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(54) Titre : PROCEDE DE FABRICATION D'UN DETERGENT HAUTE DENSITE
(54) Title: PROCESS FOR MAKING HIGH DENSITY DETERGENT

(57) **Abrégé/Abstract:**

A process for producing high density detergent compositions is provided. The process is a continuous process during which high density detergent agglomerates are produced by feeding a surfactant paste and dry starting detergent material into a mixer/densifier having a centrally positioned rotating shaft which experiences minimal vibration via the use of tuned dampers mounted on the shaft. The process produces a free flowing, high density detergent composition which can be commercially sold as a low dosage or "compact" detergent composition.

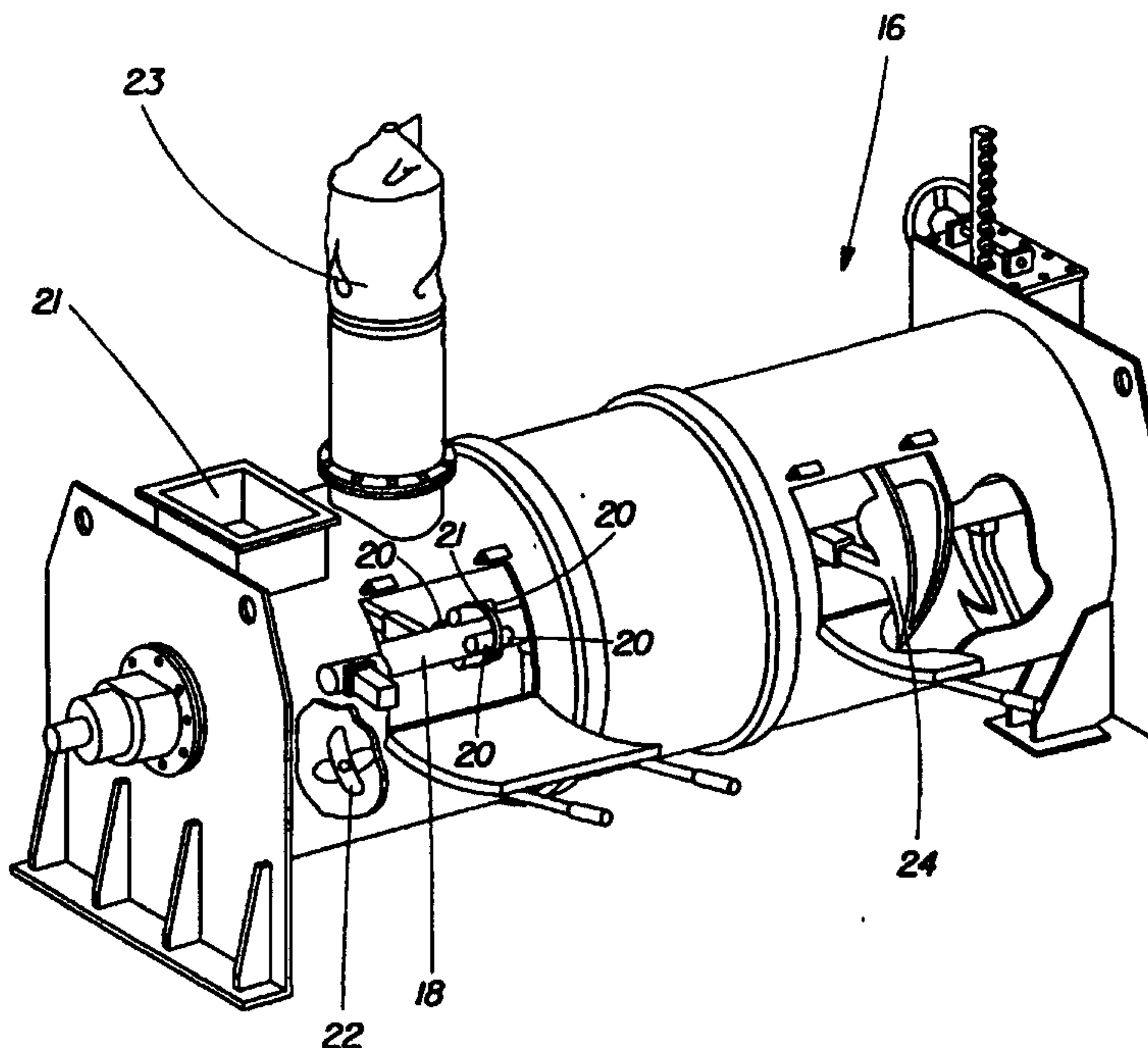


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(21) International Application Number: PCT/US97/12948 (22) International Filing Date: 30 July 1997 (30.07.97) (30) Priority Data: 60/023,902 14 August 1996 (14.08.96) US (71) Applicant (for all designated States except US): THE PROCTER & GAMBLE COMPANY [US/US]; One Procter & Gamble Plaza, Cincinnati, OH 45202 (US). (72) Inventors; and (75) Inventors/Applicants (for US only): ZAFFIRO, Paul, Anthony [US/US]; 6285 Delcrest Drive, Fairfield, OH 45014 (US). HALVORSEN, William, Garrett [US/US]; 1311 Dillon Avenue, Cincinnati, OH 45208 (US). CAPECI, Scott, William [US/US]; 3285 Citation Lane, North Bend, OH 45052 (US). WILLIAMS, Antonio [US/US]; 2011 Crest Road, Cincinnati, OH 45240 (US). DALTON, Larry, Vincent [US/US]; 8682 Hollyhock Drive, Finneytown, OH 45231 (US). (74) Agents: REED, T., David et al.; The Procter & Gamble Company, 5299 Spring Grove Avenue, Cincinnati, OH 45217 (US).		(81) Designated States: BR, CA, CN, JP, MX, US, European patent (AT, BE, CH, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE). Published <i>With international search report. Before the expiration of the time limit for amending the claims and to be republished in the event of the receipt of amendments.</i>

(54) Title: PROCESS FOR MAKING HIGH DENSITY DETERGENT



(57) Abstract

A process for producing high density detergent compositions is provided. The process is a continuous process during which high density detergent agglomerates are produced by feeding a surfactant paste and dry starting detergent material into a mixer/densifier having a centrally positioned rotating shaft which experiences minimal vibration via the use of tuned dampers mounted on the shaft. The process produces a free flowing, high density detergent composition which can be commercially sold as a low dosage or "compact" detergent composition.

PROCESS FOR MAKING HIGH DENSITY DETERGENT

FIELD OF THE INVENTION

The present invention generally relates to a process for producing high density detergent compositions. More particularly, the invention is directed to a continuous process during which high density detergent agglomerates are produced by feeding a surfactant paste and dry starting detergent material into a mixer/densifier having a centrally positioned rotating shaft with minimal vibration. The process produces a free flowing, high density detergent composition which can be commercially sold as a low dosage or "compact" detergent composition.

BACKGROUND OF THE INVENTION

Recently, there has been considerable interest within the detergent industry for laundry detergents which are "compact" and therefore, have low dosage volumes. To facilitate production of these so-called low dosage detergents, many attempts have been made to produce high bulk density detergents, for example with a density of 600 g/l or higher. The low dosage detergents are currently in high demand as they conserve resources and can be sold in small packages which are more convenient for consumers.

Generally, there are two primary types of processes by which detergent granules or powders can be prepared. The first type of process involves spray-drying an aqueous detergent slurry in a spray-drying tower to produce highly porous detergent granules. In the second type of process, the various detergent components are dry mixed after which they are agglomerated with a binder such as a nonionic or anionic surfactant. In both processes, the most important factors which govern the density of the resulting detergent granules are the density, porosity and surface area of the various starting materials and their respective chemical composition. These parameters, however, can only be varied within a limited range. Thus, a substantial bulk density increase can only be achieved by additional processing steps which lead to densification of the detergent granules.

