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PATENT SPECIFICATION

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COMPLETE SPECIFICATION

Apparatus for the preparation of Detergent Compositions

We, COLGATE-PALMOLIVE COMPANY, a Corporation organised and existing under the Laws of the State of Delaware, United States of America, of 300 Park Avenue, New York, New York 10022, United States of America, 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 an apparatus suitable for the preparation of detergent compositions having a low bulk density.

The term "low bulk density" as used herein refers to compositions having an apparent density of less than 0.45 grams per cubic centimetre. By "apparent density" is meant the weight per unit volume of the material indicated by the context. Thus, in the case of freely flowing particulate products, the term refers to the untamped weight per unit volume of the particulate material as it is charged into a container.

The present invention provides apparatus suitable for preparing a low bulk density detergent composition economically and in such a manner as to minimize the water content of the composition immediately prior to the drying step, thereby reducing the need for large quantities of drying heat and large drying apparatus. In addition, the apparatus allows control over the product properties, thereby facilitating consistent production of a uniform product.

The preparation of a detergent composition having a low bulk density may involve intimately mixing a component of the composition with another component, the relative proportions of these components being maintained substantially constant, and at least one of these components being a premix slurry having such a high solids content as to be difficultly flowable.

According to the invention, apparatus for this purpose comprises a first automatic con-

trol system for metering a continuous flow of the component having a high solids content to a point of addition of the other component, a second automatic control system for metering a continuous flow of the other component to the point of addition, means to mix the components intimately under conditions of high shear (as hereinafter defined), and means to remove water from the intimately mixed mixture, thereby forming an apparently dry detergent composition of low bulk density, as hereinbefore defined.

The apparatus is particularly useful for the preparation of detergent compositions of low bulk density by the process which is the subject of British patent application No. 51970/68 (Serial No. 1151767) which has been divided herefrom.

In one form of the invention use is made of metering units each employing two receptacles, for reasons discussed more fully below, whereby continuous weighing and control of the rates of flow of the components can be obtained. These units are especially commercially desirable in plants having a capacity in excess of 1500 pounds per hour since, in plants of that capacity, such units will enable more accurate and less costly control at high solids contents to be achieved. Each such unit comprises an intermittent receptacle and a continuous receptacle for the stream to be controlled. Series connections are maintained between the receptacles and the respective source of supply and the rest of the apparatus. Means are provided for continuously weighing the contents of the continuous receptacle and for continuously weighing the combined contents of both receptacles and translating this into control signals in terms of instantaneous rate of change of weight independent of the actual weight itself. The control system further includes means for providing a continuous predetermined rate of change command signal independent of the time reference and for comparing it with one or both of the fore-

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going instantaneous rate of change of weight signals for controlling discharge of the stream of materials from the continuous receptacle, thereby reducing to zero the differences between those signals being compared. The signals for each stream may also be compared, and the relative rates of feed controlled in relation thereto.

As indicated in the complete specification of the above-mentioned divided out application, certain materials should be added immediately prior to the high shear mixing step. This is especially important where a degradable or hydratable material such as sodium tripolyphosphate or sodium trimetaphosphate is used and reactions immediately begin to occur. For example, sodium tripolyphosphate is subject both to hydration and hydrolysis. When hydration occurs water is removed from the mixture. When working at high solids concentration, hydration causes the slurry to become even less fluid so that it cannot be flowed or adequately mixed. Other reactions such as hydrolysis cause degradation of the material, resulting in an undesirable final product. Accordingly, it is necessary to minimize both the hold-up time and the mixing time. It is therefore preferred that the premix slurry and the additive material remain in the feeders for as short a period of time as possible. The time after all of the components of the final product have been commingled and before the high shear mixing step is begun is suitably five seconds or less. The throughput time in the high shear mixer will advantageously be less than thirty seconds, and preferably between five and twenty seconds. The preferred control system has the ability accurately to combine a plurality of ingredients in a minimum of time whereby the relative proportions of ingredients can be adjusted in accordance with an instantaneously responsive control mechanism that measures the rate of feed of each of the component streams, immediately compensates for variations in rate change or component demand and is capable of carrying out all of these necessary functions with a total hold-up time of less than five minutes. Thus, the combination of an intermittent receptacle and a continuous receptacle in a manner described more fully hereinafter in combination with a high solids feed of detergent component streams results in a flexible system capable of producing a high quality product with a minimum of undesirable side products.

The invention may be performed in various ways, and a specific embodiment will now be described by way of example with reference to the accompanying drawing, which is a partially schematic flow diagram of this embodiment.

