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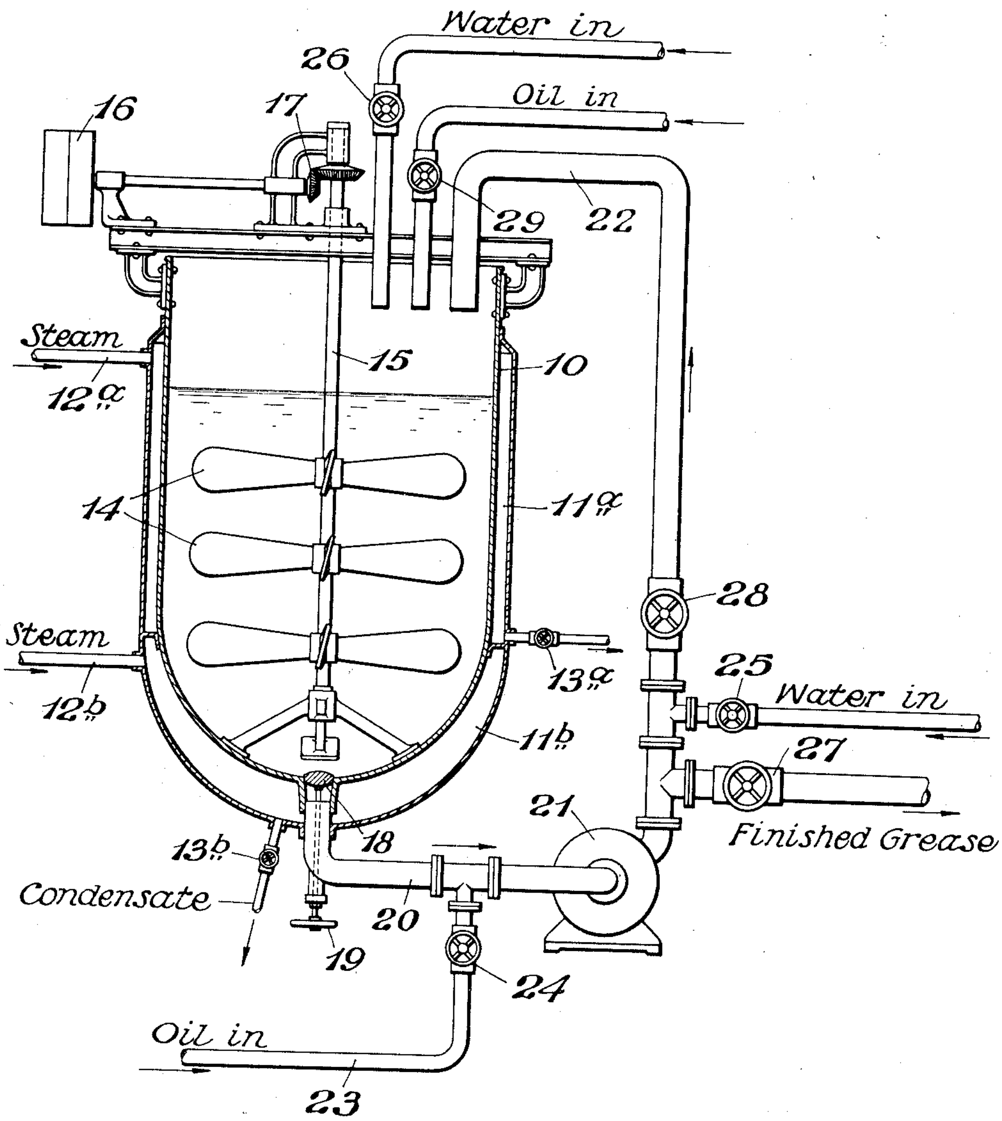


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## GREASE MANUFACTURE

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This invention relates to the manufacture of greases and particularly to the manufacture of calcium soap greases in open kettles.

It is an object of my invention to provide a method for the manufacture of calcium soap greases such as cup greases, pressure gun greases, transmission greases, etc., which will produce a higher quality and more uniform grease than has previously been possible using open kettles. It is a further object of my invention to provide a method of grease manufacture in which the necessary time for the completion of a batch of grease is greatly reduced. A still further object of my invention is to provide a process whereby a grease free from lumps and having a smooth, uniform texture may be produced. Another important object of my invention is to provide a method whereby a greater amount of oil and a lesser amount of soap can be used to produce a grease of a given consistency as compared with prior art methods. It is also an object of my invention to provide a method of grease manufacture which is simpler to carry out than those of the prior art. Another object is to provide a method of grease manufacture which will produce greases the consistencies of which will stay constant on storage. Other and more detailed objects of my invention will become apparent as the description thereof proceeds.