There have been many attempts in the art for providing processes which increase the density of detergent granules or powders. Particular attention has been given to densification of spray-dried granules by post tower treatment. For example, one attempt involves a batch process in which spray-dried or granulated detergent powders containing sodium tripolyphosphate and sodium sulfate are densified and spheronized in a Marumerizer®. This apparatus comprises a substantially horizontal, roughened, rotatable table positioned within and at the base of a substantially vertical, smooth walled cylinder. This process, however, is essentially a batch process and is therefore less suitable for the large scale production of detergent powders. More recently, other attempts have been made to provide a continuous

processes for increasing the density of "post-tower" or spray dried detergent granules. Typically, such processes require a first apparatus which pulverizes or grinds the granules and a second apparatus which increases the density of the pulverized granules by agglomeration. These processes achieve the desired increase in density by treating or densifying "post tower" or spray dried granules.

All of the aforementioned processes are directed primarily to densifying or otherwise processing spray dried granules. Currently, the relative amounts and types of materials subjected to spray drying processes in the production of detergent granules has been limited. For example, it has been difficult to attain high levels of surfactant in the resulting detergent composition, a feature which facilitates production of low dosage detergents. Thus, it would be desirable to have a process by which detergent compositions can be produced without having the limitations imposed by conventional spray drying techniques. To that end, the art is also replete with disclosures of processes which entail agglomerating detergent compositions. For example, attempts have been made to agglomerate detergent builders by mixing zeolite and/or layered silicates in a mixer to form free flowing agglomerates.

However, in all of the aforementioned processes, continuous large-scale production tends to have its difficulties, especially relative to obtaining acceptable product consistently and with minimal wear and tear on the manufacturing equipment. For instance, while certain mixer/densifiers work extremely well at the lab scale or even at the pilot plant scale, their performance is not always reproducible in large-scale commercial continuous manufacturing facilities.

One problem that has arisen involves excessive vibration of the rotating shafts in commercial scale mixer/densifiers which can have deleterious effects on the detergent composition produced as well as on the mixer/densifiers and other closely located manufacturing equipment. This problem can also lead to structural damage to the manufacturing building for which substantial expenditures may be required for repair. Thus, there remains a need for a means by which commercial scale mixer/densifiers used to produce low dosage, high density detergent compositions can be operated continuously without significant mechanical vibration and damage resulting therefrom.

Accordingly, there remains a need in the art to have a process for continuously producing a high density detergent composition that involves a mixer/densifier with minimal mechanical vibration. Also, there remains a need for such a process which is more efficient and economical to facilitate large-scale production of low dosage or compact detergents.

BACKGROUND ART

The following references are directed to densifying spray-dried granules: Appel et al, U.S. Patent No. 5,133,924 (Lever); Bortolotti et al, U.S. Patent No. 5,160,657 (Lever); Johnson

et al, British Patent No. 1,517,713 (Unilever); and Curtis, European Patent Application 451,894. The following references are directed to producing detergents by agglomeration: Capeci et al, U.S. Patent 5,366,652; Capeci et al, U.S. Patent 5,486,303; Capeci et al, U.S. Patent 5,489,392; Capeci et al, U. S. Patent 5,516,448; Beerse et al, U.S. Patent No. 5,108,646 (Procter & Gamble); Hollingsworth et al, European Patent Application 351,937 (Unilever); and Swatling et al, U.S. Patent No. 5,205,958.

SUMMARY OF THE INVENTION

The present invention meets the aforementioned needs in the art by providing a process which continuously produces a high density detergent composition via a process during which high density detergent agglomerates are produced by feeding a surfactant paste and dry starting detergent material into a mixer/densifier having a centrally positioned rotating shaft with minimal vibration. In the process, tuned dampers have been mounted on the centrally rotating shaft in the mixer/densifier unexpectedly resulting in a process which produces a high density detergent composition with minimal mechanical vibration and repair expenses normally associated such vibration. The process produces a free flowing, high density detergent composition which can be commercially sold as a low dosage or "compact" detergent composition.

As used herein, the term "agglomerates" refers to particles formed by agglomerating starting detergent ingredients which typically have a smaller mean particle size than the formed agglomerates. All percentages and ratios used herein are expressed as percentages by weight (anhydrous basis) unless otherwise indicated. All viscosities referenced herein are measured at 70°C ($\pm 5^\circ\text{C}$) and at shear rates of about 10 to 100 sec^{-1} .

In accordance with one aspect of the invention, a process for preparing a crisp, free flowing, high density detergent composition is provided. The process comprises the steps of (a) continuously mixing a detergent surfactant paste having a viscosity of from about 5,000 cps to about 100,000 cps and dry starting detergent material comprising an aluminosilicate builder into a high speed mixer/densifier to obtain detergent agglomerates; (b) mixing the detergent agglomerates in a moderate speed mixer/densifier to further densify and agglomerate the detergent agglomerates, wherein the moderate speed mixer/densifier includes a centrally rotating shaft on which a tuned damper apparatus is mounted such that the peak vibration of the shaft is from about -1.0G to about 1.0G in a frequency range of from about 10 Hz to about 20 Hz at the mid-span length of the shaft; and (c) drying the detergent agglomerates so as to form the high density detergent composition.

In accordance with another aspect of the invention, another continuous process for producing a high density detergent composition is provided. The process comprises the steps of: (a) continuously mixing spray-dried detergent granules into a moderate speed

mixer/densifier, wherein the speed mixer/densifier includes a centrally rotating shaft on which a tuned damper apparatus is mounted such that the peak vibration of the shaft is from about -1.0G to about 1.0G in a frequency range of from about 10 Hz to about 20 Hz at the mid-span length of the shaft; and (b) drying the detergent agglomerates so as to form the detergent composition. Additionally, the detergent compositions made by any of the processes described herein is also provided by the invention.

Accordingly, it is an object of the invention to provide a process for continuously producing a high density detergent composition that involves a mixer/densifier having minimal mechanical vibration. Also, it is an object of the invention to provide such a process which is more efficient and economical to facilitate large-scale production of low dosage or compact detergents. These and other objects, features and attendant advantages of the present invention will become apparent to those skilled in the art from a reading of the following detailed description of the preferred embodiments, drawings and the appended claims.

BRIEF DESCRIPTION OF THE DRAWINGS

Fig. 1 is a flow chart illustrating a preferred process in which two mixer/densifiers, a fluid bed dryer, a fluid bed cooler and screening apparatus are serially positioned in accordance with a highly preferred embodiment of the invention;

Fig. 2 is a perspective partial cut-away view of a mixer/densifier used in accordance with the process which illustrates the tuned damper mounted on the centrally rotating shaft;

Fig. 3 is a side cross-sectional view of the centrally rotating shaft having a tuned damper mounted thereon; and

Fig. 4 is an end view of the rotating shaft in the moderate speed mixer/densifier illustrating the tuned damper with four damping apparatus mounted about the shaft via connecting bars and clamps.

DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENT

Process

The present process can be used in the production of low dosage, high density detergent compositions containing agglomerates produced directly from starting detergent ingredients as well as compositions containing conventional spray-dried granules that have undergone "post-tower" densification. By "spray-dried granules", we mean porous granules that have been made by drying an aqueous slurry of detergent ingredients including surfactant, builder and other standard adjunct detergent ingredients. Typically, the drying is completed by spraying the aqueous slurry into a tower having a counter current hot air stream. By "post-tower" densification, we mean those detergent granules which have been processed through a conventional spray-drying tower or similar apparatus, and thereafter, through mixer/densifiers as described herein.

