PATENT SPECIFICATION

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COMPLETE SPECIFICATION

Improvements in the Production of Crude Rubber from the Serum of Latex Creaming Operations

I, THOMAS ADAM CLAYTON, of 23, Albemarle Street, London, W.1, of British nationality, do hereby declare the invention (Communicated by United States Rubber Company, of 1230, Sixth Avenue, New York, 20, United States of America, a corporation organized and existing under the laws of the State of New Jersey, United States of America), for which I pray that a patent may be granted to me and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to the preparation of off-grade crude rubbers from the serums from latex creaming operations.

It is known that the alkaline serum portion from latex creaming operations, i.e. the skim from mechanical creaming (centrifuging) of latex and the rubber-poor lower liquid fraction from chemical creaming of latex, has a much lower ratio of rubber to non-rubber solids than normal latex. In mixing such serum from conventional creaming operations with acid in the preparation of off-grade crude rubber therefrom, the particles flocculate and rise to the top in the form of discrete aggregates which do not coalesce in the form of a lump, as in the case of normal latex coagulation, and these aggregates or flocs are not workable for formation into crude rubber by the usual milling methods. The centrifuging or chemical creaming operations may be regulated so that the serum will have a sufficiently high ratio of rubber to non-rubber solids to assure acid coagulation of the serum in the form of a workable coherent lump but this necessitates diverting a correspondingly larger proportion of rubber to the serum with a consequent loss of rubber from the more valuable latex concentrate.

By the present invention, latex serums that normally flocculate on mixing with acid are made to produce a workable lump coagulum that may be readily sheeted in conventional milling apparatus.

The method according to the present invention comprises the steps of flocculating the serum from the latex concentration in known manner by mixing with an acid to reduce the pH to the isoelectric point or below, separating the flocs from the subnatant liquid, mixing with the wet flocs a liquid containing dispersed normal or creamed rubber particles in such amount that the dry weight of the added rubber (as determined by acid coagulation) is at least 0.25 times the dry weight of the wet flocs, adding acid to said mixture of flocs and rubber particles to coagulate the same into a coherent lump, constituting off grade crude rubber, which may thereafter be sheeted. According to a preferred embodiment of the present invention the liquid containing the normal or creamed rubber particles is plant wash water from latex or cream storage or creaming tanks and pipes, or from latex centrifuges or centrifuged latex storage tanks.

The plant wash water or other source of normal or creamed latex particles is added in any desired larger proportion to the flocs from the serum, as for example, in amounts such that the dryweight of the added rubber (as determined by acid coagulation) is up to two times the dry weight of the flocs, or larger amounts may be added if excess washings are available. The flocs and latex particles, with addition of more water if desired, are then mixed with additional sulfuric, acetic or formic acid to lower the pH of the flocs and latex particles to the iso-electric point or below, whereby there is

formed a coherent lump coagulum on standing which may be readily sheeted in the usual manner, as on mills for forming crepe. It has been found that with the preliminary separation of the flocs from the initial serum flocculation in the process of the present invention, a much smaller amount of added normal or concentrated latex particles will give a workable lump coagulum than if the normal or concentrated latex particles were added to the initial serum and the mixture was coagulated directly with acid. In adding normal or concentrated latex particles to the serum before coagulation, it takes an amount of added rubber (as determined by acid coagulation) at least about two times the weight of dry rubber (as determined by acid coagulation) in the serum, to assure a workable lump coagulum.

The following examples illustrate the invention, all parts and percentages referred to being by weight:

EXAMPLE I.

480 parts of a serum from a creaming operation having a total solids (T.S.) of 7.2% and dry rubber content by acid coagulation (D.R.C.) of 3.6% and ammonia (NH_3) content of 1.9% was run into a tank. A 5% aqueous solution of sulfuric acid was added to the serum with mixing until flocculation occurred. The batch was allowed to stand several hours, whereupon pinhead size flocs rose to the surface leaving an amber-coloured underlayer which was drained from the flocs. 100 parts of plant wash water having a T.S. of 12.9% and D.R.C. of 12.0% and NH_3 content of 0.3% were mixed with the flocs in the tank, after which 5% aqueous sulfuric acid was added in amount to cause coagulation and, on standing, separation of a coherent lump coagulum which was removed and sheeted on conventional milling apparatus, generally used for crepe. The dry weight of the added rubber was 0.69 times the dry weight of the wet flocs.

