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DETERGENT BRIQUETTE

James Douglas MacMahon, Niagara Falls, N. Y., assignor to The Mathieson Alkali Works, Inc., New York, N. Y., a corporation of Virginia

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This invention relates to improved briquetted detergent compositions and more particularly to detergent briquettes characterized by a unique combination of detergent characteristics and other physical and chemical properties which make them highly satisfactory for use in mechanical washing operations.

Modern mechanical methods and operations for washing dishes, milk cans and the like, particularly where the operation is continuous or prolonged, have presented the serious problem of maintaining an alkali concentration in the wash tanks between desirable and restricted limits. Commercial experience has shown that this may be accomplished in a dependable and virtually automatic manner by dissolving alkaline briquettes in suitable auxiliary equipment and dispensing the resulting solution into the wash tanks at a predetermined rate. A very considerable amount of research has been carried out in view of developing detergents having physical and chemical characteristics satisfactory for this purpose.

The problem presented involves, not merely the production of a material or mixture of materials having the desired detergent characteristics but also the development of a product which, in addition to meeting that requirement, can be economically produced in the desired physical form possessing other essential physical characteristics. For instance, it is desirable that the detergent be in briquette form; that the briquettes be sufficiently hard and strong to withstand ordinary handling; that the briquettes be chemically and physically stable and non-deliquescent so as to withstand storage and the necessary handling and of such structure as will not disintegrate under the conditions of use.

In as much as the control of the rate at which the alkali is dispensed into the washing operations largely depends upon the dissolving rate of the briquette, it is desirable that the briquette not only have a satisfactory degree of uniformity in its composition but also that it have a uniform solubility rate. It is, of course, also essential that the composition of the cleansing material be such as to avoid harmfully affecting the material being washed either by attacking the material or by forming a deposit or coating thereon. It is further essential that the composition of the detergent be such as to avoid deleteriously attacking the parts of the mechanical washer and the deposition of scale in the various chambers thereof.

It has been proposed to produce detergent briquettes for such use by fusing the detergent or detergent mixtures and casting the fused material by drawing it off into molds to cool. For example, briquettes have been produced by fusing mixtures of trisodium phosphate and soda ash. However, the relatively high temperature required

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to fuse the detergent or detergent mixture has been a decided handicap in the production of satisfactory detergent briquettes, as many substances, the presence of which is highly desirable in detergent mixtures, are driven off or decomposed at temperatures below their fusion point or at temperatures necessary for fusing other desirable constituents.

This temperature requirement has not permitted the incorporation in detergent mixtures so produced of many effective water conditioners and surface active agents such as synthetic detergent and wetting agents. Consequently, the use of such fused detergent briquettes has not been wholly satisfactory. For instance, particularly under adverse water conditions, their use has resulted in the precipitation of the natural hardness of the water supply and the tendency to form scale on the inner surfaces of the mechanical washers with which the detergent is used. Rapidity of this scale formation depends upon the degree and nature of the hardness of the water and in general increases with the concentration of the hardness of the water supply. If not periodically removed, this scale interferes with the normal functioning of the equipment. Further this precipitated hardness interferes to a greater or less extent with the cleansing operation.

The presence of a water conditioning polyphosphate, such as tetrasodium pyrophosphate, ($Na_4P_2O_7$), sodium tetraphosphate, ($Na_6P_4O_{13}$), sodium triphosphate, ($Na_5P_3O_{10}$), and sodium hexametaphosphate, ($Na_6P_6O_{18}$), in the alkaline solution tanks of the mechanical washers has been found to inhibit or greatly retard scale formation. Also, the addition of surface active agents, such as the non-saponaceous, organic, synthetic detergents, has been found further to enhance the cleansing action and to afford improved rinsing. However, for the reasons stated above, the incorporation of these materials in such anhydrous detergent briquettes formed by fusion at high temperatures, has been impractical.

Detergents have heretofore been produced in block form by crystallization or solidification of the detergent or detergent mixtures from aqueous solutions; for instance, by the evaporation of water therefrom or by causing a chemical or physical union of the water, or a portion thereof, with the detergent. The resulting blocks of detergent material have usually been reduced to a granular or powdered form before used. It has also been proposed to use such detergent blocks in detergent operations. However, so far as I am aware, the detergent blocks produced according to the prior literature of the art have fallen short of the requirements, essential to their satisfactory commercial use in mechanical washing operations.