Reference is now made to Fig. 1 which presents a flow chart illustrating the instant process and various embodiments thereof. In the first step of the process, the invention entails continuously mixing into a high speed mixer/densifier 10 several streams of starting detergent ingredients including a surfactant paste stream 12 and a dry starting detergent material stream 14. The surfactant paste 12 preferably comprises from about 25% to about 65%, preferably from about 35% to about 55% and, most preferably from about 38% to about 44%, of a detergent surfactant in an aqueous paste form. Preferably, the dry starting detergent material 14 comprises from about 20% to about 50%, preferably from about 25% to about 45% and, most preferably from about 30% to about 40% of an aluminosilicate or zeolite builder, and from about 10% to about 40%, preferably from about 15% to about 30% and, most preferably from about 15% to about 25% of a sodium carbonate. The surfactant paste 12 and the dry starting detergent material 14 are continuously mixed within the ratio ranges described herein so as to insure production of the desired free flowing, crisp, high density detergent composition. Preferably, the ratio of the surfactant paste 12 to the dry starting detergent material 14 is from about 1:10 to about 10:1, more preferably from about 1:4 to about 4:1 and, most preferably from about 2:1 to about 2:3.

It should be understood that additional starting detergent ingredients several of which are described hereinafter may be mixed into high speed mixer/densifier 10 without departing from the scope of the invention. It has been found that the first processing step can be successfully completed, under the process parameters described herein, in a high speed mixer/densifier 10 which preferably is a Lödige CBTM mixer or similar brand mixer. These types of mixers essentially consist of a horizontal, hollow static cylinder having a centrally mounted rotating shaft around which several plough-shaped blades are attached. Preferably, the shaft rotates at a speed of from about 100 rpm to about 2500 rpm, more preferably from about 300 rpm to about 1600 rpm. Preferably, the mean residence time of the detergent ingredients in the high speed mixer/densifier 10 is preferably in range from about 2 seconds to about 45 seconds, and most preferably from about 5 seconds to about 15 seconds.

The resulting detergent agglomerates formed in the high speed mixer/densifier 10 are then fed into a lower or moderate speed mixer/densifier 16 during which further build-up agglomeration is carried forth. In the alternative embodiment, a single stream of spray-dried granules (not shown) is inputted directly into the moderate speed mixer/densifier, wherein the high speed mixer/densifier 10 is strictly optional for this particular embodiment of the invention. The moderate speed mixer/densifier 16 used in the present process should include liquid distribution and agglomeration tools so that both techniques can be carried forth simultaneously. It is preferable to have the moderate speed mixer/densifier 16 to be, for example, a Lödige KM (Ploughshare) mixer, Drais® K-T 160 mixer or similar brand mixer. The residence time in the

moderate speed mixer/densifier 16 is preferably from about 0.5 minutes to about 15 minutes, most preferably the residence time is about 1 to about 10 minutes.

Reference is now made to Fig. 2 in which a perspective partial cut-away view of moderate speed mixer/densifier 16 is depicted. As can be seen from Fig. 2, a centrally located shaft 18 rotates inside the moderate speed mixer/densifier 16 (e.g. Lödige KM (Ploughshare) mixer). In accordance with the invention, at least one tuned damper 20 is mounted on the rotating shaft 18. Also, the moderate speed mixer/densifier 16 accomplishes liquid distribution via cutters 22 mounted on the inside wall of the mixer/densifier 16 and are generally smaller in size than the rotating shaft 18. The cutters typically operate at least about 3600 rpm. The moderate speed mixer/densifier 16 also has ploughs 24 that are mounted to the centrally rotating shaft 18 to aid in the mixing/densifying operations in the process and a pair of inlet ports 21 and 23 that are capable of receiving detergent material from the high speed mixer/densifier 10.

As alluded to earlier, the rotating shaft 18 has a tendency to experience relatively intense mechanical vibration which detrimentally affects the process. In this regard, it has been found that by mounting one or more tuned dampers 20 at various points along the length of the rotating shaft 18 minimizes or eliminates such undesirable mechanical vibration. Turning now collectively to Figs. 3 and 4, wherein Fig. 3 discloses a partial side cross-sectional view of the tuned damper 20 and Fig. 4 shows an end view of shaft 18 with the tuned damper 20 mounted thereon. It can be seen from Figs. 3 and 4 that the tuned damper 20 includes four cylinder-shaped damping apparatus 26 to minimize the shaft 18 mechanical vibration. The tuned damper 20 includes at least two, and preferably four, of these so-called cylinder-shaped damping apparatus 26, which are mounted about every 90° on the shaft 18 and connected via connecting bars 23. The damping apparatus 26 can be attached to the rotating shaft 18 by any known convenient mechanical apparatus such as a split ring assembly that is then enclosed by a cover casing 21 as depicted in Figs. 2, 3 and 4. The outer casing 21 of the damping apparatus of the tuned damper 20 is preferably made of a material able to withstand the environment to which it is exposed. In the current process, the environment is detergent ingredients which permits the outer casing 21 to be made from plastic.

The damping apparatus 26 includes a pair of tuning rods 28 and 30 mounted adjacent to a pair of flexible or spring-like members 32 and 34. The flexible members 32 and 34 may be made of any flexible material such as an elastomeric material including nitrile, natural or synthetic butyl rubbers (e.g. from Ilene Industries, Inc.) or can be molded to the final shape by a supplier (e.g. Forsheda AB, Sweden). The flexible members 32 and 34 contact a weight 35 which is instrumental in damping the mechanical vibration resulting from the continuous rotating speed of the shaft 18.

Preferably, the tuned damper 20 is adjusted via manipulation and mounting location of the damping apparatus 26 such that the peak vibration of the shaft 18 is within a selected range. Specifically, the tuned damper 20 is adjusted via sizing of the flexible members 32 and 34 to achieve the desired natural frequency of the tuned damper 20. Preferably, the tuned damper 20 is tuned such that its natural frequency is about 95% of the shaft 18 natural frequency. If more than one tuned damper 20 is used, the natural frequency of each tuned damper 20 mounted on shaft 18 can be distributed at several frequencies in the 10 Hz to 20 Hz range, or can all be tuned to the same frequency.

When the tuned damper 20 is properly tuned, the preferred peak vibration range for the shaft 18 is from about -1.0G to about 1.0G, more preferably from about -0.5G to about 0.5G, and most preferably from about -0.25G to about 0.25G, in a frequency range of from about 10 Hz to about 20 Hz as measured at the mid-span length of the shaft 18. Such peak vibration ranges represent a dramatic improvement over peak vibrations typically experienced in commercial scale manufacturing facilities which can be on the order of $\pm 3.5G$. The peak vibration is preferably measured using 90 second intervals. As those skilled in the art will appreciate, such peak vibration measurements are easily completed using a conventional accelerometer (available from PCB 308B ICP Company, Buffalo, NY).

For example, during continuous operation of the process, the vibration of shaft 18 is recorded using the accelerometer attached to a power supply (e.g., PCB 480E090 from ICP Company, and a battery operated digital tape recorder such as the Sony TCD-D3 DAT recorder). The recorder and power supply are mounted in a waterproof, shock proof enclosure (such as the Carlon CJ1085 Enclosure). The sensor is attached to the shaft 18 by means of dental cement or other rigid adhesive and is positioned such that radial shaft vibration is measured. The vibration is recorded for at least 2 hours, preferably for 4 hours. Upon completion of the run, the tape recorder is removed from the enclosure and played back. The data is plotted in 90 second intervals on either a digital or analog plotting device (such as the Hewlett-Packard 3560A or 35670A analyzers). The peak and rms values of the vibration can be determined from a PC-based software program such as Excel or Matlab.

While not intending to be bound by theory, the negative peak vibrations associated with the moderate speed mixer/densifier 16 are especially exacerbated when the so-called "detergent wall" is present on the inner surface of the mixer/densifier 16. During the process, an optional detergent coating or wall of at least about a 1 mm can cover a portion of the inner wall surface of the moderate speed mixer/densifier 16. The portion of the inner wall covered by this coating having a minimum thickness of 1 mm can be substantial (up to 100% of the inner surface covered) or a mere 50% can be covered. The presence of this detergent coating provides an irregular surface contour against which the ploughs 24 are contacted during rotation of the shaft

18 of the moderate speed mixer/densifier 16. This irregular surface results in the shaft 18 being subjected to irregular forces, thereby further enhancing the overall mechanical vibration. The tuned damper 20, however, eliminates or minimizes this additional mechanical vibration, as well.

