Sodium Silicate: The Key Ingredient in Detergent Agglomeration

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ABSTRACT

The rise of sodium silicate as an agglomerating agent in detergent powders is discussed. Various ingredient ratios and agglomeration techniques/processes are reviewed as such parameters affect finished product characteristics.

AGGLOMERATION

Detergent agglomeration is a combined physical/chemical process in which dry raw materials are combined into a uniformly distributed particle by the spray application of a liquid sodium silicate binder. The process may require drying. When hydration of sodium tri-polyphosphate (STPP) and soda ash is included the process always requires conditioning or aging. Some basic requirements to produce a lump free uniform product are: liquid must be uniformly dispersed as fine discrete droplets, and solids must be suspended in space and in motion so that fresh material is continually exposed. The degree of liquid dispersion will depend on the rate of liquid addition and the rotational speed. The process is best accomplished with specialized equipment such as the O'Brien Agglomerator, Schugi Agglomerator and Patterson-Kelley Continuous Zig-Zag® Mixer. However, ribbon blenders, conical mixers, pug mills, disc pelletizers and rotary dryers also are used.

Due to the limitations on bulk density with the present agglomeration equipment, the process is best adaptable to the manufacturing of automatic dishwasher detergents (ADW) and concentrated heavy-duty laundry detergents (HDLD).

Agglomeration has been most widely used for the production of the denser (¼ cup) products. Agglomeration yields the proper density (35-45 lb/ft^3), the correct particle size range (-10 + 80 mesh) and in some cases enables the manufacturer to add high levels of nonionic surfactant (15-20%). If a lighter density product is desired, the manufacturer will have to use expensive light density raw materials and lower levels of liquid sodium silicate. If the total silicate level needs to be increased, Britesil® hydrous sodium silicate is recommended.

The consumer appeal of light density, large box HDLD products will be the major limiting factor in the magnitude of the switch toward agglomeration. Unless consumer appeal is changed or an economical method is developed to produce a spray-dried type detergent on an agglomerator, the market size for agglomerated products will remain small in comparison to spray dried products. In addition, because of the high investment in present spray driers, the switch to agglomeration would only occur through attrition or the need for additional capacity.

Agglomeration is of interest to spray dry manufacturers due to the significant energy savings. However, as stated, the agglomeration process is limited to the manufacturing of concentrated HDLD. Spray dryer manufacturers who desire products with bulk densities below 30 lb/ft^3 cannot at this time switch to agglomeration.

Agglomeration offers dry blenders of HDLD and ADW several advantages that include: reduced segragation of powders, ability to use different particle size raw materials, improved flow properties, less attrition of particles, reduced costs by utilizing liquid ingredients, ability to vary bulk densities and particle size of the finished product, homogeneity of finished product and continuous processing.

Schugi Agglomerator

The Schugi is a high intensity continuous agglomerator that allows addition of large amounts of liquid to a given formulation. Loadings of up to 50% water are not unusual. In certain applications it is very successful, such as hydrating STPP or soda ash. Schugi plant capacities start at 500 lb/hr and go to 50 ton/hr.

The unit can be stripped down quickly for cleaning, allowing much flexibility in changing from one formulation to another. Typically, a fluidbed dryer is added to the process to increase particle size, condition, increase bulk density if needed, and dry the granules.

Patterson-Kelley Zig-Zag®

The P-K Zig-Zag is a versatile unit that readily adapts to different formulations. It can produce particle sizes from 10 mesh downwards. Liquid addition is limited to ca. 25%, but once a product has been made and dried, it can be passed through the unit again for another addition of liquid.

Zig-Zag blenders can be supplied in capacities up to 150 ton/hr. Like the Schugi, the P-K needs some sort of drying and or conditioning after the granule is manufactured.

O'Brien Agglomerator

The O'Brien is by far the most popular unit among detergent makers. It was produced specifically for detergent agglomeration. The O'Brien's rotating drum/falling curtain action coupled with a very good conditioner gives a well formed granule that tends to the high side in bulk density. This lends to its wide acceptance in auto dish manufacture. Due to the configuration of the liquid addition, 20-25% liquid per pass through is maximum. Perhaps, as with the P-K, a double pass is necessary to add on the desired amount of liquid. The unit is difficult to clean, and so it usually is dedicated to one product.

PROPERTIES OF SILICATES

The common ingredient in all agglomerated detergents is the liquid sodium silicate binder. The key properties of sodium silicate that effect the properties of the finished product are: ratio, concentration and viscosity. Because these properties are interrelated, a balance must be maintained which will involve compromise.