EXAMPLE II.

The procedure of Example I was carried out using 250 parts of a serum from a creaming operation having a T.S. of 7.1% and a D.R.C. of 3.2% and an NH_3 content of 1.9%. After flocculating and drawing off the amber-coloured underlayer, 79 parts of plant wash water having a T.S. of 12.6% and a D.R.C. of 11.6% and an NH_3 content of 0.3% were added to the flocs in the tank, after which the mixture was coagulated with sulfuric acid to form a coherent lump or off-grade crude rubber which was removed and sheeted. The dry weight of the added rubber was 1.14 times the dry weight of the wet flocs.

EXAMPLE III.

A 5% aqueous solution of sulfuric acid in amount to flocculate 150 parts of a serum from a creaming operation was run into a tank. The 150 parts of the serum having a T.S. of 7.6% and a D.R.C. of 4.4% and an NH_3 content of 1.9% were then run into a tank containing the sulfuric acid, and mixed therewith. After standing several hours, the flocs rose to the surface, more in the form of a thick cream than the pinhead size flocs in Examples I and II. The amber-coloured underlayer was drained from the upper-layer of cream-like flocs and 20 parts of a plant wash water having a T.S. of 12.9% and a D.R.C. of 12.4% and an NH_3 content of 0.3% were mixed with the cream-like flocs in the tank. An aqueous 5% solution of sulfuric acid was added in amount to cause coagulation, which on standing resulted in a coherent lump coagulum which was removed and sheeted on conventional milling apparatus. The dry weight of the added rubber was 0.37 times the dry weight of the wet flocs.

EXAMPLE IV.

The procedure of Example III was carried out using 100 parts of a serum having a T.S. of 7.3% and a D.R.C. of 3.9% and an NH_3 content of 1.9%. After flocculating to a cream-like floc, as in Example III, and drawing off the amber-coloured underlayer, 27.4 parts of plant wash water having a T.S. of 14.4% and a D.R.C. of 13.7% and an NH_3 content of 0.2% were added to the cream-like flocs in the tank, after which the mixture was coagulated with sulfuric acid to a coherent lump, which was removed and made into off-grade crude rubber. The dry weight of the added rubber was 0.98 times the dry weight of the wet flocs.

What I claim is:—

1. A method for the preparation of off-grade crude rubber from the serum from a latex creaming operation which comprises flocculating the serum with acid, separating the flocs from the lower aqueous layer, mixing with the separated flocs a liquid containing dispersed normal or creamed rubber particles in such amount that the dry weight of the added rubber (as determined by acid coagulation) is at least one-fourth the dry weight of the flocs, adding acid to said mixture of flocs and rubber particles to coagulate the same into a coherent lump constituting off-grade crude rubber.

2. A method according to claim 1, including the step of sheeting said off-grade rubber.

3. The method of claim 1 in which the liquid containing the dispersed normal or

formed a coherent lump coagulum on standing which may be readily sheeted in the usual manner, as on mills for forming crepe. It has been found that with the preliminary separation of the flocs from the initial serum flocculation in the process of the present invention, a much smaller amount of added normal or concentrated latex particles will give a workable lump coagulum than if the normal or concentrated latex particles were added to the initial serum and the mixture was coagulated directly with acid. In adding normal or concentrated latex particles to the serum before coagulation, it takes an amount of added rubber (as determined by acid coagulation) at least about two times the weight of dry rubber (as determined by acid coagulation) in the serum, to assure a workable lump coagulum.

The following examples illustrate the invention, all parts and percentages referred to being by weight:

EXAMPLE I.