Referring collectively to Figs. 1-4, the preferred density of the resulting detergent agglomerates, or spray-dried granules of the alternative embodiment, exiting the moderate speed mixer/densifier 16 is at least 650 g/l, more preferably from about 700 g/l to about 800 g/l. The particle porosity of the resulting detergent agglomerates of the composition is preferably in a range from about 5% to about 20%, more preferably at about 10%. While the detergent agglomerates or spray-dried granules exiting the moderate speed mixer/densifier 16 are ready for packaging and sale as a low dosage, compact detergent product at this point, they can be subjected to one or more optional processing steps.

Optional Process Steps

Optionally, the detergent agglomerates may be dried in a fluid bed dryer 36 or similar apparatus. In another optional step of the present process, the detergent agglomerates exiting the fluid bed dryer 36 are further conditioned by cooling the agglomerates in a fluid bed cooler 38 or similar apparatus as are well known in the art.

Another optional process step involves adding a coating agent to improve flowability and/or minimize over agglomeration of the detergent composition in one or more of the following locations of the instant process: (1) the coating agent can be added directly after the fluid bed cooler 38 as shown by coating agent stream 40 (preferred); (2) the coating agent may be added between the fluid bed dryer 36 and the fluid bed cooler 38 as shown by coating agent stream 42; (3) the coating agent may be added between the fluid bed dryer 36 and the moderate speed mixer/densifier 16 as shown by stream 44; and/or (4) the coating agent may be added directly to the moderate speed mixer/densifier 16 and the fluid bed dryer 36 as shown by stream 46. It should be understood that the coating agent can be added in any one or a combination of streams 40, 42, 44, and 46 as shown in Fig. 1. The coating agent stream 40 is the most preferred in the instant process.

The coating agent is preferably selected from the group consisting of aluminosilicates, silicates, carbonates and mixtures thereof. The coating agent not only enhances the free flowability of the resulting detergent composition which is desirable by consumers in that it permits easy scooping of detergent during use, but also serves to control agglomeration by preventing or minimizing over agglomeration, especially when added directly to the moderate speed mixer/densifier 16. As those skilled in the art are well aware, over agglomeration can lead to very undesirable flow properties and aesthetics of the final detergent product.

Optionally, the process can comprises the step of spraying an additional binder in one or both of the mixer/densifiers 10 and 16. A binder is added for purposes of enhancing agglomeration by providing a "binding" or "sticking" agent for the detergent components. The binder is preferably selected from the group consisting of water, anionic surfactants, nonionic surfactants, polyethylene glycol, polyvinyl pyrrolidone polyacrylates, citric acid and mixtures thereof. Other suitable binder materials including those listed herein are described in Beerse et al, U.S. Patent No. 5,108,646 (Procter & Gamble Co.).

Other optional steps contemplated by the present process include screening the oversized detergent agglomerates or spray-dried granules in a screening apparatus 48 which can take a variety of forms including but not limited to conventional screens chosen for the desired particle size of the finished detergent product. Other optional steps include conditioning of the detergent agglomerates by subjecting the agglomerates to additional drying.

Another optional step of the instant process entails finishing the resulting detergent agglomerates by a variety of processes including spraying and/or admixing other conventional detergent ingredients, collectively referenced as the finishing step 50 in Fig. 1. For example, the finishing step encompasses spraying perfumes, brighteners and enzymes onto the finished agglomerates to provide a more complete detergent composition. Such techniques and ingredients are well known in the art.

Detergent Surfactant Paste

The detergent surfactant paste used in the process is preferably in the form of an aqueous viscous paste, although forms are also contemplated by the invention. This so-called viscous surfactant paste has a viscosity of from about 5,000 cps to about 100,000 cps, more preferably from about 10,000 cps to about 80,000 cps, and contains at least about 10% water, more preferably at least about 20% water. The viscosity is measured at 70°C and at shear rates of about 10 to 100 sec.⁻¹. Furthermore, the surfactant paste, if used, preferably comprises a deterative surfactant in the amounts specified previously and the balance water and other conventional detergent ingredients.

The surfactant itself, in the viscous surfactant paste, is preferably selected from anionic, nonionic, zwitterionic, ampholytic and cationic classes and compatible mixtures thereof.

Detergent surfactants useful herein are described in U.S. Patent 3,664,961, Norris, issued May 23, 1972, and in U.S. Patent 3,919,678, Laughlin et al., issued December 30, 1975.

Useful cationic surfactants also include those described in U.S. Patent 4,222,905, Cockrell, issued September 16, 1980, and in U.S. Patent 4,239,659, Murphy, issued December 16, 1980.

Of the surfactants, anionics and nonionics are preferred and anionics are most preferred.

Nonlimiting examples of the preferred anionic surfactants useful in the surfactant paste include the conventional C_{11} - C_{18} alkyl benzene sulfonates ("LAS"), primary, branched-chain and random C_{10} - C_{20} alkyl sulfates ("AS"), the C_{10} - C_{18} secondary (2,3) alkyl sulfates of the formula $CH_3(CH_2)_x(CHOSO_3^-M^+)CH_3$ and $CH_3(CH_2)_y(CHOSO_3^-M^+)CH_2CH_3$ where x and $(y + 1)$ are integers of at least about 7, preferably at least about 9, and M is a water-solubilizing cation, especially sodium, unsaturated sulfates such as oleyl sulfate, and the C_{10} - C_{18} alkyl alkoxy sulfates ("AEXS"; especially EO 1-7 ethoxy sulfates).

Optionally, other exemplary surfactants useful in the paste of the invention include and C_{10} - C_{18} alkyl alkoxy carboxylates (especially the EO 1-5 ethoxycarboxylates), the C_{10} - C_{18} glycerol ethers, the C_{10} - C_{18} alkyl polyglycosides and their corresponding sulfated polyglycosides, and C_{12} - C_{18} alpha-sulfonated fatty acid esters. If desired, the conventional nonionic and amphoteric surfactants such as the C_{12} - C_{18} alkyl ethoxylates ("AE") including the so-called narrow peaked alkyl ethoxylates and C_6 - C_{12} alkyl phenol alkoxyates (especially ethoxylates and mixed ethoxy/propoxy), C_{12} - C_{18} betaines and sulfobetaines ("sultaines"), C_{10} - C_{18} amine oxides, and the like, can also be included in the overall compositions. The C_{10} - C_{18} N-alkyl polyhydroxy fatty acid amides can also be used. Typical examples include the C_{12} - C_{18} N-methylglucamides. See WO 92/06154. Other sugar-derived surfactants include the N-alkoxy polyhydroxy fatty acid amides, such as C_{10} - C_{18} N-(3-methoxypropyl) glucamide. The N-propyl through N-hexyl C_{12} - C_{18} glucamides can be used for low sudsing. C_{10} - C_{20} conventional soaps may also be used. If high sudsing is desired, the branched-chain C_{10} - C_{16} soaps may be used. Mixtures of anionic and nonionic surfactants are especially useful. Other conventional useful surfactants are listed in standard texts.