The means for measuring and controlling the weight rates of flow of the premix slurry and the materials added just before mixing com-

prise two units, a first unit 10 for the measurement and control of the premix slurry and a second unit 110 for the measurement and control of the additive materials. These units may be similar in apparatus and function. The first unit 10 comprises two vertically aligned material receiving receptacles 21 and 22 that are preferably about the same size and are mounted in co-operative relationship upon a stationary structural support 23.

The upper receptacle 21 is of any suitable shape and may, for example, be conical with an inlet conduit 24 arranged to supply premix slurry through the normally covered top of the receptacle 21. A feed control valve (not shown) is provided in the conduit 24. The lower end of the upper receptacle 21 is connected to discharge into a conduit 25 through a transfer valve 26. The conduit 25 enters the upper end of the lower receptacle 22. The discharge from the lower receptacle 22 is effected through a conduit 27 controlled by an adjustable orifice flow metering valve 28, or other suitable valve means. The conduit 25 positioned between the upper and lower receptacles 21, 22 permits relative movement between the upper and lower receptacles. This may be accomplished by the provision of a longitudinally slidable collar 29 or other suitable means such as by making the conduit 25 longitudinally flexible.

A horizontal mounting frame 31 is provided to support the upper receptacle 21 and is secured thereto by welding or bolting to brackets 37, 37'. The mounting frame 31 is supported by hangers 41, 41', which extend downwardly from support levers 42, 42' and are pivotally connected thereto inwardly of fulcrums 46, 46' by which the support levers 42, 42' are pivotally attached to depending members 48, 48' which extend downwardly from the structural support 23. At their innermost ends, the support levers 42, 42' are connected to loading members 53, 53' which at their uppermost ends are connected at 54 to a load cell 55 mounted on the upper part of the structural support 23. The load cell 55 may be, for example, an electric strain gauge having its output amplified.

Extensions 59, 59' of the hangers 41, 41' extend downwardly and support a horizontal frame member 61. A horizontal mounting frame 63 similar to the mounting frame 31 is provided for the lower receptacle 22 and is rigidly secured thereto as by welding or bolting to brackets 64, 64' on the receptacle 22. The mounting frame 63 is supported by hangers 65, 65' which extend downwardly from support levers 71, 71' and are pivotally connected thereto inwardly of fulcrums 73, 73' by which the support levers 71, 71' are pivotally attached to the hanger extensions 59, 59' which extend below the horizontal support 61 in the manner shown. At their innermost ends, the support levers 71, 71' are connected

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to loading members 75, 75' which at their upper ends are connected at 77 to a load cell 79 mounted on the support 61. Thus, the lower receptacle 22 is directly suspended from the upper receptacle 21 and the load cell 79 measures the weight of the lower receptacle 22 and its contents while the load cell 55 measures the combined weights of both receptacles and their contents.

The second unit 110 for the measurement and control of the rate of feed of the additive material is similar to the first unit 10 for the measurement and control of the premix slurry, and comprises two vertically aligned receptacles 121 and 122 mounted in co-operative relationship on the structural support 23. The upper receptacle 121 is provided with an inlet conduit 124, connected to supply additive material thereto. The feed to the receptacle 121 through a conduit 124 is controlled by a suitable valve (not shown). The upper receptacle 121 is connected through a conduit 125 to the lower receptacle 122, and the conduit 125 is provided with a transfer valve 126. The conduit 125 between the receptacles 121 and 122 is adapted to permit relative movement between these receptacles. This may be accomplished by provision of a slidable collar 129 or other suitable means. The discharge from the lower receptacle 122 is effected through a conduit 127 controlled by an adjustable metering valve 128.

A mounting frame 131 is provided to support the upper receptacle 121 and is secured thereto by a suitable means such as brackets 137, 137'. The frame 131 is supported by hangers 141, 141' which extend downwardly from support levers 142, 142'. The hangers 141, 141' are pivotally connected to the support levers 142, 142' inwardly of fulcrums 146, 146' by which the support levers 142, 142' are pivotally attached to depending members 148, 148' which are attached to and extend downwardly from the structural support 23. At their innermost ends, the support levers 142, 142' are connected to loading members 153, 153' which at their uppermost ends are connected at 154 to a load cell 155 mounted on the upper part of the support 23.

The hangers 141, 141' are extended below the frame 131 by extensions 159, 159' which support a horizontal frame member 161. A horizontal mounting frame 163 is provided for the lower receptacle 122 and is rigidly secured thereto as by brackets 164, 164'. The mounting frame 163 is supported by hangers 165, 165' which extend downwardly from support levers 171, 171' to which they are pivotally connected inwardly of fulcrums 173, 173' by which the support levers 171, 171' are pivotally connected to the extensions 159, 159'. At their innermost ends, the support levers 171, 171' are connected to loading members 175, 175' which at their upper ends are

connected at 177 to a load cell 179 mounted on the support member 161. Thus the lower receptacle 122 is suspended from the upper receptacle 121, whereby the load cell 179 measures the weight of lower receptacle 122 and its contents while the load cell 155 measures the combined weights of both receptacles and their contents.