A 2.4 ratio silicate is recommended to start based on several differing qualities. It provides a workable liquid with regards to viscosity and still contains high solids. This high viscosity as well as its high tack, gives an excellent agglomerating ability. At 2.4 ratio or higher, silicates release their moisture quickly upon drying which helps maintain chlorine stability, a key formulating parameter for ADW. If hydration of anhydrous raw materials is to take place during agglomerating, these ratios also give the best results. Some formulators use a combination of high and low ratios, which is done mainly to coat the outer portion of the granule with a high ratio to provide a good handling characteristic. If the desired product is not obtained with a

2.4 ratio, most people switch to 2.58 ratio with good results.

As the binder, sodium silicate is the key to forming an agglomerate from the dry detergent ingredients. The silicate concentration will have a major effect on the properties of the finished product. The maximum range of liquid addition in current agglomeration equipment is 25-35%. Bulk density, particle size, absorptivity, attrition, flowability and solubility of the finished product are all affected by the silicate concentration. Increasing silicate level gives a harder balled product. This reduces the loss due to attrition and aids in flowability. At the same time, the product will be less soluble. In addition, increasing silicate level will increase bulk density and the particle size of the agglomerate and reduce the amount of liquid (if used) surfactant the product can absorb. In the other direction, a product prepared to be highly soluble and absorptive, such as the new ¼ cup concentrate, will be more prone to attrition and will not flow as well.

In general, increasing silicate level will reduce loss due to attrition, aid flowability, increase bulk density, increase particle size, reduce liquid absorptivity, and reduce solubility. Decreasing silicate level gives the opposite effects. Clearly, a balance must be established.

Depending on the particular product characteristics desired and the equipment being used, it may be necessary to dilute and/or heat the silicate. This dilution or heating will lower the viscosity to 125-200 centipoises to allow easier distribution and wetting of the dry ingredients. Lowering the viscosity and/or solids to 40-45% will reduce the size of the liquid droplets which will reduce the particle size of the agglomerate. The use of heat, however, will speed drying and could change the capacity of the solids to absorb moisture. In the same manner, bulk density is also affected by the amount of dilution. For a given formula, if a full strength silicate is used, a higher bulk density will result versus a silicate that has been diluted. Diluting the silicate will add moisture to the product which may, depending on the total level of moisture, require additional drying with increased cost. In the same manner, heating of the silicate increases costs. Therefore, the combination of dilution and heating is commonly employed. The effects of temperature and dilution on viscosity are shown in Figures 1 and 2.

In addition to the above properties of silicate with regard to agglomeration, silicates have traditionally had a high value as detergent additives.

Solid silicates are added to agglomerated products for various reasons. In heavy duty laundry products the use of Britesil® hydrous silicate has several advantages. These products frequently are formulated with bulk densities in the low thirties. Britesil® can help maintain this mark easier than liquid silicate.

Britesil® hydrous silicate also produces a starting place for the granule to begin to take shape. By being readily soluble, its surface partially dissolves and becomes tacky. Other raw materials then attach and the granule has begun taking shape. One other important advantage to Britesil® is the increased absorptivity it provides. In the new quarter-cup concentrates, where there is a high amount of nonionic surfactant add-on, high absorptivity is a requirement. Britesil® provides this increase, even when liquid silicate is being used to agglomerate the product.

The amount of moisture to add is a critical factor in the agglomeration of detergents. Water is needed to obtain the particle growth, and is also taken up in the hydration of soda ash and STPP.

Soda ash forms hydrates with 1, 7 and 10 moles of water; theoretically it can take up 17%, 120% or 170% of its weight in water to form these hydrates. STPP forms a hexahydrate, and theoretically can take up 29% of its weight.

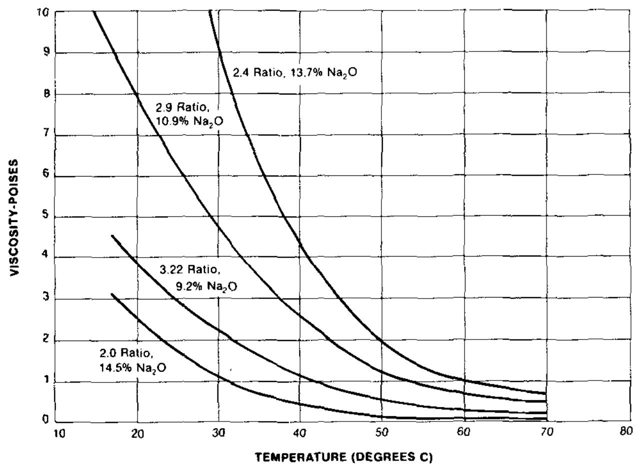

FIG. 1. Viscosities of sodium silicate solutions as a function of temperature.