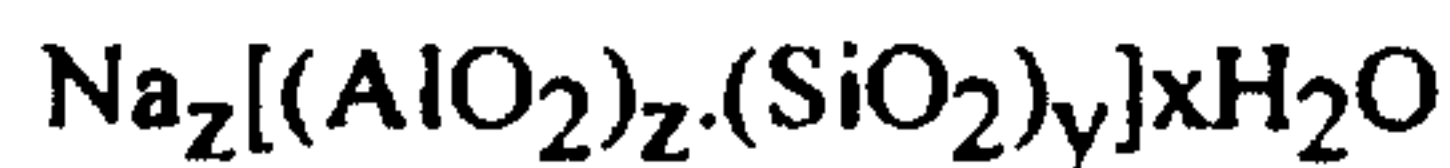
Dry Detergent Material

The starting dry detergent material of the present process preferably comprises a detergent aluminosilicate builder which are referenced as aluminosilicate ion exchange materials and sodium carbonate. The aluminosilicate ion exchange materials used herein as a detergent builder preferably have both a high calcium ion exchange capacity and a high exchange rate. Without intending to be limited by theory, it is believed that such high calcium ion exchange rate and capacity are a function of several interrelated factors which derive from the method by which the aluminosilicate ion exchange material is produced. In that regard, the aluminosilicate ion exchange materials used herein are preferably produced in accordance with Corkill et al, U.S. Patent No. 4,605,509 (Procter & Gamble).

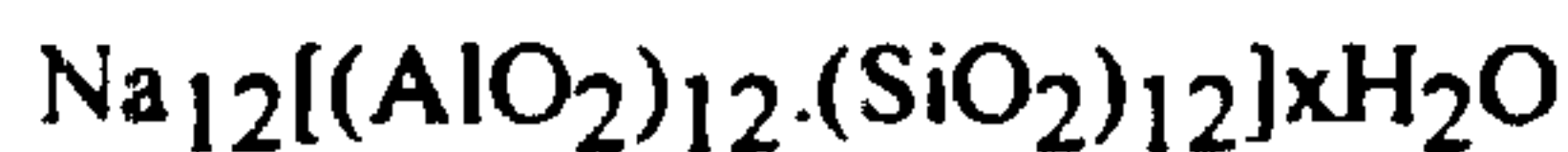
Preferably, the aluminosilicate ion exchange material is in "sodium" form since the potassium and hydrogen forms of the instant aluminosilicate do not exhibit the as high of an

exchange rate and capacity as provided by the sodium form. Additionally, the aluminosilicate ion exchange material preferably is in over dried form so as to facilitate production of crisp detergent agglomerates as described herein. The aluminosilicate ion exchange materials used herein preferably have particle size diameters which optimize their effectiveness as detergent builders. The term "particle size diameter" as used herein represents the average particle size diameter of a given aluminosilicate ion exchange material as determined by conventional analytical techniques, such as microscopic determination and scanning electron microscope (SEM). The preferred particle size diameter of the aluminosilicate is from about 0.1 micron to about 10 microns, more preferably from about 0.5 microns to about 9 microns. Most preferably, the particle size diameter is from about 1 microns to about 8 microns.

Preferably, the aluminosilicate ion exchange material has the formula



wherein z and y are integers of at least 6, the molar ratio of z to y is from about 1 to about 5 and x is from about 10 to about 264. More preferably, the aluminosilicate has the formula



wherein x is from about 20 to about 30, preferably about 27. These preferred aluminosilicates are available commercially, for example under designations Zeolite A, Zeolite B and Zeolite X.

Alternatively, naturally-occurring or synthetically derived aluminosilicate ion exchange materials suitable for use herein can be made as described in Krummel et al, U.S. Patent No. 3,985,669.

The aluminosilicates used herein are further characterized by their ion exchange capacity which is at least about 200 mg equivalent of CaCO_3 hardness/gram, calculated on an anhydrous basis, and which is preferably in a range from about 300 to 352 mg equivalent of CaCO_3 hardness/gram. Additionally, the instant aluminosilicate ion exchange materials are still further characterized by their calcium ion exchange rate which is at least about 2 grains Ca^{++} /gallon/minute/-gram/gallon, and more preferably in a range from about 2 grains Ca^{++} /gallon/minute/-gram/gallon to about 6 grains Ca^{++} /gallon/minute/-gram/gallon.

Adjunct Detergent Ingredients

The starting dry detergent material in the present process can include additional detergent ingredients and/or, any number of additional ingredients can be incorporated in the detergent composition during subsequent steps of the present process. These adjunct ingredients include other detergency builders, bleaches, bleach activators, suds boosters or suds suppressers, anti-tarnish and anticorrosion agents, soil suspending agents, soil release agents, germicides, pH adjusting agents, non-builder alkalinity sources, chelating agents, smectite clays, enzymes, enzyme-stabilizing agents and perfumes. See U.S. Patent 3,936,537, issued February 3, 1976 to Baskerville, Jr. et al.

Other builders can be generally selected from the various water-soluble, alkali metal, ammonium or substituted ammonium phosphates, polyphosphates, phosphonates, polyphosphonates, carbonates, borates, polyhydroxy sulfonates, polyacetates, carboxylates, and polycarboxylates. Preferred are the alkali metal, especially sodium, salts of the above. Preferred for use herein are the phosphates, carbonates, C₁₀₋₁₈ fatty acids, polycarboxylates, and mixtures thereof. More preferred are sodium tripolyphosphate, tetrasodium pyrophosphate, citrate, tartrate mono- and di-succinates, and mixtures thereof (see below).

In comparison with amorphous sodium silicates, crystalline layered sodium silicates exhibit a clearly increased calcium and magnesium ion exchange capacity. In addition, the layered sodium silicates prefer magnesium ions over calcium ions, a feature necessary to insure that substantially all of the "hardness" is removed from the wash water. These crystalline layered sodium silicates, however, are generally more expensive than amorphous silicates as well as other builders. Accordingly, in order to provide an economically feasible laundry detergent, the proportion of crystalline layered sodium silicates used must be determined judiciously.

The crystalline layered sodium silicates suitable for use herein preferably have the formula



wherein M is sodium or hydrogen, x is from about 1.9 to about 4 and y is from about 0 to about 20. More preferably, the crystalline layered sodium silicate has the formula



wherein M is sodium or hydrogen, and y is from about 0 to about 20. These and other crystalline layered sodium silicates are discussed in Corkill et al, U.S. Patent No. 4,605,509.

Specific examples of inorganic phosphate builders are sodium and potassium tripolyphosphate, pyrophosphate, polymeric metaphosphate having a degree of polymerization of from about 6 to 21, and orthophosphates. Examples of polyphosphonate builders are the sodium and potassium salts of ethylene diphosphonic acid, the sodium and potassium salts of ethane 1-hydroxy-1, 1-diphosphonic acid and the sodium and potassium salts of ethane, 1,1,2-triphosphonic acid. Other phosphorus builder compounds are disclosed in U.S. Patents 3,159,581; 3,213,030; 3,422,021; 3,422,137; 3,400,176 and 3,400,148.

Examples of nonphosphorus, inorganic builders are tetraborate decahydrate and silicates having a weight ratio of SiO₂ to alkali metal oxide of from about 0.5 to about 4.0, preferably from about 1.0 to about 2.4. Water-soluble, nonphosphorus organic builders useful herein include the various alkali metal, ammonium and substituted ammonium polyacetates, carboxylates, polycarboxylates and polyhydroxy sulfonates. Examples of polyacetate and polycarboxylate builders are the sodium, potassium, lithium, ammonium and substituted

ammonium salts of ethylene diamine tetraacetic acid, nitrilotriacetic acid, oxydisuccinic acid, mellitic acid, benzene polycarboxylic acids, and citric acid.

Polymeric polycarboxylate builders are set forth in U.S. Patent 3,308,067, Diehl, issued March 7, 1967. Such materials include the water-soluble salts of homo- and copolymers of aliphatic carboxylic acids such as maleic acid, itaconic acid, mesaconic acid, fumaric acid, aconitic acid, citraconic acid and methylene malonic acid. Some of these materials are useful as the water-soluble anionic polymer as hereinafter described, but only if in intimate admixture with the non-soap anionic surfactant.

