

1

2,912,379

FIRE EXTINGUISHING COMPOSITION

John H. McCracken, Madison, and Larry Jacobson, Painesville, Ohio, assignors to Diamond Alkali Company, Cleveland, Ohio, a corporation of Delaware

No Drawing. Application July 16, 1956
Serial No. 597,897

11 Claims. (Cl. 252-7)

This invention relates to a dry, pulverulent, free-flowing, chemical fire extinguishing composition containing an alkali metal bicarbonate as the principal fire extinguishing component, and more particularly relates to such a fire extinguishing composition including an alkaline earth metal carbonate dispersed therein, the surfaces of said particles of alkaline earth metal carbonate having a coating of an organic material deposited thereon.

It has heretofore been proposed to combine alkali metal bicarbonates, specifically sodium bicarbonate with other materials to form a dry, pulverulent, fire extinguishing composition, for expulsion from a container under pressure of a noncombustible gas. The various additives which have heretofore been proposed for specific purposes, such as imparting free-flowing characteristics to the composition and maintaining such characteristics over extended periods of time during storage and shipment, rendering the composition in general nonhygroscopic in order to prevent caking during storage and shipment, and in providing auxiliary fire extinguishing components to the composition, have failed to coact effectively in producing the desired combination of properties. In fact, one or more of such additives may effectively defeat one or more highly desirable properties of a fire extinguisher composition imparted by other additives. Also, compositions heretofore proposed have contained one or more ingredients which increase the bulk of the composition, i.e., increase the volume which a given weight of the material will occupy.

However, with the advent of more strict control of apparatus for employing dry fire extinguishing compositions, particularly by fire underwriters, the economic factor of providing a large volume with a minimum weight has been discarded in favor of the other more desirable properties of such compositions related directly to function as a fire extinguishing means.

Among such more desirable properties are those of controlled rate of discharge from the containing vessel under the force of the expelling gas, resistance to agglomeration, extremely low moisture adsorption under conditions of high humidity, and a high degree of water repellency in contact with water. While all of these properties are important in the aggregate, the flowability, or rate of expulsion of the composition from the fire extinguisher may be regarded as the most important, since if the composition possesses all of the other attributes of a suitable dry chemical fire extinguishing composition, and possesses undesirable flowability properties such composition may be regarded as wholly unsuitable, since its primary function is thereby seriously impaired.

One of the objects of the present invention is to provide a composition for dry chemical fire extinguisher use, which composition has a minimum of additives which coact effectively to produce a high degree of flowability, for example, characteristic of a mobile liquid, under conditions of storage and use, a higher bulk density than that of the principal fire extinguishing ingredient, e.g.,

2

sodium bicarbonate in a finely divided state, and which does not tend to agglomerate on standing even in an atmosphere of relatively high humidity nor tend to form a coherent mass even after extended periods of storage without agitation.

Another object of the invention is to provide a dry chemical fire extinguishing composition having a high degree of effectiveness under a wide variety of conditions of use from those encountered with small pressurized cylinders to those encountered in application from moving vehicles.

These and other objects will be apparent to those skilled in the art from the description of the invention set forth hereinafter.

It has now been discovered that an alkali metal bicarbonate in a relatively finely divided state acquires the desired degree of flowability for dry chemical fire extinguisher use when admixed with an alkaline earth metal carbonate, also in a relatively finely divided state, the surfaces of the particles of said alkaline earth metal carbonate having deposited thereon a coating of a fatty acid having more than eight carbon atoms, and that the addition of an alkaline earth metal salt of a fatty acid having more than eight carbon atoms, to this composition also increases the bulk density of the mixture without impairing the other desirable properties thereof.

Accordingly, to the accomplishment of the above-stated objects, the present invention contemplates a dry chemical fire extinguishing composition, the principal fire extinguishing component of which is an alkali metal bicarbonate in a relatively finely divided state, and dispersed therein an alkaline earth metal carbonate the surfaces of the particles of which have deposited thereon a coating of a fatty acid having more than eight carbon atoms. Further improvements contemplated by the present invention result from adding a relatively small amount of a water insoluble salt of an alkaline earth metal and a fatty acid having more than eight carbon atoms, to the combination of the alkali metal bicarbonate and alkaline earth carbonate, the particles of said alkaline earth metal carbonate having a surface coating of a fatty acid as indicated above.

