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DETERGENT BRIQUETTE AND PROCESS FOR THE PRODUCTION THEREOF

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This invention relates to a new improved detergent briquette and a process for the production thereof. More particularly it relates to a water-soluble detergent briquette characterized by the fact that it is readily disintegrated by mild agitation when placed in water, while still being of sufficient surface and internal strength, in combination, to withstand the ordinary shocks and abrasions of packaging, handling and transportation without rubbing off of the surface material or fracturing or crumbling of the briquette. Similarly this invention relates to a process for producing such a briquette.

It has been known to produce form-retaining detergent products by high pressure pressing of powdered materials but this method presents many disadvantages. The greatest of these is that when higher pressures (over approximately 100 lbs./sq. in.) are used the detergent composition, unless it consists principally of inorganic detergent or contains very little free water or other plasticizing or solvent material, will cohere to a certain extent thereby rendering impossible the rapid disintegration of the briquette in water, since the highly cohering cake, being relatively impermeable to water, presents only a limited surface available for solution.

If the detergent composition particles are pressed under low pressure the particles will not cohere so completely as to prevent disintegration when placed in water but, on the contrary, will be so lightly bound to one another that they will break apart when subjected to comparatively slight handling and transportation shocks.

The present invention, contrary to those methods mentioned above, enables one to produce a product that will be of commercially acceptable strength, i. e., strength sufficient to inhibit crumbling or breakage, while yet being easily disintegrable in mildly agitated water.

In accordance with the invention particles of detergent containing hydratable inorganic salt are lightly compressed to a form-retaining condition and thereafter the surfaces of the resulting briquette are moistened so as to hydrate the inorganic sale and increase the strength and abrasion resistance of the product. Briquettes so moistened and hydrated are found to possess surface strength and surface hardness greater than that of untreated briquettes.

To form the disintegrable products of this invention it is generally desirable to employ low pressure pressing of the briquette, usually from 3–40 pounds per square inch and preferably from 4–6 pounds per square inch pressure being used to press a briquette approximately ½ inch thick. Use of low pressures prevents the formation of too strong a bond between adjoining detergent composition particles and so insures that the briquette will be easily disintegrated by water.

However, while examples of pressure ranges have been given, it is plain that the optimum pressing pressure is dependent upon the particular composition of the detergent product, since the applied pressure that will form a cohesive, yet water-disintegrable briquette will vary, depending on the inherent plasticity of the detergent composition ingredients. Such plasticity will be increased, and the required compacting pressure correspondingly decreased as the free water content and/or the percentage of more plastic constituents of the composition are/is raised. An uncompensated decrease in either the free water or more plastic constituents will naturally result in an increase in the optimum compacting pressure.

The term optimum compacting pressure, or optimum pressing pressure is defined as that pressure at which the briquette formed will be easily disintegrable by slightly agitated water while, after surface treatment according to the process of this invention, still being capable of being handled in the usual channels of commerce without excessive crumbling, fracturing or rubbing off of detergent composition. A briquette is considered readily disintegrable if it is more than 95% disintegrated within two minutes in mildly agitated warm water. However in certain instances, e. g., dishwashing, it will be highly advantageous to form briquettes that will disintegrate under the same conditions in thirty seconds or less.

In the best mode of this invention the detergent composition particles will be in the forms of hollow beads or spheres immediately prior to pressing, such as those particles obtained by spray-drying. Of course during the pressing operation some of these beads will be crushed but nevertheless the briquettes made from them will be more readily soluble than briquettes made from solid particles due to the higher surface/weight ratio of the hollow beads.

The different ingredients of the detergent composition are uniformly distributed in the various beads since said beads are sprayed from a nearly-uniform slurry of components. It is not necessary however that spray-dried beads be used. Other heat- or air-dried compositions are also suitable or the various ingredients may be uniformly mixed in a substantially dry state and then pressed or they may be mixed and ground or powdered before pressing.

It may be found advantageous to add some water or other plasticizing agent to the detergent composition particles or the mixed or powdered components of a detergent composition before compacting to promote adhesion or cohesion of the particles. In some instances it will be desirable to allow such added water or plasticizer to "work into" or permeate the composition before pressing to avoid excessive moisture on the pressing surfaces and consequent imperfect die release and briquette surface marks.

The process of moistening of the surfaces of the pressed briquette with steam or a water spray may be carried out by impinging onto the briquette surface the stream of steam or spray of water while preferably passing the briquette on a continuous mesh screen or perforated belt through the moistening area.