Other suitable polycarboxylates for use herein are the polyacetal carboxylates described in U.S. Patent 4,144,226, issued March 13, 1979 to Crutchfield et al, and U.S. Patent 4,246,495, issued March 27, 1979 to Crutchfield et al. These polyacetal carboxylates can be prepared by bringing together under polymerization conditions an ester of glyoxylic acid and a polymerization initiator. The resulting polyacetal carboxylate ester is then attached to chemically stable end groups to stabilize the polyacetal carboxylate against rapid depolymerization in alkaline solution, converted to the corresponding salt, and added to a detergent composition. Particularly preferred polycarboxylate builders are the ether carboxylate builder compositions comprising a combination of tartrate monosuccinate and tartrate disuccinate described in U.S. Patent 4,663,071, Bush et al., issued May 5, 1987.

Bleaching agents and activators are described in U.S. Patent 4,412,934, Chung et al., issued November 1, 1983, and in U.S. Patent 4,483,781, Hartman, issued November 20, 1984. Chelating agents are also described in U.S. Patent 4,663,071, Bush et al., from Column 17, line 54 through Column 18, line 68. Suds modifiers are also optional ingredients and are described in U.S. Patents 3,933,672, issued January 20, 1976 to Bartoletta et al., and 4,136,045, issued January 23, 1979 to Gault et al.

Suitable smectite clays for use herein are described in U.S. Patent 4,762,645, Tucker et al, issued August 9, 1988, Column 6, line 3 through Column 7, line 24. Suitable additional detergency builders for use herein are enumerated in the Baskerville patent, Column 13, line 54 through Column 16, line 16, and in U.S. Patent 4,663,071, Bush et al, issued May 5, 1987.

In order to make the present invention more readily understood, reference is made to the following examples, which are intended to be illustrative only and not intended to be limiting in scope.

EXAMPLE I

This Example illustrates the process of the invention which produces free flowing, crisp, high density detergent composition. Two feed streams of various detergent starting ingredients are continuously fed, at a rate of 2800 kg/hr, into a Lödige CB-30 mixer/densifier, one of which comprises a surfactant paste containing surfactant and water and the other stream containing starting dry detergent material containing aluminosilicate and sodium carbonate. The rotational speed of the shaft in the Lödige CB-30 mixer/densifier is about 1400 rpm and the mean residence time is about 10 seconds. The contents from the Lödige CB-30 mixer/densifier are continuously fed into a Lödige KM 600 mixer/densifier for further agglomeration during which the mean residence time is about 6 minutes. The Lödige KM 600 mixer/densifier includes four tune dampers connected by a central ring. The tuned dampers are located midway down the length of the mixer and are placed around the shaft in 90° intervals. Each mass or weight element weighs 30 lbs. and the flexible elements in the tuned dampers are sized and tuned to about 25 Hz. All four tune dampers are then enclosed in a single cover casing. The resulting detergent agglomerates are then fed to a fluid bed dryer and then to a fluid bed cooler, the mean residence time being about 10 minutes and 15 minutes, respectively. A coating agent, aluminosilicate, is fed about midway down the moderate speed mixer/densifier 16 to control and prevent over agglomeration. The detergent agglomerates are then screened with conventional screening apparatus resulting in a uniform particle size distribution. The composition of the detergent agglomerates exiting the fluid bed cooler is set forth in Table I below:

TABLE I

<u>Component</u>	<u>% Weight of Total Feed</u>
C ₁₄₋₁₅ alkyl sulfate/alkyl ethoxy sulfate	29.1
Aluminosilicate	34.4
Sodium carbonate	17.5
Polyethylene glycol (MW 4000)	1.3
Misc. (water, etc.)	<u>16.7</u>
	100.0

Additional detergent ingredients including perfumes, enzymes, and other minors are sprayed onto the agglomerates described above in the finishing step to result in a finished detergent composition. The relative proportions of the overall finished detergent composition produced by the process of instant process is presented in Table II below:

TABLE II

<u>Component</u>	<u>(% weight)</u>
	<u>A</u>
C ₁₄₋₁₅ alkyl sulfate/C ₁₄₋₁₅ alkyl ethoxy sulfate	16.3
Neodol 23-6.5 ¹	3.0
C ₁₂₋₁₄ N-methyl glucamide	0.9
Polyacrylate (MW=4500)	3.0
Polyethylene glycol (MW=4000)	1.2
Sodium Sulfate	8.9
Aluminosilicate	26.3
Sodium carbonate	27.2
Protease enzyme	0.4
Amylase enzyme	0.1
Lipase enzyme	0.2
Cellulase enzyme	0.1
Minors (water, perfume, etc.)	<u>12.4</u>
	100.0

¹ C₁₂₋₁₃ alkyl ethoxylate (EO=6.5) commercially available from Shell Oil Company.

The density of the resulting detergent composition is 796 g/l, the mean particle size is 613 microns.

EXAMPLE II

This Example illustrates another process in accordance with the invention in which granules are first spray dried then mixed in a moderate speed mixer/densifier. The composition of the detergent in Table III below is made in a 10 foot diameter spray drying tower with counter current air at 300°C at a rate of 2800 lb/hr. The granules are fed into a Lödige KM-600 with a residence time of about 12 minutes. The Lödige KM 600 includes four tune dampers connected by a central ring. The tuned dampers are located midway down the length of the mixer and are placed around the shaft in 90° intervals. Each mass or weight element weighs 30 lbs. and the flexible elements are sized and tuned to about 25.Hz. All four tune dampers are then enclosed in a single cover casing.

TABLE III

<u>Component</u>	<u>% Weight of Total Feed</u>
C ₁₄₋₁₅ alkyl sulfate/alkyl ethoxy sulfate	10.2
C ₁₂₋₁₃ linear alkylbenzene sulfonate	10.2
Aluminosilicate	27.0
Sodium carbonate	21.9

Polyethylene glycol (MW 4000)	1.4
Polyacrylate (MW=4500)	4.0
Sodium Sulfate	13.0
Brightener	0.3
Sodium Silicate (1.6r)	1.0
Misc. (water, perfume, etc.)	<u>11.0</u>
	100.0

Additional detergent ingredients including perfumes and enzymes are sprayed onto the granules described above in the finishing step to result in a finished detergent composition. The relative proportions of the overall finished detergent composition produced by the process of instant process is presented in Table IV below:

TABLE IV

<u>Component</u>	<u>(% weight)</u>
	<u>A</u>
C ₁₂₋₁₆ linear alkylbenzene sulfonate	9.0
C ₁₄₋₁₅ alkyl sulfate/C ₁₄₋₁₅ alkyl ethoxy sulfate	9.0
Neodol 23-6.5 ¹	2.0
Polyacrylate (MW=4500)	3.5
Polyethylene glycol (MW=4000)	1.2
Sodium Sulfate	11.4
Sodium Silicate (1.6r)	0.9
Aluminosilicate	23.8
Brightener	0.3
Sodium carbonate	28.4
Protease enzyme	0.4
Amylase enzyme	0.1
Lipase enzyme	0.2
Cellulase enzyme	0.1
Minors (water, perfume, etc.)	<u>9.7</u>
	100.0

¹ C₁₂₋₁₃ alkyl ethoxylate (EO=6.5) commercially available from Shell Oil Company.

The density of the resulting detergent composition is 822 g/l.

Having thus described the invention in detail, it will be clear to those skilled in the art that various changes may be made without departing from the scope of the invention and the invention is not to be considered limited to what is described in the specification.