I have previously discovered that detergent briquettes having desirable physical characteristics may be produced at moderate temperatures which permit incorporation therein of water conditioners and surface active agents, such as previously mentioned, by the use of various essential ingredients, including silicates, in proportions within certain rather definite ranges. However, in certain mechanical washing operations, and for other specialized purposes, the use of a detergent containing a silicate may be objectionable.

As distinguished from detergent briquettes of that general type containing a silicate as an essential ingredient, the briquette of my present invention contains no silicate. I have now discovered that by incorporating in a briquette of the type just described, a substantial proportion of a sodium polyphosphate, a briquette having the desired physical properties may be produced without the use of a silicate.

The essential ingredients of my improved detergent briquettes are trisodium phosphate or soda ash, a sodium polyphosphate, such as tetrasodium pyrophosphate, sodium tetraphosphate, sodium tripolyphosphate or sodium hexametaphosphate, and water. One or more of these polyphosphates may be incorporated in my improved product. Where only one polyphosphate is incorporated in the briquetted product, I prefer to use either the tripolyphosphate or the pyrophosphate, but frequently two or more of these polyphosphates may with advantage be incorporated in the product. Though neither sodium carbonate nor trisodium phosphate is an essential ingredient of my improved briquettes, one or the other must be used, and it is frequently advantageous to use both and both may be used where desired. Other detergent aids, for instance, surface active agents, may also be included.

The proportions of the several ingredients may be varied over a considerable range, as hereinafter specied, to meet specific detergent requirements, without the loss of the desirable physical or mechanical characteristics of the resultant briquettes. Though briquettes having highly satisfactory physical and chemical characteristics may be prepared in accordance with my present invention from the previously noted essential ingredients alone, as previously indicated, other desirable detergent aids may be incorporated in the briquettes without destroying the desirable physical properties of the briquettes or the detergent properties of such added materials.

Of the various polyphosphates noted, the use of tetrasodium pyrophosphate or sodium tripolyphosphate has been found particularly advantageous from the standpoint of imparting to the briquette optimum physical and molding characteristics.

An advantage of my present invention is that both trisodium phosphate and soda ash may be incorporated in the silicate-free briquette, if desired, or either may be entirely omitted.

In the preparation of my briquettes, the trisodium phosphate, when used, may be introduced in the form of the ordinary commercial hydrated product, generally represented by the formula $Na_3PO_4.12H_2O$. Theoretically, this material contains 56.8% water. However, the commercial product usually contains somewhat less water. Trisodium phosphate having a higher or lower water content may be used, providing appropriate compensation be made for the different proportions of water contained therein. Also material

which reacts under the compounding conditions to form trisodium phosphate, for instance, disodium phosphate and caustic soda, may be substituted for an equivalent proportion of the trisodium phosphate, appropriate allowance being made for the water content of such reacting materials and water produced by the reaction.

The sodium carbonate constituent of my briquettes, when used, may conveniently be supplied as anhydrous soda ash and the proportions and ranges of proportions specified herein are based on the use of sodium carbonate in this form. However, the sodium carbonate may be supplied in the form of a hydrate such as mono- or decahydrate, appropriate allowance being made for the differences in composition. Similarly the sodium carbonate may be supplied in the form of materials such as sodium bicarbonate and caustic soda which react under the compounding conditions to form sodium carbonate.

The proportions of the sodium carbonate and trisodium phosphate constituents are subject to a considerable latitude of variation to meet particular detergent requirements. Where either trisodium phosphate or soda ash is omitted, the other should be used in an amount not less than about one-tenth the formula weight. Similarly, where both are used, the amount should aggregate not less than about one-tenth the formula weight. The aggregate amount of these two materials may range as high as about one-half the formula weight, or either may be used, exclusive of the other, in this amount.

The amount of water present in the detergent composition is of major importance with respect to molding time and mechanical structure of the resulting briquettes. It is also of importance with respect to the active detergent content of he briquettes. I have found the permissible range of proportion of water in my present briquetted product to be from about 30% to about 50%. Within this range the proportion of water may be varied without encountering any destructive effect on the physical structure of the briquette. However, I have found that proportions of water approaching the lower limit of this range, say about 30%–35%, result in more desirable pouring and congealing and structural characteristics of the resultant briquettes. An increased molding time is generally encountered as the proportion of water approaches the upper limits of this range but this may, to a considerable extent, be corrected by using substantial proportions of trisodium phosphate in the upper portion of the prescribed range for that constituent.

In determining the quantity of water, if any, to be added, as such, in the compounding of my briquettes, due consideration must be given to the amount of water present in the various constituents, either as water of crystallization or otherwise, and the water forms by chemical reaction. Also a small amount of water may be vaporized or lost during the compounding of the detergent mixture. However, when compounded by the process herein described, the amount of water thus lost is usually of no particular consequence. If the amount of water thus lost is excessive, additional water may be added to the batch.