The moisture may be applied to the briquettes as a fine mist or spray or as either wet or dry steam. It is preferred to utilize steam since, because of the heat it possesses, it fuses or dissolves more readily the surface of the detergent briquette, while still hydrating the hydratable inorganic salt present, and enables one to secure a harder more continuous surface after treatment, than with a water spray. Then too, steam treated briquettes dry more rapidly, due to their greater heat content and so are ready for packaging sooner. While steam treatment of the briquette surface is the preferred embodiment of this invention, it must be pointed out that water sprayed briquettes also are possessed of the same general desirable characteristics and in instances where steam is not readily available or when it is desired that more water be added to the briquette than is feasible by steam treatment, spray treatment may be even more advantageous.

The amount of moisture to be applied to the briquette

surface will be enough to cause the surface material to hydrate and fuse or dissolve sufficiently so that upon solidification of the fused surface either by hydration and drying, partial drying, cooling, crystallization, or combination thereof, there will be formed a film or layer of sufficient strength so as to prevent fracturing of the product or rubbing off of surface material when the briquettes are subjected to normal commercial handling and transportation. Usually the amount of moisture added will be less than ten percent of the dry weight of the detergent briquette and in most cases will be between ½ percent and five percent, but as is plain the amount used will be dependent on the particular detergent composition employed, capacity for hydration of its inorganic salt content and its moisture and/or plasticizer content.

It is believed that when moisture is applied to the briquette surface a number of physical actions takes place to promote the formation of a film or skin of harder material than the briquette interior. In the case of hydratable, crystallizable detergent compositions or detergent compositions containing hydratable, crystallizable ingredients, those components will crystallize and such action will create a greater binding force between adjacent particles. In some instances the moisture added will only partially hydrate salts and this too will create a crystal bond between adjacent particles. Crystalline bonds and crystals, it is believed, may also entrap and bind non-crystallizable components of the detergent compositions. It is evident that the briquettes treated according to the process of this invention, initially containing hydratable salt as they do, will be of a greater degree of hydration at their exterior surfaces than in the inner portions of the briquettes.

Particularly where the briquette is composed of a pressed hetrogeneous powder the dissolving, hydrating and recrystallizing action caused by application of moisture will make the surface more homogeneous, increasing the surface density and the cohesive force between particles. This will also be true where the detergent composition is in the form of homogeneous spray-dried beads.

Where the detergent composition contains amounts of relatively water-soluble non-crystalline, and non-crystallizable ingredients the application of moisture will promote surface hardening to some extent even with respect to the action of water on these substances, because it dissolves detergent composition constituents and so increases their adhesive power, possibly by increasing their contact surfaces.

It is also known that the moisture application rounds out the minor irregularly shaped projections that may be found on briquetted detergents by dissolving or melting the material thereof (this is readily done because such projections are of comparatively large area/volume ratios) and allowing it to spread out in a film over the briquette body. Since in ordinary handling and transportation of briquettes the outjutting portions of those irregularly formed are most easily broken off it is obvious that the invented process will decrease the amount of surface rub-off, crumbling and breakage of detergent briquettes subjected to it.

After the detergent briquettes have been pressed and moistened they may be air- or heat-dried. Preferably this drying is done by passing the briquettes on continuous mesh belts through a drying atmosphere but also contemplated is the mere storage of the product after moistening, until it is sufficiently dry to withstand handling. Briquettes containing hydratable material sometimes require no drying at all after the moistening step since the hydratable material will combine with the added moisture.

The present invention is of a process applicable to water soluble anionic synthetic organic detergent materials containing hydratable salt. Among suitable detergent compounds alkyl benzene sulfonates, fatty acid monoglyceride sulfates, fatty alcohol sulfates, all with alkyl or acyl groups of from 10–18 carbon atoms, are the preferred compounds while sodium tripolyphosphate, tetrasodium pyrophosphate and sodium sulfate are the most prominent of the builders and extenders. Of course normal adjuvant material such as perfumes, foam stabilizers, foaming agents, whiteners, preservatives, etc., may also be detergent ingredients.

Detergent briquettes made by the process of the instant invention are ideal for use in commercial or domestic laundries since they furnish a means of conveniently and accurately measuring, without weighing, a predetermined quantity of detergent into known amounts of wash water, thereby leading to a conservation of detergent and to the obtaining of consistently cleaner clothes because the optimum detergent concentration for effective cleansing action can always be guaranteed.

While it is contemplated that the invented briquettes will find their greatest use in the laundering of textiles, particularly soiled clothing, they will of course also be advantageously employed in making detergent solutions such as those used for washing dishes, floors, walls, automobiles, machinery, manufactured articles, fruits and vegetables. The briquettes can be used in making textile dyeing baths, wool scours, and human hair shampoo solutions. They may be substituted for soap and detergent bars, powders and liquids for the cleaning of the human hands and body. In short the invented detergent briquettes will find application in almost every situation where detergents are employed.