For purposes of understanding the operation of the measuring and controlling apparatus, the upper receptacle 21 will be referred to as the first intermittent receptacle and the lower receptacle 22 will be referred to as the first continuous receptacle. Similarly, the upper receptacle 121 will be referred to as the second intermittent receptacle and the lower receptacle 122 will be referred to as the second continuous receptacle.

In operation, with suitable tare adjustments having been made and with the first and second continuous receptacles 22, 122 full and the first and second intermittent receptacles 21, 121 empty, the weights of the contents of the receptacles 22, 122 are continuously measured by the load cells 79, 179.

Discharge of material from the receptacles 22, 122 into a feeder 200 takes place continuously through the automatically adjustable flow metering valves 28, 128 and conduits 202, 204, at constant predetermined rates under control of the system as will appear hereinafter. The feeder 200 may comprise any suitable positive feed device. Advantageously, feeder 200 comprises an open-throat "progressing cavity" pump. Alternatively, the material from the receptacles 22 and 122 may be fed into separate positive feed devices, in which case the streams of material are combined just prior to charging of the material into the high shear mixing device to be described below.

Signals representing the weights of the contents of the first and second continuous receptacles 22, 122, as continuously measured by the load cells 79, 179, are fed into circuits 81, 181 that provide in leads 82, 182 electrical output signal potentials that are functions of the instantaneous rates of change of weight of the contents of the receptacles 22, 122 respectively. The value of these signals is thus independent of the magnitude of the weight of the contents of each of the continuous receptacles 22, 122. The control section of the system includes separately energized command signal generators 83, 183 that produce in leads 84, 184 electrical signal potentials which may be adjusted by setting members 85, 185 to produce signals corresponding to different desired rates of change of weight of the contents of the receptacles 22, 122. The setting members 85, 185 are calibrated to the desired rates of flow through the valves 28, 128 respectively. The measured signal potentials in the leads 82, 182 are compared with

the command signals in the leads 84, 184 respectively as by a potentiometer arrangement within self-balancing comparators designated 86, 186. The outputs of the comparators 86, 186 are connected by leads 80, 180 to servomotors 87, 187 respectively which adjust the sizes of the flow orifices of the valves 28, 128. Thus, depending upon the settings of the members 85, 185, the premix slurry discharged from the first continuous receptacle 22 and the additive materials discharging from the second continuous receptacle 122 discharge through conduits 27, 127 at controlled predetermined rates representing a controlled rate of change of weight of the contents of the first and second continuous receptacles 22, 122.

The automatic control for the system 10 for the premix slurry as operated during refilling of the first continuous receptacle operates in the following manner. At a predetermined time prior to complete discharge of all the contents from first continuous receptacle 22, the intermittent receptacle 21 is quickly filled with premix slurry by opening the supply valve (not shown) in the conduit 24, and then closing this valve to cut off further supply of premix slurry when the first intermittent receptacle 21 reaches a predetermined level. The first intermittent receptacle 21 will preferably be filled with enough material for one filling of the first continuous receptacle 22. During the filling of the first intermittent receptacle 21, as at all times throughout the operation of the process, the first continuous receptacle 22 continuously discharges material at a controlled rate. When the first intermittent receptacle 21 is filled, and the valve in the conduit 24 is closed, the weight of the combined contents of both receptacles 21 and 22 is continuously measured by the load cell 55 which feeds a corresponding signal into a circuit 91 thereby producing in a lead 92 an electrical output signal potential that is a function of the instantaneous rate of change of weight of the contents of the combined receptacles 21 and 22. This instantaneous change of weight of the combined receptacles is the same as the instantaneous rate of change of weight of the lower continuous receptacle 22 alone, because the continuous receptacle 22 continues to discharge from the system a certain number of weight units per unit of time at the same controlled rate as previously. Accordingly, the measured signal potential in the lead 92 is equal to that in the lead 82.