The various types of calcium soap greases are by far the commonest and most important types of greases used by modern industry. In general these greases consist of calcium soap, oil and water, although other constituents may be present in some cases. The various grades of cup greases may contain from 10% up to 25% or even 30% of calcium soap, from ½% to 1½% of water, together with lubricating oil of the desired viscosity to make 100%. A typical pressure gun grease is similar to a cup grease but contains in the neighborhood of 10% calcium soap. Transmission greases are again similar but are commonly characterized by a lower soap content, for instance 3% to 6%. In all these cases the optimum water content varies directly with the soap content so that in the case of transmission greases the most desirable water content is somewhat under ½%. All of the percentages given are on a weight basis.

These various types of calcium soap greases have in the past been made in mixers of the open kettle type of from 5 to 15 feet in diameter and of depth somewhat greater than their diameter. These kettles are usually and preferably heated by means of steam jackets, but in some cases they are heated by direct firing. More recently, closed kettles in which the grease can be subjected to super-atmospheric pressures have been used and it is possible to achieve some of the improvements which characterize my invention by the use of pressure equipment of this type. This equipment is, however, highly expensive and has other disadvantages. My invention makes it possible to achieve the results characteristic of the use of pressure kettles without their disadvantages by the use of open kettles of the type heretofore used.

Kettles used in grease manufacture are customarily equipped with internal stirrers of a rotary type.

Various procedures have been used in the past in making calcium soap greases in open kettles. One of the best of those known to the prior art and one which is used in common practice in the manufacture of cup greases consists of charging the lime, fatty acid and about 10% of the total oil required into an open kettle equipped with an internal mixer and heating to about 235° F. to 250° F., at which temperature neutralization, or as commonly called in the trade, "saponification" is completed. According to this prior art method the soap thus formed was then allowed to cool to about the boiling point of water or slightly above the boiling point of water, after which the required amount of water was rapidly added. Before the water could turn to steam and be driven off from the soap, cold oil was added as rapidly as possible to cool the batch and the grease was thus formed.

In many cases it has been found desirable to use a little caustic soda along with the lime. This appears to speed up the "saponification" and to give a somewhat better result. From 1% to 10% of the required lime may suitably be replaced by caustic soda. Although my invention is applicable to greases made from lime without the addition of soda, it is particularly applicable to greases in which some soda is used.

My improved method of making cup greases, pressure gun greases and transmission greases, while similar to the old up to the point where the "saponification" has been completed at a temperature of about 215° F. to 250° F. and preferably 230° F. to 250° F., differs radically in procedure from this point on. Furthermore, I prefer to use more oil at the start of the batch than has been characteristic of the prior art. I prefer to add from about 15% to about 30% of the total oil required at the beginning of the batch instead of about 10% as has heretofore been typical. After "saponification" is completed I discontinue heating the kettle and small streams of oil and water are started in at once. The water stream is regulated by the amount of foaming. The batch will rise a few inches and must not be allowed to foam very much. Cutting down the size of the water stream will reduce the foaming should it be considered to be too great.

The batch is circulated by means of a pump taking suction from the bottom of the kettle and discharging into the top; this circulation is started at the time the oil and water streams are started. As the batch cools down it will go through a characteristic change in condition and appearance at about the boiling point of water. This change in condition turns the batch from a "soapy" structure to a typical grease and the batch becomes much heavier.

I believe that this change in condition is caused by an inversion of the emulsion. In other words the soapy structure appears to be an oil-in-water emulsion, whereas the final grease should be a water-in-oil emulsion. Some of the advantages of my invention appear to be due to the fact that the grease constituents are kept in constant equilibrium with water during the cooling and up to the point at which this change in condition occurs.

When the cooling has proceeded past the point at which this change in condition occurs, the water content of the finished grease can be considered fixed and the water stream is shut off. The batch is then finished by adding the remainder of the required oil. This oil can be added as rapidly as it can be incorporated in the grease.

