCANIZED RUBBER

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This invention relates to the art of reclaiming rubber, and more particularly refers to improvements in methods of regenerating vulcanized rubber scraps.

It is well known that vulcanized rubber scraps may be plasticized and made available for reprocessing by the ordinary methods of rubber goods manufacture. The process of treatment is known as reclaiming, and the product as reclaimed rubber, reclaim or shoddy.

Many method have been devised. However, all the processes heretofore used on a commercial scale were based essentially upon the depolymerizing action of heat on the rubber itself. Processes in which heat as a depolymerizing agent is used in the presence of substances assisting the regeneration of the rubber are extensively applied. Among these, the most widely known and applied is the alkali process, which consists in heating ground scrap with a dilute solution of caustic soda at temperatures about 350° F. for twelve to twenty hours, and similar processes, in which sodium sulphite and bisulphite, or else magnesium sulphate, zinc chloride, etc., are used instead of the sodium hydrate.

Processes in which small percentages of plasticizing substances of various characters are added to the vulcanised scrap are also known; however, these substances are not eliminated during regeneration, and while they impart to the product some particular characteristics these are always obtained at the expense of its mechanical properties.

Solution processes in which the vulcanized scrap is dissolved by the aid of solvents while subjected to high temperatures, for instance, 175° C., but at any rate never less than 120° C., have also been suggested, the solvent being subsequently recovered by suitable treatment, leaving as a result the reclaimed or regenerated product. These processes have also had some practical application but have not received wide acceptance, because the regenerated products resulting thereby have poor mechanical characteristics. In addition, there are processes representing modifications of such solution processes, in that the dissolving operation is interrupted at a somewhat earlier stage, as soon as the product is capable of being dispersed in water, the treating agents being also in these cases recovered, and the rubber being coagulated from the aqueous emulsion by suitable means.

The primary object of this invention is to provide a novel and improved method of treating vulcanized rubber scrap, whereby a regenerated product of superior quality, practically comparable to crude rubber, is obtained.

Another object is to provide a method of regenerating vulcanized rubber scrap, entailing a succession of relatively simple steps, making it possible not only to produce a superior product, but also to recover the various agents used in the treatment, practically in their entirety.

A further object is to provide a novel and improved method of regenerating vulcanized rubber scrap, whereby the reclaimed product is practically free of the plasticizing agents which might have been present in the original mixture, as well as of the resinous substances resulting from the natural aging of the vulcanized material, or due to the harmful effects of a possible oxidation during the drying operation.

A still further object is to provide a method of treating vulcanized rubber scrap, whereby it is possible to obtain a reclaimed product having a very low acetonic content, in which the original characteristics of the rubber have been restored virtually in their entirety.

Other objects and advantages of the present invention will more fully appear as the description proceeds and will be set forth and claimed in the appended claims.

The method forming the subject of the present invention represents a radical departure from all the other processes heretofore known, in that it utilizes the capacity of the vulcanized scrap to expand or swell by absorption of swelling or solvent substances, without being brought even to the initial stages of actual solution.

In the first stage, the material is treated with a swelling or solvent substance, at a low temperature enabling the material to become impregnated with the said substance, without, however, becoming dissolved therein.

In a succeeding stage, in which the regeneration proper of the rubber takes place under the action of heat, the solvent substance is recovered practically in its entirety, its separation from the regenerated product being effected through suitable washings carried out with a liquid capable of dissolving the swelling or solvent substance only, to the exclusion of the rubber. Thus a regenerated product is obtained, having an exceedingly low acetonic content, which has been freed not only of the swelling or solvent substance, but also of the plasticizing agents which may have been present in the original mixture, and the resinous products derived from the natural aging of the vulcanized material.

The basic characteristics of this new method

of treatment, therefore, resides in the impregnation, at a low temperature, of the previously ground vulcanized rubber, with a high percentage of a swelling or solvent substance. In this connection, it is well to state that by low temperature, a temperature of less than 50° C. is meant, or in any event a temperature materially lower than that required for bringing the vulcanized rubber into solution. It is to be understood that the term "vulcanized rubber" as used herein connotes any vulcanized rubber composition containing natural or artificial rubber in a vulcanized condition with or without softeners, fillers or any other of the compounding ingredients usual in the rubber industry.

The percentage of swelling or solvent substance which may be employed in this treatment of vulcanized rubber is subject to considerable variations, according to the type of scrap treated and the character of the substance itself. At any rate, its proportion is always very high, usually about 100% of the weight of scrap treated, although in special cases it may assume different values. In practice this proportion is determined by the maximum quantity that the scrap is capable of absorbing, without losing its original form as resulting from the grinding operation.

The swollen scrap is then cooked in an autoclave in the presence of water or of one of the usual devulcanizing agents, such as for instance caustic alkalis, alkaline sulphides, alkaline sulphites and hyposulphites, etc. The pressure and temperature of the autoclave, the duration of the heating period, and the concentration of the alkaline liquid if used, depend upon the character of the scrap to be regenerated.

If the operation has been conducted in the presence of an alkaline agent, upon the completion of the heat treatment in the autoclave, the scrap is washed, and then, after drying, is subjected to the washing operation for the recovery of the swelling or solvent substance employed in the previous stage.

This washing operation, by means of which the swelling or solvent agent is recovered practically in its entirety for re-use in subsequent treatments, acts at the same time as a means of purifying the regenerated rubber, in that, as previously stated, it also eliminates from it the plasticizing agents present in the original mixture and those resinous products which may have been formed as a result of the natural aging of the vulcanized rubber.