After the first intermittent receptacle 21 has been filled and before the first continuous receptacle 22 is empty, a switching device 93, 94 is automatically operated to substitute the measured signal potential in the lead 92 for that in the lead 82 at the comparator 86 so that the comparator is comparing the measured signal potential corresponding to the change

in weight of the combined receptacles with the command signal of the generator 83 to control the orifice size of the metering valve 28. The rate of discharge of the material after the switching device 93, 94 has automatically substituted the potential of the lead 92 for that of the lead 82 does not change, for the reasons set forth above. Next, the transfer valve 26 between the first intermittent receptacle 21 and the first continuous receptacle 22 is opened to discharge the contents of the first continuous receptacle 22 quickly after which the valve 26 is closed. This operation does not change the controlled rate of change of the weight of the premix slurry since control of the orifice size of the flow metering valve 28 and therefore the rate of discharge of the material is continuously maintained and controlled in relation to the change in weight of the first intermittent and continuous receptacles combined. After closing the transfer valve 26, the switching device 93, 94 is automatically actuated to substitute the measured signal potential in the lead 82 for that in the lead 92, whereby the load cell 79 again supplies the measured weight control. At this time, the first intermittent receptacle 21 can be refilled without affecting the weight rate control of discharge of the contents of the first continuous receptacle 22. The foregoing cycle of operation is repeated throughout the operation of the process.

The cycle of operation of the measuring and controlling system 110 for the additive material, comprising the second intermittent receptacle 121 and the second continuous receptacle 122, is the same as that described with reference to the system 10 for controlling the premix slurry, and will not be described in detail.

The control system 110 for the additive material is adjusted to such a feed rate as may be predetermined to be desirable in relation to the feed rate of the premix slurry.

In an alternative control system the control valve for the premix slurry is dispensed with, the control of this slurry being afforded by the feeder 200. In such a case the signal in the line 80 may be used to control the speed of a variable speed motor 206 which drives the shaft 208 of the worm 210 of feeder 200, the premix slurry thus being metered from the first continuous receptacle 22 by the screw feeder 200.

Since it is of importance closely to control the ratio between the premix slurry and the additive materials there is additionally provided a master controller 212 which compares the signals from the comparators 86 and 186, which signals represent the flow rate error of the premix slurry and of the additive material respectively. The master controller 212 is set to a predetermined desired ratio and adjusts either of the controllers 86, 186 or both to

maintain the rates of flow of premix slurry and additive material in accordance with the predetermined ratio.

Thus, two different streams are continuously supplied in accurately metered flow to a common point. The master controller 212 and the comparators 86, 186 can be reset manually to adjust the rates of feed and the ratios thereof relative to each other. Thus, the mixture proportions may be accurately set and maintained at all times and a correct formulation of the premix slurry and the additive material maintained even where demand or rate of flow of either material varies. The control system, because of its instantaneous response, facilitates the production of a high quality, uniform product.

The high solids content mixture of premix slurry and additive materials is passed substantially immediately, i.e. within five seconds, through a conduit 220 to a high shear mixer 222. A feed line 224 is positioned to inject a gaseous material into the mixture in the conduit 220 before the mixture enters the high shear mixer 222. The gaseous material is fed under pressure through the conduit 224 by a variable delivery compressor 226 the inlet of which is connected by a conduit 227 to a source of supply for the gaseous material. Where the gaseous material is air the compressor inlet may be open to the atmosphere. A discharge member 225 in the form of a short length of tubing is provided for injecting the gaseous material into the flowing stream in the conduit 220. The length of tubing is provided with two diametrically opposite pinholes, and the tubing adjacent the pinholes is surrounded by a flexible rubber sleeve which fits over the pinholes, in the manner of a bicycle tyre valve. As gaseous material under pressure is fed through the feed conduit 224 it forces the flexible sleeve away from the metal tubing, thereby allowing the gaseous material to discharge into the conduit 220. When the flow of gaseous material is terminated, the flexible sleeve covers the pinholes thereby eliminating any possibility of clogging thereof by back pressure from the mixture flowing in the conduit 220.

Another feed line 228 is provided for use when an additional component is to be added, such as the caustic alkali required when an alkali metal trimetaphosphate is the hydratable material. The conduit 228 is fed from a metered source (not shown) which may be of any suitable type. Where an alkali metal trimetaphosphate is to be added and the line 228 is arranged to supply a caustic alkali solution, the reaction between the hydratable material and the water present in the premix slurry is not initiated until the trimetaphosphate is brought into the presence of the caustic alkali. In such instance, the material does not form substantial amounts of hydrate

until the caustic alkali is added. Thus, all or a part of the trimetaphosphate may be incorporated into the premix slurry earlier than would otherwise be possible. This may be accomplished, for example, by discharging both receptacles 22 and 122 into the conduit 202 at the intake end of the feeder 200. The exact point or time of addition of the hydratable material to the premix slurry depends upon many variable and operating parameters, such as the rate at which hydration occurs, the temperatures involved, the solids content and the relative amounts of the compounds.

