

Metallic Sodium Dispersions

Description

Metallic Sodium Dispersions are stable suspensions of sodium metal particles in an inert carrier such as an aromatic or an aliphatic hydrocarbon or a wax.

The particle size of the sodium may range from less than one micrometer to 1000 micrometers. Dispersing aids, such as aluminum stearate or oleic acid, are also incorporated in the formulation to prevent re-agglomeration of the sodium particles. Depending upon the sodium concentration and the type of vehicle used, the dispersion viscosity may range from a fluid liquid or, in the case of a wax vehicle, a solid.

Sodium dispersions are used in organic reactions to control reaction rate, increase reaction rate, lower reaction temperature, increase yield, decrease side reaction products or as a substitute for more expensive reagents. "Sodium is an extremely active reducing agent, and has proved useful in many other types of organic reactions such as condensations, polymerizations, isomerizations, metalations, and the preparation of metal alkyls and aryls...sodium is also an important reducing agent for 'winning' metals from their salts (titanium and potassium)..sodium reacts with hydrogen, carbon dioxide, sulfur dioxide and ammonia..." (U.S.I. Industrial Chemicals Co., literature - 1955)

Objective

The preparation of sodium dispersion, recommended by suppliers of metallic sodium, range from the use of blenders to gear pumps or high shear mixing equipment. These methods will prepare dispersions having a particle size of from one to 50 micrometers but with a random distribution of these sizes rather than a narrow size distribution. The use of Gaulin and Rannie equipment will enable the preparation of a dispersion having a smaller particle size than any of the normally used methods, with a much narrower spread in the range of sizes in the dispersion. With the correct selection of formulation and processing technique, the homogenizer can produce a dispersion with a size range of from 0.5 micrometers to 5 micrometers with most particles 2 or less micrometers. The colloid mill can produce a size range of from one to 10 micrometers. The use of this smaller particle size sodium dispersion provides for a faster reaction with improved yields on some of the more difficult organic syntheses.

Equipment and Processing

Both the colloid mill and homogenizer are used for sodium dispersions, depending upon the particle size desired and the quantity of dispersion. The colloid mill is a standard configuration except for "O" rings which must be Viton. Formulations will contain from 20 to 50% sodium metal (percentage depends upon the particle size desired in the final dispersion), 0.1 to 2.0% dispersing agent based on the total weight of dispersion with the balance being hydrocarbon or wax. The dispersing agent most commonly used is aluminum stearate. The dispersing agent is dissolved in the continuous phase fluid and the fluid is heated to 105° to 110° C.

Molten sodium is added to the dispersing agent and continuous phase liquid with agitation. The mixture is then passed through the colloid mill or the homogenizer. The clearance or gap of the mill may range from 0.002 to 0.030 inches. The homogenizing pressure may range from 1500 to 3000 psi.



WARNING: Sodium, in contact with water, is explosive and, therefore, water-cooling must not be used in the mill, nor should water lubrication on the plungers of the homogenizer be used. An oil-drip lubrication setup is recommended for the homogenizer, if this is found necessary. For short runs on the colloid mill, no cooling will be needed; however, for long continuous runs, an oil cooling system should be used.

The premix for the homogenizer must be more uniform and have a smaller particle size than that for the colloid mill. For small laboratory samples, it is possible to use the mill by recirculating the continuous phase and the dispersing agent through the mill back into the small hopper, at a close gap setting, until the temperature of 100 to 110° C is reached. At this point, the gap setting on the mill should be opened to a minimum of 0.020 inches and the molten sodium slowly added with a small agitator operating in the mill hopper. As soon as the sodium has been incorporated into the milled mixture, the gap setting is closed to obtain the desired particle size, and the dispersion is drawn off from the discharge of the mill.

For large-scale operation with the colloid mill, the dispersion would be prepared in a premix kettle and then pumped from the premix kettle through the colloid mill. With the continuous operation, circulation of oil through the cooling jacket of the mill is mandatory to protect the bearings and gears. Oil temperature should not exceed 90° C. The homogenizer will not be used unless dispersions in the one micrometer particle size range is required. The homogenizer is used, preferably, for large-scale production of dispersions. The initial premix is much more critical than in the case of the colloid mill, and the premix must be pump fed to the homogenizer. Again, no water must be allowed to come in contact with the sodium metal.

The clean up of either the mill or homogenizer is accomplished by first flushing with the same continuous phase liquid used in the preparation of the dispersion. Again, the dispersing agent in the liquid will aid in this flushing. This is followed by flushing with alcohol or steam. Steam will decompose the small particles of sodium left after the flushing operation sufficiently slowly so that explosions will not occur. Again, water must never be used.

Testing

Sodium dispersions are checked microscopically for particle size by diluting some of the sodium dispersion with the continuous phase liquid used in its preparation. Shelf life tests in glass bottles are used to observe visual separation or settling.



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