By circulating the batch and adding the water and oil in the described method I am able to finish the grease batch in about two-thirds the time required by the old procedure.

Other advantages of my method are:

1. The chance of producing a grease full of "lumps" is greatly reduced, due to the fact that a larger amount of oil is used at the start and all changes take place at a gradual and uniform rate. In the old method in which a large amount of water and oil is dumped in on a heavy soap a large portion of the batches turn out to be lumpy or otherwise unsatisfactory.

2. My procedure also gives a smooth grease of uniform texture without graininess. This is especially valuable in the case of heavy pressure gun greases.

3. I am enabled to use a greater amount of oil in proportion to the soap, which is one of the principal advantages of my method. Oil is much cheaper than soap and it is desired to use as high a proportion of it as possible and yet make the same quality of product.

The preceding description has had to do principally with the manufacture of greases from ordinary fatty acids such as oleic acid, mixed animal fatty acids, etc. My invention is, however, particularly important in the manufacture of greases for use at more elevated temperatures from fatty acids made from hydrogenated fats. These fatty acids are very high in true stearic acid content and although the use of soaps made from these acids is highly desirable it has been found very difficult to produce the desired greases from them since it is necessary to use more elevated temperatures than in the case of oleic acid or mixed fatty acids. In the prior art procedure in which greases were made by the open kettle method from fatty acids split from hydrogenated fats, about one batch out of four was spoiled and even the remaining batches were of poor quality. The greases produced were often cloudy and the oil would tend to "leak" out of them.

As an example of the manufacture of greases according to my invention from fatty acids split from hydrogenated fats (in other words, from relatively pure stearic acid), I add the required amount of lime to a small amount of the previous batch of grease left in the kettle and I then add about 25% of the total required oil. After the old grease, lime and oil have been stirred up to a smooth consistency I add the fatty acids. If the oil is added after the fatty acids there is considerably more trouble from foaming.

Next I turn steam into the steam jacket surrounding the bottom of the kettle and when the batch has reached a smooth consistency and is up to about 150° F. to 175° F. temperature, I add the caustic soda solution. The batch will foam very little unless the operator tries to speed up by using side jacket steam in order to gain time. I try to keep down foaming as much as possible.

As in the preparation of cup greases, I circulate the mix by means of a small pump beginning when the batch reaches 200° F. As stated before, this cuts down the tendency to foam and aids in keeping all constituents in a smooth even mix. I let the temperature of the batch get up to 230° F. to 250° F. at which time the soap will be clear and stringy in appearance when hot and brittle when cold. Then I turn off the steam, bleed the jackets, start a small stream of water in the mixer and regulate the size of the stream by the amount of foaming. Circulation of the mixture through the pump is continued. If the mix starts to rise in the kettle the stream of water is cut off until it settles down.

As the mix cools down it will go through a characteristic change in appearance at about 220° F. to 225° F. Instead of a "soapy" appearance it will assume the typical appearance of a grease and will get much heavier. After this change has taken place a small stream of oil may be turned in to hasten the cooling. At or slightly above the boiling point of water the mix will be smooth in appearance and free from foam and tend to absorb more of the water instead of throwing it off as vapor, even though the mix is still above the boiling point of water. The proper moisture content can be fixed at this point before cooling the batch further.

After the addition of water is stopped, the remainder of the oil is added as rapidly as it can be incorporated in the grease. The temperature of the oil should be about 130° F. to 150° F. The above procedure will finish up a batch of this type of grease in about 5 to 5½ hours with the temperature of the batch at the finish about 190° F. The steam pressure which I use is about 100 pounds per square inch and some variation in time would result if steam at pressures other than that were used.

In operating according to my invention in the manufacture of greases from fatty acids split from hydrogenated fats, I find that the following advantages over prior art methods are obtained: the manipulation is much simpler and easier to carry out; I consistently obtain batches the properties of which check each other very closely; samples taken for grease consistency on the day the batch is made give a true indication of the consistency of the product after some time in storage; it is possible to use more oil in proportion to the soap used and yet get the same consistency, thereby cutting down the cost of my product without sacrificing quality; as the mixes are filled out and become cooler, the mass in the center of the larger containers does not lose moisture and separate or become crumbly, as sometimes happens with the old method of procedure; a high quality grease can be made from a poorer grade of fatty acid; a heavy grade of grease can be "graded up" to any lighter grade by adding more oil, whereas this was not possible with older methods; and I do not get grainy, cloudy or off test batches by this new procedure and can duplicate in an open kettle the greases made by pressure mixers.