The mixer 222 for intimately and continuously mixing the components and evenly dispersing the gaseous material therein must impart a high shear to the mixture. Suitable mixers are generally relatively small in capacity so that at any particular moment only a small quantity of slurry is being worked upon, thereby allowing a major portion of the power input to be used directly on the slurry without becoming dissipated in an attempt to mix large quantities of material at one time. Thus, the mixer 222 will advantageously have a capacity of less than one gallon, whereby material can be passed through the mixer in less than thirty seconds while being subjected to extremely high shear for this short period of time. The mixer may be of any suitable high shear type and, for example, may comprise a mixing chamber 230 having two stators 232, 234 and a rotor 236 adapted to rotate between the stators. The internal faces of both of the stators and both faces of the rotor may be provided with concentric rows of blades 238 arranged so that the blades of the rotor 236 mesh closely but out of contact with the blades of the stators 232, 234. The slurry may advantageously pass from an inlet 240 at the centre of one stator, between the blades of that stator and the rotor, across the rotor, and between the blades of the other stator and the rotor, and then to an exit 242 at the far side of the mixer. In traversing this course through the mixer the slurry and the gaseous material are torn, stretched, and cut by the blades into numerous streams, and minute bubbles of the gaseous material are evenly distributed throughout the slurry. Other types of mixer may be used so long as the mixer is capable of imparting large quantities of energy and high shear rates to the slurry whereby a major proportion (i.e. more than 50 percent) of the gaseous material is broken into discrete bubbles having an average diameter of less than 0.0085 millimetre and preferably less than 0.054 millimetre.

The forces applied to the mixture vary with the equipment used and the operating parameters of the process. By "high shear" is meant a shearing action wherein there is a "Shear Factor (F)" as defined below in excess of 5 and preferably in excess of 6.5.

$$\text{Shear Factor (F)} = \frac{2 \times 10^{-6} r RT}{dC}$$

wherein:

- r = the radius in inches of the mixer rotor or its equivalent;
 R = the number of revolutions per minute of the mixer rotor or its equivalent;
 T = the rate of feed in pounds per hour of the material to be sheared.
 d = the clearance in inches between the blades on the rotor and those on the stator or their equivalents;
 and
 C = the volumetric capacity of the mixer in cubic inches.

A tube 244 is provided at the outlet 242 of the mixer 222 for transfer of the intimately mixed material from the mixer on to a conveyor 250. The tube 244 also serves to adjust the internal pressure in the mixer 222 and for this purpose may be varied in length and diameter. For example, by increasing the length or decreasing the diameter of the tube 244, the pressure within the mixer 222 can be increased. It is desirable to use a pressure-controlling member of this type instead of a throttle valve, to avoid a sudden drop in pressure which may cause instability of the product. The length and diameter of the tube may be selected to maintain the mixer pressure within the range 20 to 100 pounds per square inch gauge, preferably within the range 40 to 65 pounds per square inch gauge. Advantageously, the length of the tube is at least ten times its internal diameter.

The conveyor 250 may comprise a continuously moving belt whereby the product is rapidly removed, thereby eliminating piling up of product to an undesirable height which might tend to break down the structure of the product, causing the final bulk density to be higher than desired. It may in some cases be desirable to maintain the temperature of the product on the conveyor 250 at a predetermined level for a predetermined time, in which instance the conveyor 250 may be provided with means 252 to heat and/or cool it. The means 252 is shown as a chamber; however, equivalent equipment such as a thermostatically controlled electrical resistance heater may be used. Means may also be provided, if

necessary, for breaking the product into particles or for breaking apart lumps of product in the event that such are produced. The product is discharged from the conveyor 250 onto a transfer conveyor 254 which feeds the product into a dryer 256 which, in the embodiment shown, is a rotary kiln or tumbling drum dryer; but which may be of any other suitable form. Thus, the dryer 256 may comprise a fluidized bed drying chamber, a continuous belt which passes through a heating tunnel, a spray drying tower or a vertical shaft heating chamber. For reasons of economy, however, it is preferred that a tumbling drum type of dryer of the type illustrated be used. Especially economical drying is made possible by virtue of the low free moisture content of the product. The final product issuing from the dryer 256 is passed to suitable removal means such as a conveyor 258 which delivers the material to storage, packaging, or other desired location. Perfume may be added to the product at any suitable time, as at the end of the drying step.

The following examples illustrate the invention. Unless otherwise indicated, all parts, proportions and percentages in the examples and elsewhere are by weight, and all inorganic salts are anhydrous.