WHAT IS CLAIMED IS:

1. A process for continuously preparing a high density detergent composition characterized by the steps of:
 - (a) continuously mixing a detergent surfactant paste having a viscosity of from about 5,000 cps to about 100,000 cps and dry starting detergent material comprising an aluminosilicate builder into a high speed mixer/densifier to obtain detergent agglomerates;
 - (b) mixing said detergent agglomerates in a moderate speed mixer/densifier to further densify and agglomerate said detergent agglomerates, wherein said moderate speed mixer/densifier includes a centrally rotating shaft on which a tuned damper apparatus is mounted such that the peak vibration of said shaft is from -1.0G to 1.0G in a frequency range of from 10 Hz to 20 Hz at the mid-span length of said shaft; and
 - (c) drying said detergent agglomerates so as to form said high density detergent composition.
2. The process of claim 1 wherein said moderate speed mixer/densifier includes at least a 1 mm detergent coating a portion of the inner wall surface of said moderate speed mixer/densifier.
3. The process of claim 1 or 2 wherein the density of said high density detergent composition is at least 650 g/l.
4. The process of any one of claims 1 to 3 wherein the peak vibration of said shaft is from -0.5G to 0.5G.
5. The process of any one of claims 1 to 4 wherein the peak vibration of said shaft is from -0.25G to 0.25G.
6. The process of any one of claims 1 to 5 further characterized by the step of adding a coating agent after said high speed mixer/densifier, wherein said coating agent is selected from the group consisting of aluminosilicates, carbonates, silicates and mixtures thereof.

7. The process of any one of claims 1 to 6 further characterized by the step of cooling said detergent agglomerates.
8. The process of any one of claims 1 to 7 wherein the mean residence time of said detergent agglomerates in said high speed mixer/densifier is in range from 2 seconds to 45 seconds.
9. The process of any one of claims 1 to 8 wherein the mean residence time of said detergent agglomerates in said moderate speed mixer/densifier is in range from 0.5 minutes to 15 minutes.
10. A process for continuously preparing a high density detergent composition characterized by the steps of:
 - (a) continuously mixing spray-dried detergent granules into a moderate speed mixer/densifier, wherein said speed mixer/densifier includes a centrally rotating shaft on which a tuned damper apparatus is mounted such that the peak vibration of said shaft is from -1.0G to 1.0G in a frequency range of from 10 Hz to 20 Hz at the mid-span length of said shaft; and
 - (b) drying said detergent agglomerates so as to form said high density detergent composition.

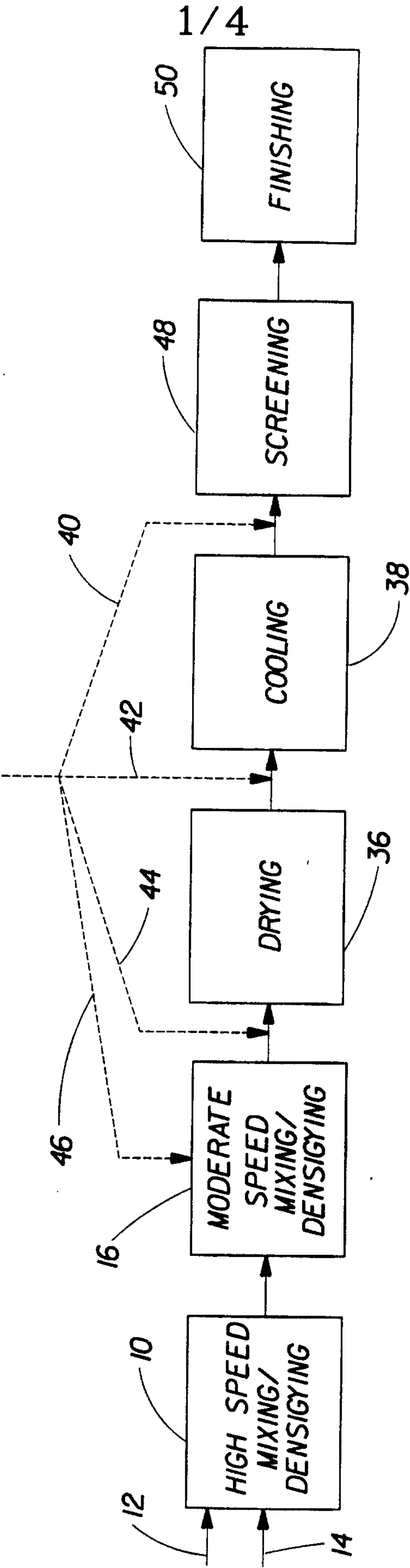


Fig. 1

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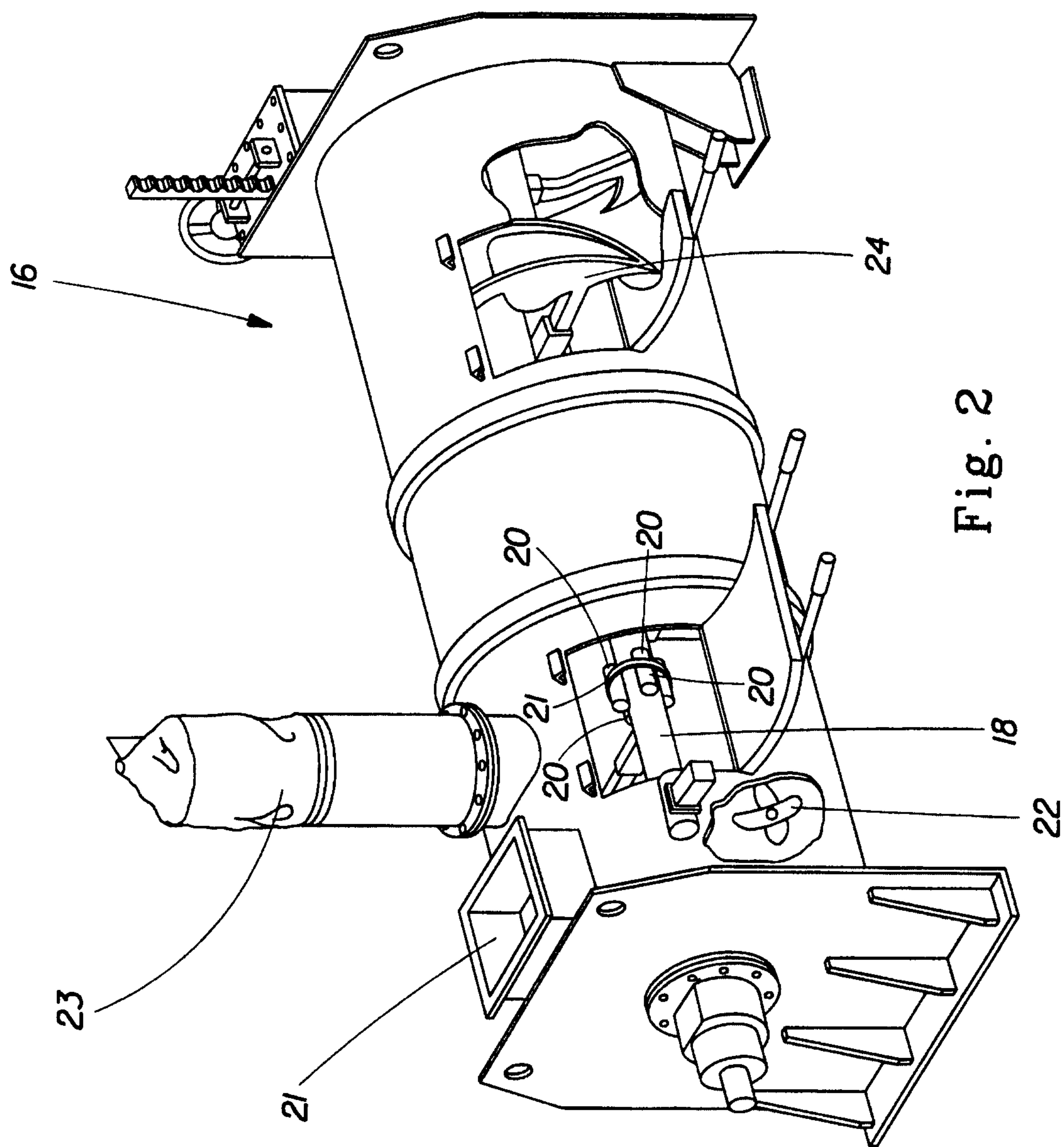


