

PATENT SPECIFICATION

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(54) COAGULATION OF RUBBER LATEX

(71) We, THE BOARD OF THE RUBBER RESEARCH INSTITUTE OF MALAYSIA, a body Corporate of Malaysia, of 260, Jalan Ampang, P.O. Box 150, Kuala Lumpur, Malaysia, do hereby declare the invention, for which we pray that a Patent may be granted to us, 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 processing of natural rubber whereby the fresh or preserved field latex is continuously and rapidly gelled by the application of heat and the rubber coagulum is ready for drying or crumbled prior to drying. The dry rubber content of the field latex may range from 10 percent to 40 percent. The invention also applies to field latex whose dry rubber content has been raised above 40 percent by light centrifugation. This process of heat gelation can be effected by a heat exchanger or other means where the external heating medium is maintained at a temperature usually exceeding 100°C.

The conventional method of processing natural rubber provides for the coagulation of preserved field latex, usually by acid and other chemicals, and the subsequent maturation of the coagulum in receptacles usually for at least five hours before the coagulum is crumbled and ready for drying. The disadvantages in this method are:

1. The chemical cost for coagulation can be high
2. The high capital expenditure and the large floor area occupied by the receptacles for storage of the coagulum, and
3. The labour and the equipment for handling and crumbling the coagulum.

Previous attempts at continuous coagulation of field latex with the object of eliminating the intermediate steps of storage and handling of coagulum have not been successful. These attempts used chemical agents to effect coagulation, had processing difficulties and produced rubbers which did not meet the required standards for technically specified rubbers.

The basis of the present invention is that fresh or preserved field latex can be rapidly gelled by heat supplied to it through a heat exchanger. The temperature of gelation is usually between 70 to 80°C but the range may extend from 60 to 110°C, depending on the stability of the latex. The heating medium in the heat exchanger must be capable of being operated at temperatures between 100°C and 300°C. At these high operating temperatures, the latex can be gelled within a very short time, generally within three minutes and often less than a minute; the output of gelled rubber coagulum from the heat exchanger can therefore be maintained at a high level.

The present invention accordingly provides a method of producing raw solid natural rubber in bulk form, which method comprises the steps of:—

- (1) Passing a continuous stream of fresh or preserved natural rubber field latex through a heat exchanger in which the heating medium is maintained at a temperature from 100°C. to 300°C., so as to raise the temperature of the latex to from 60°C to 110°C., whereby gelation of the rubber is caused to take place rapidly;
- (2) optionally, crumbling the coagulum
- (3) drying the coagulum and
- (4) pressing and palleting the coagulum to form raw solid natural rubber in bulk.

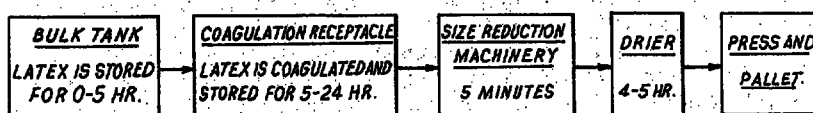
The gelled coagulum is ready for drying or crumbled prior to drying. The temperature of the heating medium can be adjusted to control the time of gelation of latex of different stability. The flow chart of this continuous heat gelation

process is compared with that of the conventional process for the production of block rubbers in Table A. A rough indication of the time spent in each operation or station is included in this table to emphasise the relative continuity of the two processes from the latex to the dry rubber.

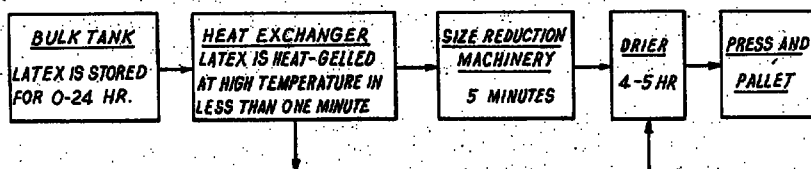
This process avoids the high cost of coagulation by chemical means, dispenses with the need to store rubber coagulum in receptacles for at least five hours and reduces the amount of processing machinery required. A rubber processing factory using this process requires less floor area than that of a conventional process factory.