EXAMPLE I

An open throat "progressing cavity" feeder 200 was continuously fed with two streams of material. One stream consisted of a slurry made from the following materials:

	Parts by weight
Water	15.6
50° Bé caustic soda solution	11.2
Sodium toluene sulphonate (commercial grade)	3.0
Tridecylbenzene sulphonic acid (96% sulphonic acid, 2% sulphuric acid, 1% free oil, 1% moisture)	35.2
Sodium sulphate	13.7
Sodium carboxymethyl cellulose (74% active ingredient, 26% inert solids)	1.6
Sodium silicate (44.1% solids having an $\text{Na}_2\text{O}:\text{SiO}_2$ ratio of 1:2.0)	19.7

The slurry constituting the first stream was maintained at a temperature of about 57°C. and was delivered to the progressing cavity feeder 200 from the receptacle 22 of the metering device 10 at the rate of 304 pounds per hour. The second stream consisted of Form II powdered pentasodium tripolyphosphate delivered from the receptacle 122 of the metering device 110 at a rate of 161 pounds per hour. Form II pentasodium tripolyphosphate is a phosphate which during manufacture is calcined at a low temperature as compared to Form I which is calcined at a higher temperature. Form I pentasodium tripolyphosphate hydrates more rapidly than Form II thereof. The Form II pentasodium tripolyphosphate used in this example was in the form of particles of which 86% thereof were less than 75 microns in diameter, and 11% had a diameter between 75 and 150 microns.

The mixture of powder and slurry was subjected to some preliminary mixing before discharge from the feeder 200, the material discharged from feeder 200 was a mixture of powder and slurry in a substantially constant ratio. The solids content of the material at this point was 77%.

The speed of the progressing cavity feeder was set to maintain a constant, low level in its feed hopper. The feed was pumped through the conduit 220 directly to a continuous high shear mixer 222 of the type shown and des-

cribed herein. The hold-up time in the conduit 220 was about thirty seconds. Immediately upstream of the mixer a metered stream of compressed air was introduced, and the mixture entered the mixer 222 within five seconds thereof. This air amounted to about 4.0 standard cubic feet of air per hour. The mixer was operated at a rotor speed of 400 RPM. A 14-inch length of $\frac{1}{2}$ inch inside diameter tubing on the mixer discharge maintained an operating pressure of about 60 pounds per square inch gauge at the inlet of the mixer, and provided for a gradual diminution of the pressure on the material being processed.

The material discharged from the high shear mixer was a uniformly mixed and aerated paste with a density of about 0.44 grams per cubic centimetre at a temperature of about 65°C. It was fed into a series of tubs, where it was allowed to remain in a quiescent state at room temperature for about 48 hours. During this period it solidified. The product was removed from the tubs and passed through a rotating cage mill to reduce all lumps to small particles and was then screened through a sieve having square apertures each of 2 millimetres a side. The resulting powder had an apparent density of 0.38 grams per cubic centimetre and a moisture content of about 22%, and was dried to 18% total moisture in a moving current of warm (60°C.) air. This product had a composition approximately as follows:

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	Percent
Tridecylbenzene sodium sulphonate	25
Pentasodium tripolyphosphate	37
Sodium toluene sulphonate	2
Sodium silicate	6
Sodium carboxymethyl cellulose	0.8
Sodium sulphate	11
Water (essentially all molecularly bound by the phosphate and other hydratable salts)	18
Other matter	0.2

EXAMPLES II—VIII

5 A series of experiments was carried out under varying operating conditions and with a composition having the same formulation of

starting materials as that described in Example I, except as otherwise indicated. The operating conditions are set forth in Table I below:

TABLE I

Ex.	Temperature of Premix Slurry (1)	Feed Rate (2)	Solids Content (percent) (3)	Air (Standard cubic feet per hour) (4)	Rotor Speed of Mixer (RPM) (5)	Pressure (6)	Temp. (7)	Prod. Dens. (8)	Prod.* Mois. (per cent)	Shear Factor	STS (9)
II.	125	360	75	3.1	400	35	135	0.37	23.7	7.6	2
III.	120	360	77	0.9	400	40	150	0.34	15.8	7.7	6
IV.	125	350	75	0.5	400	29	145	0.306	22.4	7.4	6
V.	160	350	75	6.3	400	39	152	0.34	22.8	7.5	2
VI.	135	459	75.5	4.6	400	42	137	0.367	26.0	9.8	2
VII.	125	348	76	4.0	400	53	140	0.371	23.6	7.4	2
VIII.	135	355	75.5	3.4	400	46	135	0.362	23.0	7.5	2
Run A	120	200	75	6.6	375	35	140			4.0	2
Run B	135	366	80.6	3.4	400	40	145			7.8	2

(1) This temperature was measured in degrees Fahrenheit at hopper 202.

(2) The feed rate was calculated in pounds per hour.