It will be noted that after the soap has been formed in a portion of the oil the procedure is much the same when using fatty acids split from hydrogenated fats as when using ordinary oleic acid or mixed fatty acids. The main differences are that when using fatty acids made from hydrogenated fats the minimum soap making temperature is higher (230° F. to 250° F. as compared with 215° F. to 250° F.) and that the characteristic structural change also occurs at a higher temperature (220° F. to 225° F. instead of about 210° F.).

In either case, one of the most important features of my invention consists in slowly cooling the grease from a temperature within the range from about 215° F. to 250° F. to a critical temperature at which the characteristic structural change occurs (about 210° F. to 225° F. depending on the particular grease) while adding water and preferably oil very gradually and with especially thorough mixing. External mixing by removing grease from the bottom of the kettle and recirculating it to the top of the kettle is important if best results are to be obtained, as it provides exceptionally good agitation and contact between the phases in a closed space out of contact with air which would carry off water vapor rapidly at the temperature required.

The water content should be fixed at about the aforementioned critical temperature. After this critical point is passed the remainder of the oil can be added rapidly and the grease is finished in the usual way.

The preceding description has had particular reference to the manufacture of calcium soap greases from fatty acids. My invention is, however, also applicable to the manufacture of greases from fats. Thus, for instance, my invention can be applied to the manufacture of a lime-tallow grease in which the batch is heated to about 300° F. and held at this temperature during the saponification of the tallow.

While water must be present during the formation of a soap from fatty acid and lime, it is a necessary constituent in the reaction whereby a fat such as tallow (i. e. a glycerine fatty acid ester) is saponified:

$$2C_3H_5(COOR)_3+3CaO+3H_2O= 2C_3H_5(OH)_3+3Ca(COOR)_2$$

By my method, where water is added continuously in a small stream a supply of water needed for the saponification is present at all times. Also I can keep the batch at a higher temperature without danger of it foaming over, than can be done where the water is added in batches.

I also find that highly superior results can be obtained by recirculating grease from the bottom to the top of the mixer during the gradual addition of the water, thereby accomplishing external mixing, and permitting the water to be absorbed by the grease without foaming.

The saponification of tallow by this method takes but five hours where by the old method it required about 20 hours. The saving in time is due to my being able to hold the batch at a higher temperature (for instance 275° F. or higher). After the saponification of the tallow is completed, the procedure for making grease is the same as in the case of greases made from fatty acids as previously described.

Thus for instance a batch of transmission grease was made with lime and tallow in seven hours with as good a yield as I usually get when using free fatty acids and lime. Furthermore, the batch was made in about the same length of time (7 hours) as when it was made by the old method from fatty acids.

The advantages which my method has over the old method in making these lime-tallow greases, are the same as those previously cited for greases made from ordinary fatty acids. As stated before, my method hastens the saponification by adding the water as fast as it is driven off and by insuring at all times a sufficient supply of water for the reaction. In the old method if sufficient water is added in a batch at the start the high temperature cannot be obtained because of foaming. The new method makes it possible to use fats or fatty acids interchangeably which sometimes has economic advantages.

As previously stated my new method of grease manufacture utilizes the customary open kettle mixers except that provision is made for external mixing by pumping a portion of the grease from the bottom of the kettle to the top.

The accompanying drawing which forms a portion of this specification shows an elevation, partly in section, of an apparatus for carrying out my invention.

Referring more particularly to the drawing, an open-topped kettle **10**, is provided with a side steam jacket **11*a*** and a bottom steam jacket **11*b***, both of which may suitably be lagged. Steam is admitted through inlets **12*a*** and **12*b*** and condensate is removed through valves **13*a*** and **13*b***. These valves can also be used to bleed steam from the jacket when it is desired to cool the grease.

The kettle is provided with the customary rotary mixers **14** carried by shaft **15** which is rotated by means of pulley **16** through gears **17**. Elements **14**, **15**, **16** and **17** may suitably be supported by an I-beam across the top of the kettle as shown.