An added advantage of this invention is that the high levels of ammonia required to preserve latex adequately for at least 24 hours do not affect significantly the time of gelation; this is particularly true when the latex has been stored for the requisite period of time. For example, latex preserved with 0.15 to 0.20% wt. (on latex) ammonia and stored for 20—24 hours gells as rapidly as lightly ammoniated fresh latex. This is an important feature of the process since previous attempts at devising a practical method of continuous coagulation of latex by chemical means have failed because latex preserved with high levels of ammonia has caused processing problems.

TABLE A.
COMPARATIVE SCHEMATIC FLOW CHARTS OF THE CONTINUOUS HEAT GELATION AND THE CONVENTIONAL ACID COAGULATION PROCESSES



(A) THE CONVENTIONAL ACID COAGULATION PROCESS



(B) THE CONTINUOUS HEAT GELATION PROCESS

The efficiency and the time of gelation of latex can be improved by the addition of heat-sensitising agents to the latex. The user of these heat sensitising agents, however, can be dispensed with when gelation is brought about by maintaining the heating medium at a high temperature. Examples of useful heat sensitising agents are the polypropylene glycols (molecular weight 500—1000) and polyvinyl methyl ether. The polypropylene glycols are particularly suitable since these chemicals do not destabilise the latex. Generally from 0.1% to 1% by weight on the weight of the dry rubber provides a satisfactory heat sensitising effect, but a higher dosage may be used for the purpose of improving certain technological properties of the resultant rubber.

The rubber latex may be passed through a plurality of tubes contained in a heat exchanger. Suitable tube (internal) diameters vary between 0.25 in. to 1.0 in. If suitable tubes with internal diameter less than 0.25 in. can be made, then the gelled coagulum may be dried directly, thereby omitting the additional step of size reduction. A suitable heat transfer medium for such a heat exchanger is petroleum oil which is capable of prolonged service at elevated temperatures of about 260°C. Suitable commercial grades include the range of high temperature heat transfer fluids sold under the Trade Marks Essotherm and Teresso. An example of a suitable design of a heat exchanger using high temperature fluids is given in Figures 1 and 2.

Electrical heating can also provide the necessary heat required to gell latex and is more convenient to use. Electrical heating elements can be used to heat up two metal plates, held together by mechanical means, which plates constitute the heating medium. The rubber latex is passed through the cut-out sections of these

plates. A suitable design of a heat exchanger using electrical heat is given in Figure 3. The design provides for easy disassembly of the heat exchanger for cleaning and maintenance. Where electricity is expensive the heat exchanger can employ the high temperature heat transfer oil system.

Operation of the heat exchanger with the heating medium at temperatures around 100°C results in long gelling times and/or requires the use of high dosages of sensitizing chemicals, and is accordingly not preferred. Operation of the heat exchanger with the heating medium at higher temperatures, e.g. at least 200°C, results in shorter gelling times and requires the use of little or no sensitizing chemicals, but the gelled latex coagulum is liable to adhere to the surface of the heat exchanger. To avoid this danger, it is an important preferred feature of the invention that the surfaces of the heat exchanger in contact with the latex coagulum are smooth. This may be achieved by electro-chrome plating, polishing, honing or other means. The maximum permissible temperature of the heating medium of the heat exchanger is determined by the requirement that the coagulum does not char or stick at the surface of the heat exchanger; this will vary from one design to another but is likely in any case to be no more than 300°C. Heat transfer oils currently available are generally recommended for use at no more than 300°C.

A further advantage of this invention is that wet crumbs made from heat gelled coagulum dry faster than crumbs derived from acid coagulated coagulum. Thus, acid coagulated crumbs normally take about 5 hours to dry; heat gelled crumbs take about 20% shorter time.

In the accompanying drawings:—

Figure 1 is a longitudinal section through a heat exchanger using high temperature heating fluid.

Figure 2 is a cross-section on the line A—A of Figure 1.

Figure 3 is a cross-section view of a heat exchanger using electrical heat.

Referring to Figures 1 and 2, natural rubber latex is pumped through a pipe 10 into an inlet manifold 12 which is terminated by a baffle plate 14. The latex then passes through pipes 16, in which it is heated and gelled, and the coagulum emerges at truncated outlets 18 and is carried away on chute 20 for crumbling and drying. The pipes 16 have an internal diameter of from 6 to 25 mm. The surfaces of the pipe 10, manifold 12 and pipes 16 which contact the rubber latex are smoothed to reduce viscous drag and adhesion of the gelled coagulum by electrochrome plating, polishing, honing or other means.