(3) The solids content was calculated as including all components and without any hydration.

(4) Standard cubic feet was calculated at a pressure of 760 millimetres mercury and a temperature of 0 degrees Centigrade.

(5) The rotor had a diameter of 8 inches. The mixer had a capacity of 79 cubic inches, and the clearance between the blades of rotor and stator was 0.060 inch.

(6) The pressure was measured in pounds per square inch gauge at the inlet of the high shear mixer.

(7) This temperature was measured in degrees Fahrenheit at the inlet of the high shear mixer.

(8) The product density was measured in grams per cubic centimetre.

(9) The amount of sodium toluene sulphonate in the final product expressed as percent by weight.

*Product Moisture

In Examples IV and V, Form I pentasodium tripolyphosphate was substituted for the Form II pentasodium tripolyphosphate used in most of the other example. The Form I compound hydrates more rapidly than Form II pentasodium tripolyphosphate. A comparison of the product of Example IV with that of the otherwise similar Example III in which Form II pentasodium tripolyphosphate was used showed the improvement possible through use of a more rapidly hydratable material to impart structural strength to the product as it leaves the high shear mixing device.

As a comparison to the examples of the invention set forth above, Run A was performed wherein the entire formulation was made up in a batching device and the hydrat-

able material, Form II pentasodium tripolyphosphate, was included in this batch instead of being added later in the processing as in the above examples. The completely formulated material was fed by the feeder 200 to the high shear mixer 222. During the course of a half hour run, the mix became too thick to pump due to substantial hydration of the hydratable material.

Run B illustrates another comparison run wherein insufficient (only 4%) hydratable material was used. The formulation differed from that used for Examples I—VIII. In Run B, the feeder 200 was continuously fed with a first stream comprising a slurry made from the following materials:

	Percent
Water	27.77
50° Bé caustic soda solution	12.60
Sodium toluene sulphonate (powdered commercial grade)	3.41
Tridecylbenzene sulphonic acid	49.67
Form II Pentasodium tripolyphosphate	6.55

The slurry constituting the first stream was maintained at a temperature of about 155°F. and was delivered to the feeder 200 at the rate of 188 parts by weight per hour. A second stream comprising 178 parts by weight per hour of anhydrous sodium sulphate as filler was delivered to the feeder 200 and therein combined with the initial slurry.

The feed was pumped to a high shear mixer under approximately the same conditions as those described in Example I and about 3.4 standard cubic feet per hour of air was introduced. A pressure of about 40 pounds per square inch gauge was maintained at the inlet to the mixer. The material discharged from the mixer was a wet paste and failed to form an acceptable product even though

a lesser amount of water was used than in the examples set forth above. The cause of this is believed to be lack of sufficient hydratable material to bind the water, thereby providing inadequate structural strength. In addition, it was difficult to feed the slurry due to the initial incorporation in the slurry of the hydratable material.

EXAMPLES IX—XV

In this series of examples, the hydratable material was sodium trimetaphosphate and the examples were carried out in the following manner:

The feeder 200 was continuously fed with two metered streams of material. A first stream comprised a slurry of the following:

		Parts by weight
20	Sodium toluene sulphonate	3.1
	Sodium tridecylbenzene sulphonate (54% solids, 46% water; 86% active ingredient, 14% inert ingredient)	31.0
25	Sodium silicate solution (43.5% solids, 56.5% water)	14.0
	Kettle soap (sodium salt of a mixture of fatty acids derived from tallow and coconut oils in the weight ratio of 80:20 respectively)	7.0
30	Sodium carboxymethyl cellulose (74% active ingredient, 26% inert ingredient)	0.8
	Powdered polyvinyl alcohol	0.3
	Ultra-marine blue	0.3
	Sodium sulphate powder	43.5

5 The premix slurry constituting the first stream was maintained at a temperature of about 150°F. and was delivered to the feeder 200 at a rate of 250 pounds per hour. The solids content of the premix slurry for Examples IX—XV varied between 73.5% and 79.0%. A second stream comprising powdered sodium trimetaphosphate having an average particle size less than 75 microns was metered to the feeder 200 at the rate of 154 pounds per hour. The feeder 200 pumped the mixture directly to a high shear mixer 222. Immediately upstream of the mixer 222, a 50° Bé caustic soda solution was metered into the conduit 220 at the rate of 60 pounds per hour and air was metered into the conduit at the rate shown in Table II. The product resulting from each of these examples was a low bulk density detergent composition of uniform consistency having density and moisture contents as shown in Table II. These products could be further dried in any suitable manner to an even lower moisture content.