Fig. 2

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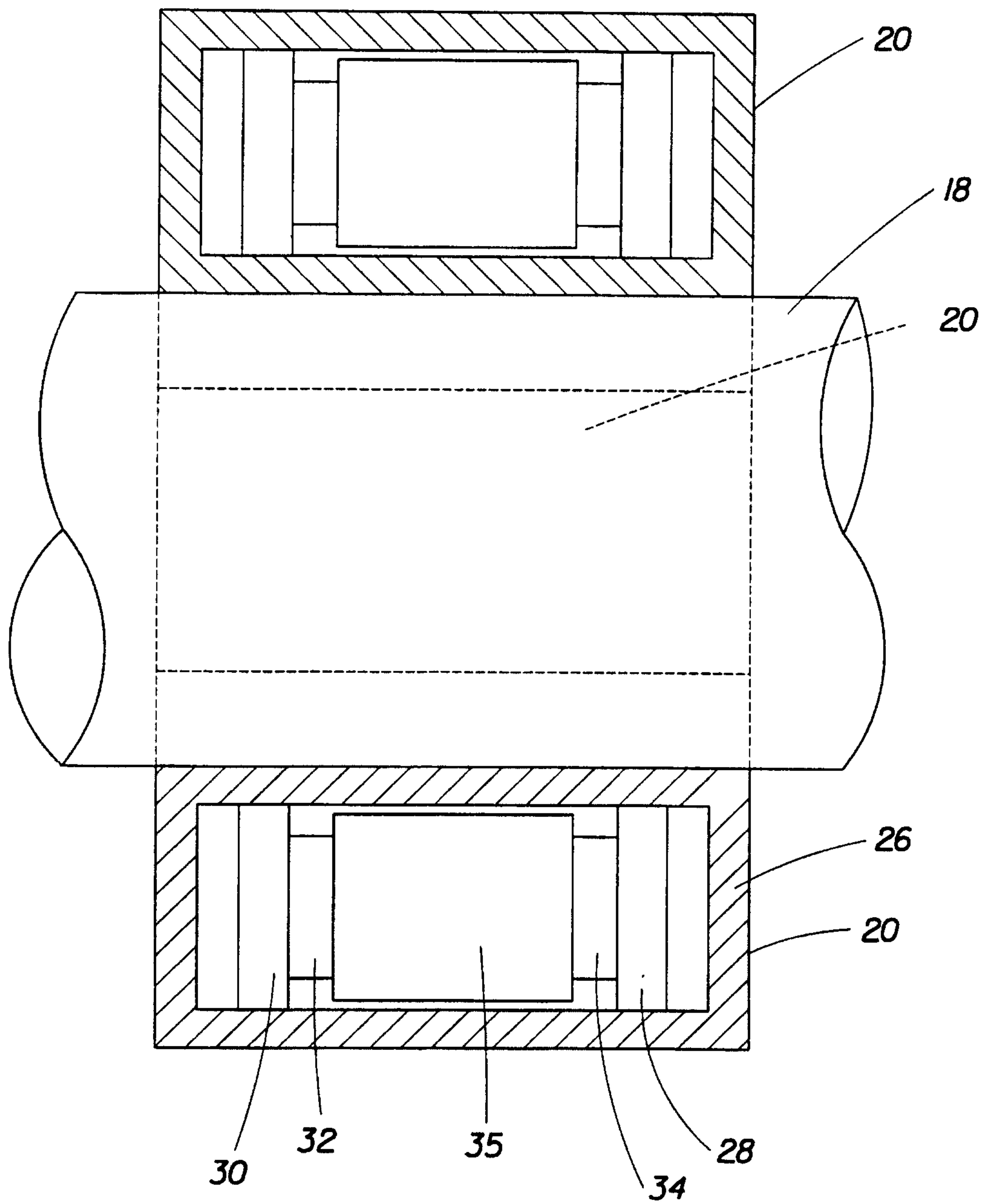


Fig. 3

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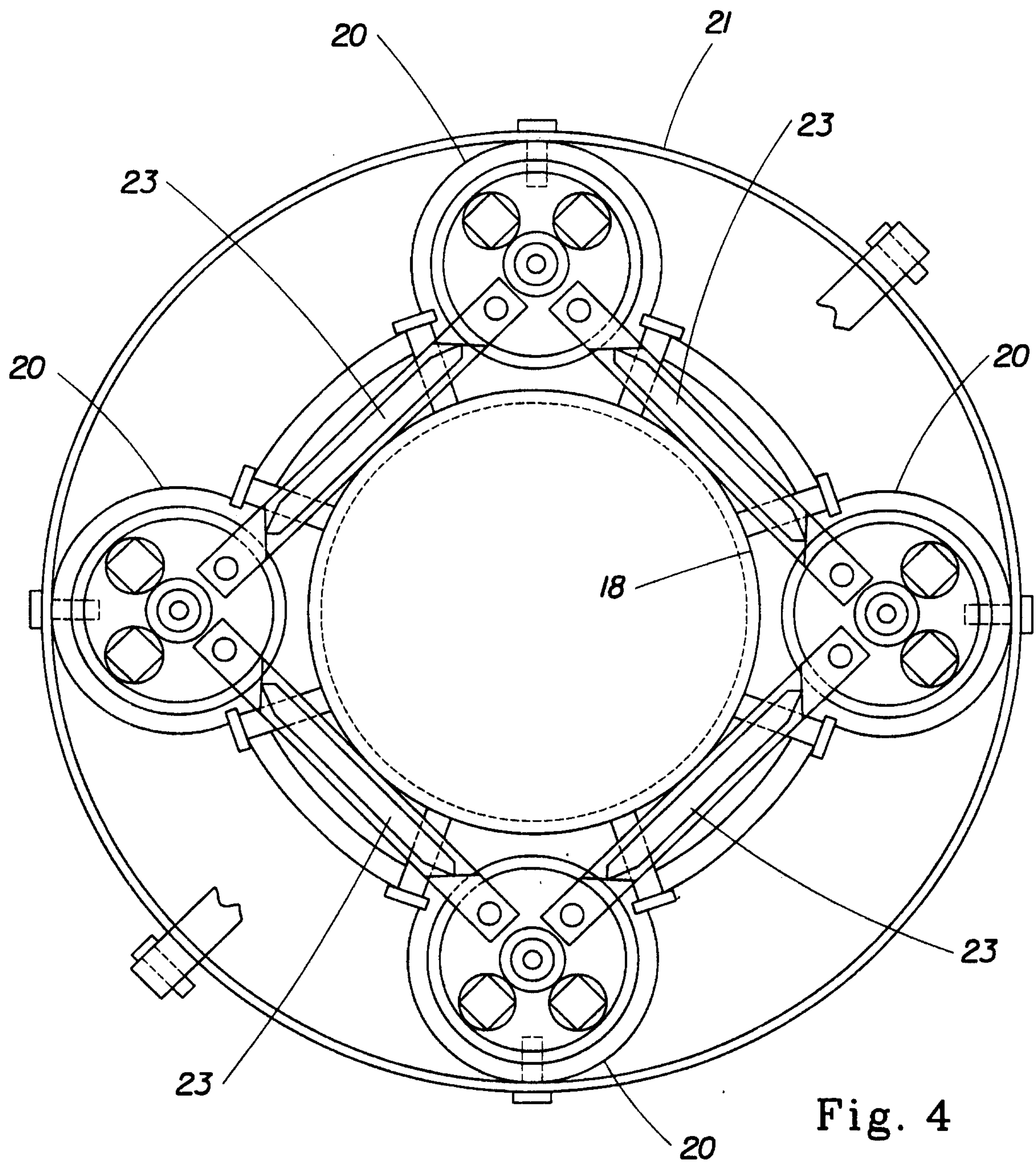


Fig. 4