The polyphosphates may be used in commercially available anhydrous form, and the proportions and ranges of proportions, as specified herein, are based on substantially anhydrous materials.

The proportion of sodium polyphosphate may vary over a considerable range to meet particular

detergent requirements. However, the sodium polyphosphate is an essential ingredient of my present briquette and should be used in proportions not less than about 5% by weight.

It is permissible to use any of the polyphosphates just mentioned in amounts ranging up to about 15% by weight. However, where the polyphosphate used is tetrasodium pyrophosphate or sodium tripolyphosphate alone, or where a preponderance of either of the polyphosphates is used in conjunction with sodium tetraphosphate or sodium hexametaphosphate, the combined proportion of polyphosphate may range as high as about 50% by weight without objectionably affecting molding time or physical characteristics of the briquette. Under the latter conditions, the amount of sodium tetraphosphate or sodium hexametaphosphate should not exceed a total of about 15% of the formula weight.

Where the sodium tetraphosphate or sodium hexametaphosphate is used to the exclusion of the other polyphosphates, I have generally found it desirable to include in the briquette a substantial proportion of trisodium phosphate, say not less than about 15% by weight. Also, where trisodium phosphate is omitted, I have found it generally desirable to include a substantial proportion of either the pyrophosphates or the tripolyphosphates, say not less than about 15%.

An important consideration from a practical commercial and economic standpoint is the time required for the briquettes after pouring, to solidify sufficiently to permit their removal from the molds, in other words, the molding time of the briquettes. The molding time of briquettes prepared from the ingredients and within the range of proportions herein specified is a highly desirable aspect of my present invention. I have observed, however, that there is a tendency toward an increase in molding time under the following conditions:

(*a*) As the proportion of trisodium phosphate is decreased;

(*b*) As the proportion of tetrasodium pyrophosphate or sodium tripolyphosphate is decreased;

(*c*) As the water content of the briquettes is increased.

Generally, the proportion of water in the briquetted composition should be kept in the lower portion of the prescribed range, as under such conditions a maximum detergent content of the briquettes is permitted. Accordingly, it is generally preferred to use as low a proportion of water as is consistent with the obtaining of the desired pouring, molding and structural characteristics of the product.

The compounding of my improved detergent briquettes is advantageously effected generally in accordance with the process described in my copending application Serial No. 582,575, filed March 13, 1945.

As therein described, the compounding operation is advantageously carried out in a conventional steam-jacketed kettle equipped with a stirring device. Excellent results in preparing and in duplicating the composition and structure of the briquettes have been obtained by adhering to the following general procedure: Where trisodium phosphate is used, the trisodium phosphates and the predetermined required amount of water, if any, are first added to the kettle together with such caustic soda as may be desired, if any. This mixture is heated and agitated until the mixture is fluid, at which time the soda ash, if any, and the polyphosphate or polyphosphates are added and thoroughly incorporated in the mixture. The mixture is then drawn off or poured into molds. During the mixing and pouring, the mixture is maintained at a temperature below which substantial evolution of steam would occur with resultant material loss in water content. Higher temperatures should be avoided as it is desirable to reduce to a minimum, the amount of water lost during the compounding operation and to avoid decomposition of less stable ingredients. By minimizing the water loss during the compounding operation, the proportion of water in the product may be effectively controlled by regulation of the total amount of water added to the batch. The temperature to which the material is heated depends primarily on the concentration of the solution in the fluid mass but is usually found to be between about 70° C. and 100° C.

Where trisodium phosphate is omitted, it is advisable to heat the required amount of water up to a temperature approaching its boiling point before adding the soda ash.

Where a surface active agent, such as the previously noted synthetic detergents, is to be incorporated in the briquettes, it is usually desirable to add such material just prior to pouring.

After all of the desired ingredients have been added and thoroughly mixed, the mixture is drawn off into suitable molds and allowed to congeal until the briquette has developed sufficient mechanical strength to permit its removal from the mold. For the briquettes of my present invention, the maximum molding time generally varies from about 1 hour to several hours, and depending upon the composition of the mixture. On cooling, detergent compositions of this type seem to expand somewhat and this, combined with their tendency to adhere to metal surfaces, has previously presented serious difficulty in the molding of such detergent materials. By using flexible briquette molds, such as molds made from rubber or similar material, as described in the above referred to co-pending application, these difficulties are eliminated.