Grease is withdrawn from the base of the kettle through valve **18** (operated by handle **19**), pipe **20** and centrifugal pump **21** and is reintroduced into the top of the kettle through pipe **22**.

Water and oil can be added along with the recirculated grease or may be introduced directly into the top of the kettle. Thus oil may be added through pipe **23** and valve **24** which take suction from pump **21** or it may be added directly to the top of the kettle through valve **29**. It is preferred not to add water directly in advance of pump **21** since this may result in vapor-locking the pump. Water is therefore added under the necessary pressure, through valve **25** or valve **26**.

The finished grease may be removed directly from the bottom of the kettle or through valve **27** by means of pump **21** (after closing valves **24**, **25** and **28**). The various pipes, etc. may suitably be lagged or otherwise insulated or heated to prevent heat losses and clogging by cold grease.

To summarize my invention, all of my methods have as a necessary step the making of a soap in the presence of oil, the relative amount of the latter varying with the hardness of the soap desired, and adding water gradually to the soap mass so that the latter becomes a "grease" of a stable nature. In most cases this is carried out at temperatures in the vicinity of the boiling point of water, but in some cases, as for instance when fatty acids split from hydrogenated fats are used, it must be done well above the boiling point of water (e. g. 220° F.). All methods require that the grease be cooled sufficiently to retain a part of the water. Vigorous mixing, preferably including external mixing by recycling material from the bottom of the kettle to the top, is also important. Also if the greases are reheated sufficiently to drive off the water, the soap and oil will separate and the greases be ruined.

My new method makes all changes in the grease more gradual, yet accomplishes these changes in a shorter overall time due to my circulating the mix by means of a pump, and to mixing the constituents at higher temperatures than was possible under the old method and while the soap is still very heavy. The gradual changes in structure of the mix ensure that all of the soap will be used to its best advantage. This gives much greater yields of grease for a given amount of soap.

While I have described my invention in connection with certain specific embodiments thereof and together with certain theories to explain its advantages it is to be understood that these are by way of illustration rather than by way of limitation and I do not mean to be limited thereto but only to a liberal, valid interpretation of the appended claims in which I will define the novel and patentable features of my invention.

I claim:

1. A method for the manufacture of a calcium soap grease in an open grease kettle which comprises forming the calcium soap in a portion of the lubricating oil required in the grease, heating the soap-oil mixture thus formed to a temperature within the range from about 230° F. to about 250° F., cooling the mixture from a temperature within said range to a temperature at which said mixture changes from a soapy structure to a greasy structure, slowly and gradually adding water to said mixture throughout substantially the whole of said cooling, said change from a soapy structure to a greasy structure occurring at a temperature lower than said first-mentioned temperature and within the range from about 210° F. to about 225° F., and adding the remainder of the required lubricating oil, the addition of water and oil and being accompanied by constant mixing of the various materials involved.

2. A method for the manufacture of a calcium soap grease in an open grease kettle which comprises forming the calcium soap in a portion of the lubricating oil required in the grease, heating the soap-oil mixture thus formed to a temperature within the range from about 230° F. to about 250° F., discontinuing heating at said temperature, slowly and gradually adding water and a small portion of the required lubricating oil while cooling the mixture from a temperature within said range to a temperature at which said mixture changes from a soapy structure to a greasy structure, said change occurring at a temperature lower than said first-mentioned temperature and within the range from about 210° F. to about 225° F., and adding the remainder of the required lubricating oil without further heating, the addition of water and oil being accompanied by constant mixing of the various materials involved.

3. A method for the manufacture of a calcium soap grease in an open mixing kettle which comprises forming the calcium soap in a portion of the lubricating oil required in the manufacture of said grease, heating the soap-oil mixture to a temperature above about 230° F., discontinuing heating at said temperature, adding water to said mixture slowly and gradually beginning at a temperature above about 230° F. while permitting said mixture to cool and while subjecting said mixture to thorough mixing, said mixing comprising externally circulating a portion of said mixture from the bottom of said kettle to the top of said kettle, continuing said cooling until a temperature is reached at which the mixture changes from a characteristic soapy appearance to a characteristic greasy appearance, and then adding the remainder of the required lubricating oil while continuing the mixing operation.