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The product densities shown in Table II illustrate an advantage of the present invention in that it permits the preparation of a product having a bulk density lower than 0.35 even though the processing conditions included the use of a solids content higher than 80%, and also notwithstanding the fact that the final product contained less than 35% by weight of tripolyphosphate, which is a relatively low tripolyphosphate content for low bulk density detergent compositions. Illustrative of the low tripolyphosphate contents obtained are Examples IX and XI which had tripolyphosphate contents of 27.8 and 27.3 respectively.

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TABLE II

Ex.	Temperature of Premix Slurry (1)	Feed Rate (2)	Solids Content percent (3)	Air (Standard cubic feet per hour) (4)	Rotor Speed of Mixer (RPM) (5)	Pressure (6)	Temp. (7)	Prod. Dens. (8)	Prod.* Mois. (per-cent) (9)	Shear Factor (9)	STS (9)
IX.	150	464	83.5	0.5	800	21	190	0.15	15.0	19.6	2.0
X.	150	464	83.5	2.2	800	23	190	0.18	15.2	19.6	2.0
XI.	150	464	83.5	0.5	800	25	190	0.38	16.0	19.6	0.0
XII.	150	464	80.4	2.2	800	21	190	0.33	17.0	19.6	0.0
XIII.	190	464	80.0	2.2	400	23	190	0.27	18.5	9.8	0.0
XIV.	190	464	80.8	0.5	800	20	190	0.28	18.1	19.6	0.0
XV.	190	464	80.8	0.5	400	23	190	0.18	17.0	9.8	0.25

(1) This temperature was measured in degrees Fahrenheit at hopper 202.

(2) The feed rate was calculated in pounds per hour.

(3) The solids content was calculated as including all components and without any hydration.

(4) Standard cubic feet was calculated at a pressure of 760 millimetres mercury and a temperature of 0 degrees Centigrade.

(5) The rotor had a diameter of 8 inches. The mixer had a capacity of 79 cubic inches, and the clearance between the blades of rotor and stator was 0.060 inch.

(6) The pressure was measured in pounds per square inch gauge at the inlet of the high shear mixer.

(7) This temperature was measured in degrees Fahrenheit at the outlet of the high shear mixer.

(8) The product density was measured in grams per cubic centimetre.

(9) The amount of sodium toluene sulphonate in the final product expressed as percent by weight.

*Product Moisture.

WHAT WE CLAIM IS:—

1. Apparatus for the production of a detergent composition wherein a component of the composition is intimately mixed with another component, the relative proportions of these components are maintained substantially constant, and at least one of these components is a premix slurry having such a high solids content as to be difficultly flowable, which apparatus comprises a first automatic control system for metering a continuous flow of the component having a high solids content to a point of addition of the other component, a second automatic control system for metering a continuous flow of the other component to the point of addition, means to mix the components intimately under conditions of high shear (as hereinbefore defined), and means to remove water from the intimately mixed mixture, thereby forming an apparently dry detergent composition of low bulk density, as hereinbefore defined.
2. Apparatus as claimed in claim 1 in which one or each of the automatic control systems comprises a support, a first weighing unit mounted on the support, an intermittent receptacle suspended from the first weighing unit, means for intermittently supplying the respective component to the intermittent receptacle, a second weighing unit carried by the intermittent receptacle, a continuous receptacle suspended from the second weighing unit, a passage including a transfer valve between the receptacles, metering means providing controlled continuous discharge of the component from the continuous receptacle, and means for cyclically connecting the first and second measuring units to a control unit which controls the metering means continuously, means to compare the rates of flow of the components as measured by the first and second automatic control systems, and means to adjust these rates of flow relative to each other to conform to a predetermined ratio therebetween.
3. Apparatus as claimed in claim 1 or claim 2 wherein the means to remove water comprises a rotary kiln.
4. Apparatus as claimed in claim 1 or claim 2 wherein the means to remove water comprises a spray drying tower.
5. Apparatus as claimed in claim 1 or claim 2 wherein the means to remove water comprises a fluidized bed drying chamber.
6. Apparatus as claimed in any of the preceding claims wherein the mixing means comprises a casing, a rotor within the casing carrying a plurality of blades extending towards the casing, and a plurality of stator blades in the casing extending towards the rotor, the rotor blades being arranged, upon rotation of the rotor, to pass closely between the stator blades.
7. Apparatus as claimed in any of the preceding claims which also includes means to inject a normally gaseous material either into or upstream of the mixing means.
8. Apparatus for the production of a detergent composition of low bulk density, as hereinbefore defined, substantially as described with reference to the accompanying drawing.

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ERRATUM

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Page 5, line 120, for "0.0085" read "0.085"

THE PATENT OFFICE
17th June 1969