Heat is supplied to the latex in the pipes 16 by means of a heating fluid surrounding the pipes 16 and retained by a cylindrical tube 22 and baffle plates 14 and 24. The heating fluid circulates via an inlet 26 and an outlet 28, and its flow along the tube 22 is interrupted by a series of baffle plates 30, of which only one is shown.

The heat exchanger shown in cross-section in Figure 3 includes two metal plates 32 and 34 bolted together. Each plate is heated by means of electrical heating elements 36, 38 maintained inside covers 40 and 42. The rubber latex is pumped through the tubes constituted by cut-out sections 44 of the plate 34 and is heated and coagulated in the same way as in the pipes 16 shown in Figures 1 and 2. The sections 44 are smoothed by electrochrome plating, polishing, honing or other means.

For both these designs, two aspects are important for their efficient performance. First, the latex pump must be capable of producing the necessary pressure to pump the gelled coagulum through the heat exchanger. The latex pump should not have high speed moving parts causing high shear in contact with the latex; otherwise the latex tends to be destabilised and clog up the pump. A pump using the positive displacement ram principle is suitable. Second, the surface of the metal in contact with the latex or gelled coagulum should preferably be smooth. This can be obtained by a highly polished or chromed surface or some other means. A smooth surface reduces adhesion between the gelled coagulum and the metal surface and facilitates easy passage of the coagulum; consequently the work demand on the latex pump is reduced.

The gelled coagulum produced by the above heat gelation machine is passed through a size reduction system such as a creper and a creper-hammermill or a granulator or an extruder before it is ready for drying in the dryers of block rubber factories.

Yet another method of achieving the continuous heat gelation of latex is the application of heat to an extruder. The latex is heat gelled in the extruder which

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simultaneously extrudes the coagulum. The extrudates are in a form suitable for drying in the dryers of block rubber factories used to produce these grades by the conventional acid coagulation process.

5 The heat gelled rubbers have nitrogen contents about 0.1% wt. and ash contents about 0.2% higher than the acid coagulated rubbers. The nitrogen content of heat gelled rubbers can be made equivalent to that of the acid coagulated rubbers by treating the latex before gelling with enzymes which are capable of removing proteins from the rubber particles. An example of such an enzyme is papain; this enzyme has the added advantage that it does not adversely affect the stability of well preserved latex. Treatment with papain also improves certain technological properties of the resultant rubber.

10 The following tests and examples serve to illustrate the various facets of this invention which have been previously discussed. All parts and percentages are by weight unless otherwise stated.

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Test 1.

This example demonstrates the predominant influence of the temperature of the heating medium on the time of gelation of latex. Latex ammoniated to high levels and stored for the normal period of time would gel as rapidly as lightly ammoniated latex.

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In this example, latex of about 30 percent dry rubber content and preserved with ammonia was contained in a standard stainless steel beaker (250 ml capacity and 1.6 mm wall thickness). 10 ml of the latex was gelled by placing the beaker on an oil bath maintained at 100°C, 150°C and 165°C. The results are given in Table 1.

TABLE 1.

Level of ammonia (% wt on latex)	Storage period of latex (hr)	Temperature of heating medium (°C)	Temperature of gelation (°C)	Time to gelation (sec)
0.05	3	100	76	67
0.05	3	150	76	51
0.05	3	165	79	41
0.15	24	100	78	116
0.15	24	150	76	110
0.15	24	165	77	54
0.03	1	165	75	75
0.07	10	165	74	54
0.15	18	165	77	70

Test 2.

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The time of gelation is not appreciably affected by the normal amounts of ammonia required to preserve latex for about 24 hours. It is also not appreciably dependent on the time of storage of the preserved latex unless the latex show signs of instability.

The experiment of Test 1 is repeated with the oil bath at 165°C and the latex preserved with 0.07% and 0.15% wt. on latex of ammonia. The results are shown in Table 2.

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TABLE 2.

Level of ammonia (% wt. on latex)	Storage period (hr)	Time to gelation (seconds)
0.07	5	84
0.07	9	80
0.07	12	70
0.15	9	96
0.15	12	85
0.15	18	70

Test 3.

The time of gelation is reduced by the use of heat-sensitising agents such as polypropylene glycol (PPG). The heat-sensitising agents can be added as the latex is received or just before gelation; the time of gelation is not affected significantly by the timing of this addition. The experiment of Test 1 is repeated with the oil bath at 165°C. The results are given in Table 3.