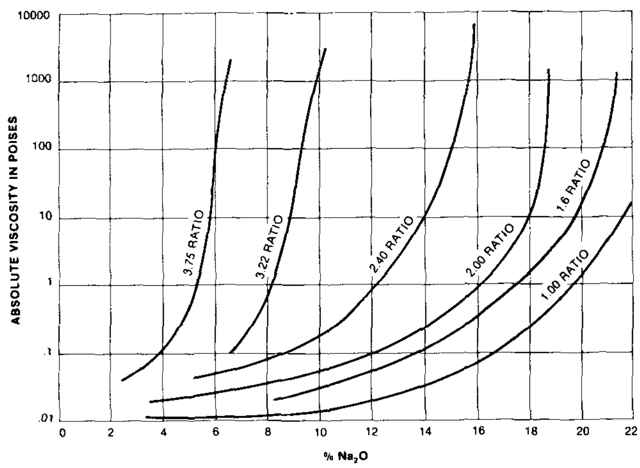

FIG. 2. Viscosities of sodium silicate solutions as a function of concentration.

This shows that, in general, a given weight of ash can take up more water than the same weight of STPP.

However, complete hydration is not necessary, and too much added water leads to the formation of oversize particle, caking and other undesirable results. Part of the added water goes for hydration, part for free water (before conditioning), and part remains with the silicate binder. The distribution conditions, and finding the right balance is the key to successful agglomeration. Hydration takes place rather slowly, and is completed in the conditioning step.

Since the agglomeration step tends to decrease the absorptivity of the raw materials, ingredients should be chosen which will give a net absorptivity somewhat higher than the desired value. This may require light soda ash or other finely divided builders. This introduces a new problem since these materials tend to be harder to agglomerate.

Usually absorptivities do not need to be higher than 15% add-on, for products conditioned and dried to ca. 20% H_2O.

As in absorptivity, the agglomeration step tends to change the bulk density of the aggregates, compared to that of the mix of dry ingredients. The raw materials should therefore be chosen to give a dry mix bulk density slightly below the desired final value.

The increase in bulk density is generally not a problem for ADW, but HDLD products are of lower density and higher surfactant absorptivity. Again, this may require the use of some light ash which is hard to agglomerate.

The use of Britesil® hydrous silicate is sometimes helpful to get lower bulk densities and higher absorptivities. As mentioned earlier, the Britesil® serves as seed to initiate the agglomeration process.

With few exceptions, silicates are compatible with most other detergent ingredients.

The use of sodium bicarbonate in an agglomerated detergent can lead to insolubles. The bicarbonate reacts with the silicate binder to insolubilize the silicate. The best solution is to reformulate the products, replacing the $NaHCO_3$ with sesquicarbonate or ash. If the bicarbonate is being used to reduce the alkalinity of a product, a reformulation with less caustic raw materials may be the solution.

The use of liquid silicates as agglomeration binders for ADW gives greatly improved stability of chlorinated isocyanurates. Loss of chlorine is caused by a reaction between the surfactant or water and the isocyanurate. In a lab study done at FMC, the best chlorine stability was obtained when the nonionic was absorbed by the STPP and soda ash, this mix was agglomerated with liquid silicate, and the chlorinated isocyanurate, Clearon® CDB, was added last. A small amount of silicate can be used to bind the Clearon® to the granules, to prevent separation. Apparently the silicate forms a coating which separates the surfactant and isocyanurate, and thus suppresses the reaction and chlorine loss. This type of mechanism is not available to dry blenders.

A troubleshooting guide is shown in Table I.

Any change in formulation or processing conditions will affect all properties of the detergent. If a change is made to correct one problem, it is necessary to consider how that will affect other characteristics.

TABLE I

Troubleshooting Guide

Causes	Problem
No agglomeration	Not enough moisture. Insufficient liquid dispersion. Silicate viscosity too high. Wrong silicate ratio. Too much light density ash. Too much mixing energy. Too high a temperature.
Too many oversize particles	Too much silicate. Too much water in raw materials. Poor dispersion. Raw material particle size too coarse. Poor mixing (overloaded equipment).
Insolubles	Overdrying. Silicate ratio too high. Wrong raw materials (i.e., $NaHCO_3$). Insufficient hydration.
Caking (poor flowability)	Too much moisture, overhydration. Too much surfactant. Particle size distribution too small. Insufficient conditioning.
Bulk density too high	Too much liquid silicate. Too much moisture. Wrong raw materials. Over handling, overconditioning.
Bulk density too low	Too little liquid silicate. Too little moisture. Wrong raw materials. Not enough agglomeration (not enough mechanical work).
Low attrition resistance	Not enough silicate. Not enough drying. Excess fines.
Low surfactant absorptivity	Too much moisture. Too much silicate. Wrong raw materials. Wrong surfactant. Too much mechanical work.
Poor product yield	See problem areas: "no agglomeration"; and "too many oversize particles."
Chlorine instability	Too much moisture. Wrong order of addition of ingredients. Wrong choice of bleach. Silicate ratio too high.