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FRIABLE CAUSTIC SODA AND METHOD OF PRODUCING SAME

John W. Koenders, Midland Mich., assignor to The Dow Chemical Company, Midland, Mich., a corporation of Michigan

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The present invention relates to improvements in the finishing of caustic soda for use in the arts and in particular to methods of treating a caustic soda finishing melt to modify the physical characteristics thereof exhibited after solidification by cooling, and to the more friable solid product so obtained.

Caustic soda is produced in large quantities by evaporation of a water solution thereof, during which, after evaporation of a portion of the water, salt is separated, if present. The final stage of evaporation is carried out usually in a large iron pot, the temperature being raised to approximately 870° F. to drive off the last of the water. It is customary, in some practices, in order to prepare a clarified finished product, to treat the concentrated soda with sulphur by throwing a few handfuls of sulphur into a pot which may contain around 14 tons of caustic soda. In case of inadvertent over-treatment with sulphur, a little sodium nitrate is added. The batch is allowed to cool somewhat and then discharged from the pot by ladling, pumping, or otherwise into iron drums or other container in which it solidifies. Such drums usually contain in the neighborhood of 700 pounds of product. The ladling or pumping temperature is preferably at, or just above, the freezing point, since solidification is then quicker in the drum and there is less liability to leakage therefrom. The temperature may be ascertained by a pyrometer, or pumping or ladling may be started when the molten caustic soda commences to freeze around the cooler edges of the pot. The cold solidified product is very hard and difficult to break up into pieces for use in the arts. It was accordingly noted that a friable grade of caustic soda more easily crushed than the usual product would find a preferential place in the market.

Among the objects of the invention is the production of a friable caustic soda product, cheaply and easily prepared, and unchanged adversely for uses in the arts, as by discoloration, in the softening process.

To the accomplishment of the foregoing and related ends, the invention, then, consists of the features and method hereinafter fully described and particularly pointed out in the claims, the following description setting forth in detail several approved combinations of ingredients and modes embodying my invention, such disclosed combinations and modes constituting, however, but several of the various ways in which my invention may be used.

A long investigation was undertaken directed to the discovery of a method of producing a soft caustic soda at will. Many attempts were made to soften a batch by adding oils, greases, waxes and the like to the pot without success, but it was finally discovered that very small quantities of certain oils could be put into the individual packages before filling with the molten caustic soda and that the effect of such oils was to induce a coarser, crystalline, friable structure, and to produce thereby a soft caustic soda. I find that such soft product may be produced both from batches which have not been treated with sulphur or nitrate and from those which have been treated with sulphur or which have been over-treated and subsequently corrected by the addition of a small quantity of nitrate. Large amounts of nitrate, for example, more than about four handfuls per 14 ton pot, have a negative action fully or partially preventing or neutralizing the softening action of the oil, but it is perfectly feasible to product soft caustic soda from such a batch which has been over-treated with sulphur and corrected with small amounts, say up to 4 handfuls, of nitrate.

Although many oils, greases and the like in varying amounts are effective in varying degrees, I have found that a mineral oil, particularly what is known as heavy motor oil, is so effective that half a teaspoonful placed in an empty drum will soften 700 lbs. of caustic soda satisfactorily. I have found further that a paraffin oil, such as the medicinal product known as "American oil", is effective, but to a somewhat less degree than the heavy motor oil, and that animal oils and vegetable oils are also effective; for example, whale oil or castor oil when placed in the drum in small amounts will produce soft caustic soda.

The following example illustrates the practice of my method and the product thereby obtained, but is not to be taken as limiting, being but one of many runs employing various oils successfully.

Electrolytic caustic soda previously concentrated to eliminate salt was boiled down in an iron kettle until it tested about 77 per cent Na_2O, NY & L, at which test it had the following approximate analysis:—

96.2	per	cent	NaOH	
.82	"	"	Na_2CO_3	
2.42	"	"	NaCl	
77.45	"	"	Na_2O	NY & L (75.04 per cent Na_2O, actual)

Such product constitutes one grade of substantially anhydrous caustic soda as produced in large quantities for general use in the arts. The

batch was treated in the usual way to clarify it and then allowed to cool to the neighborhood of 600 F., which is the approximate freezing point, whereupon it was pumped into sheet iron drums holding about 700 pounds of caustic soda each, into each of which, with the exception of a few, one-half teaspoonful of heavy motor oil had been previously introduced. After solidifying and further cooling, the caustic soda in the drums to which the oil had been added was found to be relatively soft and easily broken up with a sledge into pieces having a changed, distinctly crystalline fracture, whereas the caustic soda in those drums without oil was very hard, difficult to break up, and showed a rock-like fracture.