My invention will be further described and specifically illustrated by the following examples of proportions and ranges of proportions of the several constituents. It will be understood, however, that my invention is not limited to products having the composition illustrated. In each instance, the proportions given are percentages by weight and are on the anhydrous basis.

Example No.	Trisodium Phosphate	Sodium Carbonate	Tetrasodium Pyrophosphate	Total Water
1	55		15	30
2		45	20	35
3	15		50	35
4	10	50	5	35
5	25		25	50
6	14	26	30	30

In the foregoing tabulation, Example 1 is illustrative of the approximate maximum proportion of trisodium phosphate, Examples 1, 3 and 5 are further illustrative of a product from which the sodium carbonate constituent is entirely omitted. Example 2 is illustrative of a briquette containing no trisodium phosphate and approximately the maximum permissible amount of soda ash. Examples 3 and 4, respectively, are illustrative of the approximate maximum and minimum proportions of tetrasodium pyrophosphate

and Examples 5 and 6 are illustrative of the approximate maximum and minimum proportions of water, respectively.

These Examples 1 to 6, inclusive, are further illustrative of a particularly desirable aspect of my invention in accordance with which the polyphosphate used was tetrasodium pyrophosphate.

Additional illustrations of my invention in which polyphosphates, other than tetrasodium pyrophosphate, are used, and no soda ash employed, appear in the following tabulations. It will be understood that the proportions of the various ingredients are not limited to those illustrated and particularly that larger or smaller amounts of the polyphosphate may be used. However, the proportion of polyphosphate should be maintained within the range of about 5–50%.

Example No.	Trisodium Phosphate	Sodium Pyrophospnate	Sodium Tetra-Phosphate	Sodium Hexameta-Phosphate	Approx. Total Water
7	15	40	10		35
8	15	40		10	35
9	35	25	5		35
10	35	25		5	35

The following additional examples will serve to illustrate briquettes in which the tetraphosphate or hexametaphosphate was used alone.

Example No.	Trisodium Phosphate	Sodium Carbonate	Sodium Tetra-Phospnate	Sodium Hexameta-phosphate	Approx. Total Water
11	18.8	36.2	10		35
12	37.6	22.4		5	35

The foregoing examples are illustrative of the use of sodium tetraphosphate or sodium hexametaphosphate in conjunction with tetrasodium pyrophosphate. In these illustrations sodium tripolyphosphate may be substituted for the tetrasodium pyrophosphate, if desired. It is generally desirable, where sodium tetraphosphate or sodium hexametaphosphate is used, also to include a substantial proportion of either tetrasodium pyrophosphate or sodium tripolyphosphate in order to obtain optimum molding characteristics.

The use of sodium tripolyphosphate to the exclusion of other polyphosphates is illustrated by the following examples:

Example No.	Trisodium Phosphate	Soda Ash	Sodium Tripoly-Phosphate	Total Water
13	18.8	36.2	5	40
14	30	10	25	35
15	15		50	35

Although I have described my improved briquettes as particularly adapted for use in mechanical washers, it will be understood that they are generally useful where a detergent in briquette form is desired.

I claim:

1. A detergent briquette substantially free from a silicate, physically stable, hard, strong and nondeliquescent, consisting of a dense crystalline aggregate consisting essentially of the following constituents in proportion by weight within the respective indicated ranges: total water about 30–50%, at least one detergent of the group consisting of sodium carbonate and trisodium phosphate aggregating from about one-tenth to about one-half the total formula weight, and at least one polyphosphate of the group consisting of tetrasodium pyrophosphate, sodium tripolyphosphate, sodium tetraphosphate and sodium hexametaphosphate aggregating about 5–50%, but not exceeding about 15% of sodium tetraphosphate and sodium hexametaphosphate.

2. A detergent briquette substantially free from a silicate, physically stable, hard, strong and nondeliquescent, consisting of a dense crystalline aggregate consisting essentially of the following constitutents in proportion by weight within the following respective indicated ranges: water about 30–50%, at least one detergent of the group consisting of sodium carbonate and trisodium phosphate aggregating from about one-tenth to about one-half the total formula weight, and about 5–50% of sodium tripolyphosphate.

3. A detergent briquette substantially free from a silicate, physically stable, hard, strong and nondeliquescent, consisting of a dense crystalline aggregate consisting essentially of the following constituents in proportion by weight within the following respective indicated ranges: water about 30–50%, at least one detergent of the group consisting of sodium carbonate and trisodium phosphate aggregating from about one-tenth to about one-half of the total formula weight, and about 5–50% of tetrasodium pyrophosphate.

JAMES DOUGLAS MacMAHON.

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