The briquettes of this invention may be made of any of various shapes and sizes depending upon the purpose for which they are to be used. The briquette surfaces may be scored to facilitate division into smaller particles of known weight if it is desired to provide the consumer with means for making solutions of various concentrations without weighing the detergent, or to provide a briquette useful for making wash solutions of optimum detergent concentration where variously sized solution tubs or mixing tanks are used. Such scored briquettes in effect allow the consumer to "weigh" the detergent without a scale, and consequently are useful in enabling him to conveniently measure the amount of detergent desired.

Of course, a detergent composition can be briquetted under high pressure according to methods of the prior art. While not disintegrable in water such a briquette might still be capable of being broken up by the user before he attempts to dissolve it. However this necessitates the crumbling of the briquette, a chore which will probably be unpopular with most potential users of detergent briquettes.

In industrial applications the savings of time and money in making solutions etc., small though they may be in one instance, multiplied by the tens or thousands will militate against the purchasing of briquettes which must be reduced to powder before use.

In certain cases where harsh chemicals are included in the briquetted composition the employment of the processes and products of this invention will enable the user to avoid the intimate contact of such ingredients with his hands that is an unavoidable accompaniment of the necessary act of hand-crushing, before dissolving, such products which are briquetted under high pressure. Therefore the present briquettes will be useful in applications where operators are susceptible to dermatitis due to contact with skin-irritating detergent ingredients employed.

The following examples are given further to illustrate the nature of the invention made. It will be apparent that these examples are merely illustrative of the invention made and the invention is not to be limited thereto.

Example I

A heavy duty detergent composition containing 35% of the sodium salt of sulfonated alkylated benzene where

the alkyl group was mostly of 12 carbon atoms, 40% penta-sodium tripolyphosphate, 3% of sodium silicate (Na_2O/SiO_2 ratio of 1/3.2), 9% water and the balance of other adjuvant materials including sodium carboxymethyl cellulose, coloring agent, perfume, preservative, anti-tarnish agent, anti-caking agent, etc., was obtained by the conventional spray-drying process. This material was briquetted under pressure of 4 pounds per square inch into pieces ½" thick and weighing approximately 40 grams each.

After pressing, the briquettes were subjected to uniform treatment by saturated steam for 3 to 5 minutes and were then allowed to dry in air for 10 minutes.

The briquettes were tested for disintegrability in water and observations were made of their resistance to breakage. It was found that the briquettes disintegrated in from 60 to 90 seconds in mildly agitated water at a temperature between 80 degrees Fahrenheit and 100° F. and were sufficiently strong to permit commercial handling.

Example II

A detergent composition comprising the same ingredients as in Example I and in the same amounts was made by spray-drying the alkyl aryl sulfonate and then coating it with a composition of finely divided sodium tripolyphosphate and adjuvant materials listed in Example I above. The coating operation was carried out by tumbling in a drum the spray-dried alkyl aryl sulfonate with the finely divided other materials in the presence of moisture. The coated product thus made contained 9 percent moisture.

It was pressed into 40 gram briquettes ½ inch thick and 3½ inches in diameter using a pressure of 4.2 pounds per square inch. Two grams of water were uniformly sprayed onto each briquette so made and briquettes were allowed to dry for approximately 10 minutes in the room air.

The detergent product so processed disintegrated in 50–60 seconds when placed in warm water subjected to mild agitation but nevertheless was still sufficiently tough to permit ordinary commercial handling.

In other experiments briquettes were made with the composition of Example I, said composition in some instances being moistened before pressing. They were then pressed at 8, 16, 24, 40, 80 and 120 pounds per square inch pressure. Various briquettes made were steam treated or water sprayed while others were controls. It was found that none of the control briquettes were exhibited both the required surface strength and ready disintegrability required of the detergent briquette desired, but treated briquettes pressed at 4, 8, 16 and 24 pounds per square inch were found acceptable.

In the above specification and subsequent claims the word briquette has been used to designate shaped form-retaining compacted masses made up of a component powders, granules or other particles in which binders were not necessarily present.

Warm water is water at a temperature between 80° F. and 100° F. Where the term "mildly agitated" has been used as descriptive of the water in which a detergent briquette is dissolved, it means that the water was in such a state of motion as would be caused by the gentle movement of one's hand through it.

The above invention has been described in conjunction with various illustrative examples of acceptable briquettes. It will be obvious to those skilled in the art that other variations and modifications of the invention can be made and various equivalents substituted therein without departing from the principles disclosed herein or going outside the scope of the specification or purview of the claims.