The term alkali metal as used herein is intended to include the representative elements of group I of the periodic table, i.e., lithium, sodium, potassium, rubidium, and cesium, and the term alkaline earth metal as used herein is intended to include the representative elements of group II below beryllium in the periodic table, i.e., magnesium, calcium, strontium, and barium. It will be appreciated by those skilled in the art, however, that economic considerations dictate that the alkali metal bicarbonate preferably be sodium bicarbonate, and that the alkaline earth metal carbonates be chosen preferably from magnesium and calcium carbonates, as well as the water insoluble fatty acid salts of magnesium and calcium where applicable herein.

The particle size of the preferred sodium bicarbonate is suitably such that the amount retained on a U.S. Standard sieve No. 200, ranges from approximately 3.5% to 20%, and that the amount retained on a U.S. Standard sieve No. 325 ranges from approximately 6.5% to about 35%, with the amount passing through a U.S. Standard sieve No. 325 ranging from substantially 40% to about 90%. Sodium bicarbonate characterized by the above ranges of particle sizes, in combination with the other components of the compositions of the present invention, will insure highly satisfactory results in extinguishers wherein the bicarbonate composition is stored under pressure of a dry inert gas, for example, at about 250 lbs. per square inch, as well as compositions suitable for ap-

plication merely under the influence of the force of gravity, as from an airplane.

As to the alkaline earth metal carbonate to be employed in the compositions of the present invention, it is preferred that such alkaline earth metal carbonate, for example, calcium carbonate in addition to the fatty acid coating, have a particle size within the range of 0.5 to about 10 microns, and be relatively free of agglomerated masses. The fatty acid coating on the particles of the alkaline earth metal carbonate, may be applied, for example, by ball milling a mass of the alkaline earth metal carbonate with a suitable fatty acid until the oil absorption value becomes substantially constant. The oil absorption value is measured by taking samples, of a specified weight of the material, during the ball milling operation, combining this with small increments of a suitable vegetable oil until a uniformly coherent mass of the material is formed, and measuring the amount of oil required to achieve this result. When the amount of the semidrying oil absorbed remains at a constant minimum in any two successive samples, the coating of the fatty acid material on the alkaline earth metal carbonate is deemed to be substantially uniform.

The amount of the fatty acid material to be combined with the alkaline earth metal carbonate may range from 0.5 to 6% of the weight thereof; any amount substantially in excess of 6% is surplus and achieves little or nothing by way of coating the particles. Normally, it will be found that a suitable range for the amount of fatty acid coating on the alkaline earth metal carbonate is of the order of 1-2% thereof.

A further aspect of the composition of the present invention is the addition to the combination of alkali metal bicarbonate and fatty acid coated alkaline earth metal carbonate, of a water insoluble salt of an alkaline earth metal and a fatty acid having more than eight carbon atoms. A comparatively small amount of such material, i.e., from about 0.5% to 1% of the total composition being preferable for the purposes hereof, increases the bulk density of the composition noticeably, and contributes to the flowability characteristics of the composition whereby the loading of the composition into pressurized cylinders is substantially facilitated.

As to the qualities and characteristics of a dry chemical fire extinguishing composition, accepted as satisfactory by fire underwriters, the dry chemical charge is to be practically nontoxic under conditions of use, free-flowing within the temperature range from -40°C . to 60°C ., except that if there is any tendency to lump or cake, such lumps or cakes are to be friable when dropped from a height of 4 inches onto a smooth, hard surface, the tendency to lump or cake being measured, by specified tests carried out over an extended period of time, at a relative humidity of 80%; flowability, and operating characteristics of the composition, when employed in a pressurized cylinder type extinguisher, are indicated by measuring the distance from the nozzle within which the discharge chemical composition falls (initially a distance not less than 10 feet, and practically all of the discharged material falling beyond a point 5 feet from the nozzle); also not less than 85% of the exact rated capacity of the dry chemical is to be discharged when conditioned at all temperatures in the above-noted range, the duration of the effective discharge is not to be less than 8 seconds, and the weight of the discharge composition is not to be greater than 95% of the total amount discharged (as opposed to the rated capacity of the apparatus). In testing the effectiveness of the composition for fire extinguishing, dry bicarbonate extinguishing compositions are to be capable of extinguishing a gasoline fire in a tube under specified conditions, cotton waste saturated with gasoline, and gasoline spilled on a flat surface, as well as extinguishing fires in electrical equipment.