4. A method for the manufacture of a calcium soap grease in an open kettle which comprises forming the calcium soap in at least about 15% of the total oil required in the final grease, mixing the oil and soap, heating the oil and soap mixture to a temperature of at least about 230° F., adding water to the mixture while permitting the mixture to cool from a temperature of at least about 230° F. and while recycling a portion of the mixture from the bottom of said kettle to the top of said kettle until a temperature within the range from about 210° F. to about 225° F. is reached, at which temperature the mixture changes from a characteristic soapy appearance to a characteristic greasy appearance, discontinuing the addition of water at about the boiling point of water, further cooling the mixture, and adding the remainder of the oil required in the final grease and incorporating said oil with the other constituents present.

5. In a method for the manufacture of a calcium soap grease in an open kettle, the steps which comprise subjecting a mixture of calcium soap and lubricating oil to a temperature of at least about 230° F., and then cooling said mixture while subjecting it to vigorous mixing and while gradually adding water beginning at a temperature of at least about 230° F. and ending when a temperature is reached at which the mass changes from a characteristic soapy appearance to a characteristic greasy appearance.

6. In the manufacture of a calcium soap grease in an open kettle the steps which comprise subjecting a mixture of calcium soap and lubricating oil to a temperature within the range from about 230° F. to about 250° F., slowly and gradually adding water and further lubricating oil beginning at said temperature while permitting the mixture to cool and while subjecting the mixture to further mixing, and discontinuing the addition of water at a temperature within the range from about 210° F. to about 225° F.

7. In the manufacture of a calcium soap grease in an open kettle the steps which comprise subjecting a mixture of calcium soap and lubricating oil to a temperature within the range from about 230° F. to about 250° F. slowly and gradually adding water and further lubricating oil beginning at said temperature while permitting the mixture to cool, while subjecting the mixture to further mixing and while externally circulating a portion of the mixture from the bottom of said kettle to the top of said kettle, and discontinuing

the addition of water at about the boiling point of water.

8. A method of manufacturing a calcium soap grease in a mixer of the open steam kettle type which comprises forming a calcium soap from lime and fatty acids in from about 15% to about 30% of the total lubricating oil required in said grease, heating the soap oil mixture to a temperature of from about 230° F. to about 250° F., cooling said mixture while subjecting it to further mixing and while externally circulating a portion of said mixture from the bottom of said kettle to the top of said kettle, slowly and gradually adding water substantially throughout said cooling until a temperature within the range from about 210° F. to about 225° F. is reached, discontinuing the addition of water at about the boiling point of water, and further cooling the mixture while adding and incorporating the remainder of the required lubricating oil.

9. A method for the manufacture of a calcium soap grease in an open kettle which comprises forming soap from lime, a small amount of soda, and fatty acids split from hydrogenated fats in a portion of the lubricating oil required in the grease, mixing the soap and oil, heating the mixture to a temperature within the range from about 230° F. to about 250° F., gradually adding water and oil beginning at said temperature while cooling the mixture and while subjecting the mixture to further mixing, and completing the addition of the required water at a temperature of about 210° F.

10. A method for the manufacture of a calcium soap grease in an open kettle which comprises forming soap from lime, a small amount of soda and fatty acids split from hydrogenated fats in a portion of the lubricating oil required in the grease, mixing the soap and oil, heating the mixture to a temperature within the range from about 230° F. to about 250° F., gradually adding water and oil beginning at said temperature while cooling the mixture and while externally circulating a portion of said mixture from the bottom of said kettle to the top of said kettle, and completing the addition of the required water at a temperature of about 210° F.

11. A process for the manufacture of a calcium soap grease from lime and fat which comprises saponifying the fat with lime in the presence of a portion of the required lubricating oil while slowly and gradually adding water and while externally circulating a portion of the mixture from a point distant from the point at which said water is added to a point adjacent the point at which said water is added, completing the saponification, bringing the mixture thus formed to a temperature within the range from about 230° F. to about 250° F., slowly and gradually adding the additional water required to produce a grease structure while cooling the mixture from said temperature to a temperature at which the mixture changes from a characteristic soapy appearance to a characteristic greasy appearance.

12. Steps according to claim 5 in which said vigorous mixing comprises externally circulating a portion of the mixture from the bottom of said kettle to the top of said kettle.

13. A process according to claim 11 in which the cooling and addition of water are accompanied by additional external circulation of a portion of the mixture.

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