Other batches were treated with "American oil", whale oil, palm oil, petrolatum, stearic acid, oleic acid, castor oil, cod-liver oil, cup grease, paraffin, vaseline, light motor oil, and other substances, but it was found that more "American oil" or light motor oil was required than heavy motor oil. In some cases discoloration was more marked than in others, but the heavy motor oil gave the best result with a minimum amount of oil, using one-half teaspoonful, i. e. about 2.5 cc. per 700 pounds. "American oil" came next in effectiveness but slightly more thereof was required and the product showed some discoloration at the top of the drum. Whale oil and castor oil gave soft caustic soda which, however, showed with the castor oil a considerable discoloration and with the whale oil but slight discoloration. The heavy motor oil and "American oil" gave slight, if any, discoloration.

Further experiments involving the addition of the above named oils to the batch in the finishing pot did not produce soft caustic soda. Batches that had been inadvertently overtreated with sulphur and corrected with nitrate, when filled into iron drums containing a small amount of oil, as above described, gave a soft product if the nitrate was in small amount, but if over 4 handfuls, i. e. about 415 grams, were used in the pot of 14 to 15 tons capacity, the results were not so good. Batches which had not been treated with sulphur, when filled into drums containing a small amount of the oil, also gave a soft caustic soda product.

The invention accordingly provides a method of producing soft caustic soda from batches either with or without previous treatment with sulphur, including those which have been reoxidized with nitrate after over-treatment with sulphur. The product has a clean appearance upon breaking up, has a fibrous crystalline structure, is softer than the untreated material, much more easily broken or crushed and is preferred in the arts for certain uses. The treatment is inexpensive and does not unfit the product for its customary uses.

X-ray diffraction "patterns" have been taken of the ordinary caustic soda and of soft caustic soda prepared from the ordinary grade in accordance with my treatment.

These patterns indicate that both products have a crystalline structure, the difference being that the micro crystals of the soft product have a preferred orientation, possessing a "fibre axis", while those of the ordinary product do not exhibit orientation. The actual size of crystals was not determined nor the crystal system to which they correspond. The soft product fracture exhibits a long, somewhat fibre-like, or laminar structure, whereas the ordinary product exhibits an amorphous, or marble-like fracture.

A packaged soft, solid caustic soda may be produced as described by solidifying in iron drums in the presence of a small amount of an oil. If, however, it be desired, the solidification may take place in any way more convenient to the subsequent use or treatment. It may, for instance, be solidified in molds to form cakes, slabs, bars, etc., and the soft caustic soda thereby produced may be then crushed, broken or ground up, if desired. It may also be solidified upon a flaker to produce a softer grade of flake caustic soda. The invention is conceived, moreover, to cover not only a method of producing soft caustic soda by solidifying it in the presence of a small amount of an oil, but to cover also upon the product so obtained whether in the form of caustic soda solidified in a package, in a mold, on a flaker, or otherwise.

Other modes of applying the principle of my invention may be employed instead of those explained, change being made as regards the materials, step, or steps, employed, provided the materials, step, or steps, stated by any of the following claims or the equivalent of such materials, step, or steps, be employed.

I therefore particularly point out and distinctly claim as my invention:

1. Solid caustic soda having a fibrous crystalline structure the crystals of which exhibit a preferred orientation and possess a definite fibre axis as shown by an X-ray diffraction pattern, such caustic soda being characterized by being softer and more easily crushed than the ordinary product.

2. The method of producing a relatively soft and friable solid caustic soda which comprises charging molten caustic soda into a container to which has been added a relatively small amount of an oil and solidifying the molten material therein by cooling.

3. The method of producing a relatively soft and friable solid caustic soda which comprises charging molten caustic soda into a container to which has been added a relatively small amount of a mineral oil and solidifying the molten material therein by cooling.

4. The method of producing a relatively soft and friable solid caustic soda which comprises charging molten caustic soda into a container to which has been added a relatively small amount of paraffin oil and solidifying the molten material therein by cooling.

5. The method of producing a relatively soft and friable solid caustic soda which comprises charging molten caustic soda into a container to which has been added a relatively small amount of a heavy motor oil and solidifying the molten material therein by cooling.

6. In a method of filling molten caustic soda into a container, the step which consists in adding a relatively small amount of an oil to the container prior to introducing the molten caustic soda thereinto.

JOHN W. KOENDERS.