480 parts of a serum from a creaming operation having a total solids (T.S.) of 7.2% and dry rubber content by acid coagulation (D.R.C.) of 3.6% and ammonia (NH_3) content of 1.9% was run into a tank. A 5% aqueous solution of sulfuric acid was added to the serum with mixing until flocculation occurred. The batch was allowed to stand several hours, whereupon pinhead size flocs rose to the surface leaving an amber-coloured underlayer which was drained from the flocs. 100 parts of plant wash water having a T.S. of 12.9% and D.R.C. of 12.0% and NH_3 content of 0.3% were mixed with the flocs in the tank, after which 5% aqueous sulfuric acid was added in amount to cause coagulation and, on standing, separation of a coherent lump coagulum which was removed and sheeted on conventional milling apparatus, generally used for crepe. The dry weight of the added rubber was 0.69 times the dry weight of the wet flocs.

EXAMPLE II.

The procedure of Example I was carried out using 250 parts of a serum from a creaming operation having a T.S. of 7.1% and a D.R.C. of 3.2% and an NH_3 content of 1.9%. After flocculating and drawing off the amber-coloured underlayer, 79 parts of plant wash water having a T.S. of 12.6% and a D.R.C. of 11.6% and an NH_3 content of 0.3% were added to the flocs in the tank, after which the mixture was coagulated with sulfuric acid to form a coherent lump or off-grade crude rubber which was removed and sheeted. The dry weight of the added rubber was 1.14 times the dry weight of the wet flocs.

EXAMPLE III.

A 5% aqueous solution of sulfuric acid in amount to flocculate 150 parts of a serum from a creaming operation was run into a tank. The 150 parts of the serum having a T.S. of 7.6% and a D.R.C. of 4.4% and an NH_3 content of 1.9% were then run into a tank containing the sulfuric acid, and mixed therewith. After standing several hours, the flocs rose to the surface, more in the form of a thick cream than the pinhead size flocs in Examples I and II. The amber-coloured underlayer was drained from the upper-layer of cream-like flocs and 20 parts of a plant wash water having a T.S. of 12.9% and a D.R.C. of 12.4% and an NH_3 content of 0.3% were mixed with the cream-like flocs in the tank. An aqueous 5% solution of sulfuric acid was added in amount to cause coagulation, which on standing resulted in a coherent lump coagulum which was removed and sheeted on conventional milling apparatus. The dry weight of the added rubber was 0.37 times the dry weight of the wet flocs.

EXAMPLE IV.

The procedure of Example III was carried out using 100 parts of a serum having a T.S. of 7.3% and a D.R.C. of 3.9% and an NH_3 content of 1.9%. After flocculating to a cream-like floc, as in Example III, and drawing off the amber-coloured underlayer, 27.4 parts of plant wash water having a T.S. of 14.4% and a D.R.C. of 13.7% and an NH_3 content of 0.2% were added to the cream-like flocs in the tank, after which the mixture was coagulated with sulfuric acid to a coherent lump, which was removed and made into off-grade crude rubber. The dry weight of the added rubber was 0.98 times the dry weight of the wet flocs.

What I claim is:—

1. A method for the preparation of off-grade crude rubber from the serum from a latex creaming operation which comprises flocculating the serum with acid, separating the flocs from the lower aqueous layer, mixing with the separated flocs a liquid containing dispersed normal or creamed rubber particles in such amount that the dry weight of the added rubber (as determined by acid coagulation) is at least one-fourth the dry weight of the flocs, adding acid to said mixture of flocs and rubber particles to coagulate the same into a coherent lump constituting off-grade crude rubber.

2. A method according to claim 1, including the step of sheeting said off-grade rubber.

3. The method of claim 1 in which the liquid containing the dispersed normal or

creamed rubber particles is plant wash water from latex or creamed latex storage or chemical creaming tanks or pipes or from latex centrifuges or centrifuged latex storage tanks.

4. The method of claim 1 in which the dry weight of the added particles of normal or creamed rubber is one-fourth to two times the dry weight of the flocs.

Dated this 7th day of July, 1950.

T. A. CLAYTON.

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