Having thus described the invention, what is claimed is:

1. A process for making an improved detergent briquette which comprises compressing, at a pressure between 3 and 100 pounds per square inch, a plurality of particles of a normally solid water soluble detergent composition containing a water soluble anionic synthetic organic detergent and a hydratable inorganic salt to unitary form-retaining briquetted condition and thereafter moistening the surface of such formed briquette with an amount of water which is ½ to 10% of the weight of the detergent composition particles compressed, to increase the surface strength and abrasion resistance of the briquetted composition.

2. A process as set forth in claim 1 in which the briquette is formed by compressing detergent particles at a pressure between 3 and 40 pounds per square inch.

3. A process as set forth in claim 1 in which, after the moistening of the briquette surface, said surface is dried.

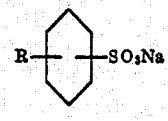
4. A process as set forth in claim 1 in which the water soluble anionic synthetic organic detergent is selected from the group consisting of sulfated and sulfonated detergents.

5. A process as set forth in claim 1 in which the particles of detergent composition are moistened before compressing to increase their cohesive and adhesive properties.

6. A process as set forth in claim 1 in which the water is applied as steam.

7. A process as set forth in claim 1 in which the particles of detergent composition are of substantially spherical shape produced by spray drying.

8. A process for making an improved detergent briquette comprising compressing a plurality of particles of a spray dried normally water soluble detergent composition, which composition comprises



where R is an alkyl group of from 10 to 18 carbon atoms, and pentasodium tripolyphosphate, at a pressure between 3 and 40 pounds per square inch to a unitary form retaining briquetted condition and thereafter moistening the surface of such formed briquette with an amount of water between ½ and 10% of the initial weight of the detergent composition particles to increase the degree of hydration of the inorganic salt and thereby increase the surface strength and abrasion resistance thereof.

9. A process for making an improved detergent briquette comprising compressing a plurality of particles of a sprayed dried detergent composition, which composition comprises an anionic organic synthetic detergent selected from the group consisting of sulfated and sulfonated detergents with a hydratable inorganic builder salt selected from the group consisting of pentasodium tripolyphosphate, tetrasodium pyrophosphate and sodium sulfate, at a pressure between 3 and 40 pounds per sq. inch, to a unitary form-retaining briquetted condition, and thereafter moistening the surface of such formed briquette with an amount of water between ½ and 10% of the initial weight of the detergent composition particles to increase the degree of hydration of the inorganic salt on the surface of the briquette and thereby increase the surface strength and abrasion resistance of the briquette.

10. A shaped water soluble detergent briquette readily disintegrable and soluble in mildly agitated warm water and possessed of commercially acceptable surface strength comprising a normally solid, water soluble detergent composition consisting essentially of a water soluble anionic synthetic organic detergent and a hydratable inorganic salt substantially uniformly distributed throughout the briquette, the detergent composition in the interior of the briquette being in the form of adhering particles lightly compacted at a pressure between 3 and 100 pounds per square inch and readily reconvertible to discrete particulate state and the detergent composition at the

surface of the briquette being more homogeneous and continuous and containing inorganic salt more highly hydrated than that in the briquette interior, resulting from application of moisture to the surface of the lightly compacted shaped briquette in amount from ½ to 10% of the initial weight of the detergent composition particles to increase the structural strength and abrasion resistance of the briquette surface.

11. A shaped water soluble detergent briquette readily disintegrable and soluble in mildly agitated warm water and possessed of commercially acceptable surface strength comprising a spray dried, normally solid, water soluble, built synthetic detergent composition consisting essentially of a water soluble anionic synthetic organic detergent and a hydratable inorganic builder salt selected from the group consisting of sodium tripolyphosphate, tetrasodium pyrophosphate and sodium sulfate, the spray-dried detergent composition in the interior of the briquette being in the form of adhering particles lightly compacted at a pressure between 3 and 40 pounds per square inch and readily reconvertible to discrete particulate state and the detergent composition at the surface of the briquette being more homogeneous and continuous and containing fused detergent composition containing an inorganic builder salt selected from the group consisting of sodium tripolyphosphate, tetrasodium polyphosphate and sodium sulfate which is more highly hydrated than the same builder salt in the interior of the briquette due to application of moisture to the surface of the lightly compacted shaped briquette in amount from ½ to 5% of the initial weight of the detergent composition particles to increase the structural strength and abrasion resistance of the briquette surface.

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UNITED STATES PATENT OFFICE

CERTIFICATE OF CORRECTION

February 24, 1959

Patent No. 2,875,155

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It is hereby certified that error appears in the printed specification of the above numbered patent requiring correction and that the said Letters Patent should read as corrected below.

Column 5, line 49, for "were" read — made —.

Signed and sealed this 11th day of August 1959.

(SEAL)

Attest:

KARL H. AXLINE

Attesting Officer

ROBERT C. WATSON

Commissioner of Patents