The washing ended, the liquid used therefor is also recovered, either by direct heating, or by the action of steam, or in vacuum, according to its particular nature.

The regenerated product is ready eventually to be rendered uniform, and, if necessary, to be refined in any one of the ways known to persons skilled in the art.

The selection of the swelling or solvent agent depends upon the character of the scrap to be treated, and is of primary importance; in any event the liquid used should preferably be capable of ready absorption by the vulcanized material, while being scarcely capable of reacting with water or with the alkaline liquid. Among the solvents best adapted for the purpose may be mentioned the medium heavy and heavy mineral oils, the various high boiling fractions resulting from petroleum distillation, and like substances, vegetable tars and oils deriving therefro, coal tar and oil derivatives thereof, resin oil, and synthetic liquids obtained through hydrogenation of naphthalene.

It is obvious, therefore, that the liquid required for the washing of the regenerated product and the recovery of the swelling or solvent agent therefrom should be properly selected with respect to the nature of the latter. The liquids used, according to cases, have been aliphatic alcohols, alky-esters, preferably acetates such as amyl acetate, ethyl acetate, butyl acetate, etc., ketones, particularly acetone, or else suitable mixtures of such solvents. In all cases the liquid used for washing should be a solvent of the swelling material, never of the regenerated rubber. Here also it may be stated that although in specific cases it may be preferable to use one liquid rather than another among those of the character mentioned, their action on the solvent agents used in the preceding stage is sufficiently similar to warrant their being considered virtual equivalents for the purposes of the present invention.

By proper selection of the various steps among those used in common practice, it is possible to recover the swelling or solvent agent and the washing liquid in full, and to have a very high rate of recovery of the alkaline liquid, if it has been used.

If the vulcanized rubber scrap to be treated contains fibrous material, the recovery of the alkaline liquid will be somewhat less; but if the fibrous material is removed before regeneration is carried out this undesirable loss will be avoided.

The above mentioned individual steps, if carried out according to the dictates of good practice, will result in the production of regenerated rubber of very high quality, in which the original characteristics have been almost completely restored. Its acetonic extract varies between 2 and 3%, never reaching over 5%, the chloroformic extract reaches maximums of high value, and the mechanical characteristics of the original mixture are almost entirely regained.

It is also interesting to observe, that by virtue of the operation leading to the recovery of the swelling or solvent agent, the resinous products due either to the natural aging of the original vulcanized material or to the harmful effects of a possible oxidation during the drying operation, are also automatically eliminated. That this is so is confirmed by the low value of the acetonic content, which also gives assurance that the products manufactured with such regenerated rubber will not subsequently age at an unduly fast rate. Furthermore, the regenerated rubber resulting from the new method described is easily workable and easily extruded.

In order to better illustrate my invention, I will now give a practical example relating to the regeneration of 100 kilograms of peelings obtained from old pneumatic tire shoes such as found on the market.

These peelings, in strip form, are ground to pieces of a size somewhat larger than a pea, and these are then placed in a rotating metallic drum together with 100 kilograms of naphtha (petroleum fraction distilling at a temperature over 250° C.). The scrap is left in the drum, at room temperature for about twelve hours, and becomes completely impregnated with the swelling agent without, however, entering into solution.

The scrap thus impregnated is placed in an autoclave in which a 5° Beaumé solution of hydrate of sodium is added in a quantity sufficient to completely cover the scrap. The pressure

in the autoclave is brought to from 12 to 15 atmospheres, and is maintained for about 3 to 4 hours.

When the resulting regenerated product is discharged, the sodium hydrate is recovered by washing with water, and the regenerated rubber is then dried. Once dry, it is placed in an extractor in which, by means of a mixture of amyl-ethyl-butyl acetates, it is freed from the naphtha. The extraction being completed, the naphtha and the mixture of acetates are separately recovered by known methods.

As a final result we have:

	Per cent
Recovery of the sodium hydrate	about 90
Recovery of the naphtha	about 95
Recovery of the mixture of acetates	about 98

The regenerated rubber is thus ready for use. Its chemical characteristics are as follows:

Reaction:		Neutral
Ash	Per cent	7 to 10
Acetonic extract after 24 hours	do	2 to 5
Chloroformic extract after 6 hours	do	13 to 20

From these figures it can be seen that the regenerated rubber possesses the characteristics of a high quality product. Used in mixtures this rubber gives products the physical characteristics of which are comparable to those of mixtures obtained by employing crude rubber.

As previously mentioned, the mode of operation may vary in its details without departing from the inventive idea. Thus with reference to the example above given, the preliminary treatment of the scrap with the swelling or solvent agent may take place in an open tank provided with a suitable agitator; instead of operating at room temperature, the tank or drum could be somewhat heated, for instance to 40° to 50° C., as a result shortening the period of treatment.

Furthermore, some other among the substances previously mentioned, or mixtures thereof in various proportions in relation to the weight of the scrap, could be used as swelling agents instead of naphtha, their proportion, however, being always very high. In such an event, the liquid employed for the recovery of the swelling agent should naturally also be selected accordingly. All of these details, as well as those accompanying the various steps of the method (concentration of the sodium hydrate or other devulcanizing agent used, temperature of the autoclave, etc.), may vary according to the kind of scrap used, and according to the possible advantage in using a given swelling or solvent agent, or a given devulcanizing agent, in preference to another; although said details can be easily determined in each particular case by a technician skilled in the art.

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