TABLE 3.

Amount of PPG added to latex (% wt. on dry rubber)	Level of ammonia (% wt. on latex)	Storage Period (hr)	Time to gelation (seconds)
0	0.05	1	80
0.25	0.05	1	62
0.50	0.05	1	56
0	0.20	24	55
0.25	0.20	24	47
0.50	0.20	24	46

Test 4.

A bulk sample of field latex has a dry rubber content of about 30%. During bad weather, the field latex may be diluted by rain water to as low as 20%. Dry rubber contents of field latex could be as high as 40% or artificially raised by light centrifugation of latex. The time of gelation is dependent on the dry rubber content of artificially diluted latex; the use of heat-sensitising agents can however reduce the time of gelation of the diluted latex to that of the undiluted latex. In practice, however, the use of heat-sensitising agents may not be necessary since diluted latex collected on rainy days is normally less stable than normal field latex.

The experiment in Test 1 is repeated with the water bath at 150°C. The results are as given in Table 4.

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TABLE 4.

Dry rubber content of latex (% dry rubber)	Level of PPG (% wt. on rubber)	Time to gelation (seconds)
40	0	70
30	0	74
25	0	112
20	0	115
25	0.3	76
20	0.5	80

Test 5.

Rubber samples were prepared from the same bulk of latex. The heat gelled rubbers with and without treatment by papain were compared with the acid coagulated control SMR 5L. The raw rubber properties of Nitrogen and Ash contents were determined. The results are given in Table 5.

TABLE 5.

Rubber	Papain % wt.	Nitrogen Content % wt.	Ash Content % wt.
Control SMR 5L	0	0.43	0.23
Heat gelled	0	0.51	0.46
Heat gelled	0.025	0.34	0.43
Heat gelled	0.050	0.26	0.40

Example 6.

A trial run was performed in the apparatus illustrated in Figures 1 and 2, except that only one tube 16 was used and the inside surface of the tube was not chromed. For this reason, a high concentration of heat-sensitizer was used to reduce adhesion between the tube surface and the gelled coagulum.

The starting material was a 30% dry rubber content field latex containing 0.1% by weight of ammonia, 2 phr of polypropylene glycol and 0.7 phr of a nonionic surfactant. This was passed through a tube 16 which was 19mm in diameter and 254 cm long, at a rate of 136.2 litres per hour, so that the residue time in the tube 16 was 19 seconds. The temperature of the heating medium was 200°C. The latex started at 30°C and gelled at 70–80°C.

WHAT WE CLAIM IS:—

1. A method of producing raw solid natural rubber in bulk form, which method comprises the steps of:—

(1) Passing a continuous stream of fresh or preserved natural rubber field latex through a heat exchanger in which the heating medium is maintained at a temperature from 100°C. to 300°C., so as to raise the temperature of the latex to from 60°C. to 110°C., whereby gelation of the rubber is caused to take place rapidly;



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(3) drying the coagulum and

(4) pressing and palleting the coagulum to form raw solid natural rubber in bulk.

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2. A method as claimed in claim 1, wherein the heating medium is maintained at a temperature of from 200°C to 300°C.

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3. A method as claimed in claim 1 or claim 2, wherein from 0.1% to 1%, by weight on the weight of the dry rubber, of a heat sensitizing agent is incorporated in the rubber latex.

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4. A method as claimed in claim 3, wherein the heat sensitizing agent is a polypropylene glycol having a molecular weight of from 500 to 1000.

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5. A method as claimed in any one of claims 1 to 4, wherein the rubber latex is passed through tubes having an internal diameter not greater than one inch contained in the heat exchanger.

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6. A method as claimed in any one of claims 1 to 4, wherein the rubber latex is passed through tubular cut-out sections of two metal plates held together mechanically and heated to the appropriate temperature.

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7. A method as claimed in any one of claims 1 to 4, wherein the rubber latex is heat gelled and extruded through a conventional extruder machine, heated to the appropriate temperature.

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8. A method as claimed in any one of claims 5 to 7, wherein the surfaces of the heat exchanger in contact with the rubber latex are made smooth.

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9. A method as claimed in any one of claims 1 to 8, wherein the rubber latex is treated before gelling with an enzyme to remove protein from the rubber particles.

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10. Rubber formed by gelling natural rubber latex according to the method claimed in any one of claims 1 to 9.

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