Factors which affect, to a certain extent, the properties of a given composition include the apparatus for

blending the ingredients, foreign materials which may be accidentally introduced into the composition by reason of the presence of such foreign substances in the blending equipment, the time during which the components of the composition are subjected to agitation to effect blending, as well as the amounts of the materials to be blended together.

For the sake of consistency in the data presented hereinbelow, the apparatus employed for mixing the components of the compositions consists of a mixer having a U-shaped chamber, and rotatable shaft mounted within the chamber, and two metallic ribbons extending spirally along, and spaced apart from the shaft, so as to present their narrowest surfaces substantially parallel to the curved portion of the U-shaped chamber, thereby providing their broad surfaces as lifters for the material being blended within the U-shaped chamber. The U-shaped chamber is 3 feet in length, 12 inches in diameter, measured on the curved portion of the U-shaped chamber, having a total capacity of 2 cubic feet; the shaft is rotated at a speed of 95 r.p.m. This apparatus is found to give maximum flowability properties, and uniformity, in the compositions of the present invention, when 100 lb. batches of such a composition are agitated therein for a period of about 10 minutes. When larger batches of material are used, or other equipment, such as a ball mill is employed, the optimum time for blending the components within such equipment is readily determined simply by experiment.

Accordingly, in order that those skilled in the art may better understand the present invention and in what manner the compositions thereof may be obtained, the following specific examples are offered:

Example I

Sodium bicarbonate in finely divided form, characterized in that 3.7% of the material is retained on a U.S. Standard No. 200 sieve, 27.7% of such material is retained on a U.S. Standard No. 325 sieve, and 68.5% of the material passes through a U.S. Standard No. 325 sieve is employed as a fire extinguisher composition without further additives. The results are shown under (1) in the table below.

Fire extinguishing compositions containing 99%, 97%, and 95% of further quantities of the same bicarbonate, in which there is dispersed 1%, 3% and 5% respectively of calcium carbonate, the particle size of which averages approximately 1 micron, and the surfaces of these particles have deposited thereon 1% of stearic acid, are obtained by blending the ingredients in the apparatus described above.

These compositions are each placed in fire extinguishers adapted for use with dry chemical extinguishing compositions under a pressure of 250 p.s.i. using dry nitrogen. Each composition is expelled from its containing vessel and observations made as to the flowability thereof.

The results of flowability tests are shown in the table below, that containing the 1% of the stearic acid coated calcium carbonate being designated (2), that containing 3% being designated (3), and that containing 5% being designated (4). A composition containing 97% of the same bicarbonate and 3% of calcium carbonate (average particle size 5 microns) without the stearic acid coating is designated (5).

	(1)	(2)	(3)	(4)	(5)
Quantity Discharged After 8 Seconds (Percent of the Amount Discharged).....	15.3	3.2	12.7	8.2	13.2
Quantity Discharged in 8 Seconds (Percent of the Total Discharged).....	84.7	96.8	87.3	91.8	86.8
Total Discharge Time in Seconds.....	11	12	12	10.5	12
Heel Left in Cylinder (Percent of Capacity).....	12.3	9.7	8.6	8.3	18.2
Total Quantity Discharged (Percent of Capacity).....	87.7	90.3	91.4	91.7	81.8

It will be noted that using this fine particle size sodium bicarbonate without additives (1), the minimum specifications for flowability, previously discussed, have been met. However, hygroscopicity and water repellency are such as to make the material wholly unsatisfactory for use in this type of fire extinguisher.

Further, it will be noted that the addition of the stearic acid-coated calcium carbonate to the sodium bicarbonate (2), (3), and (4), greatly improves the flowability characteristics of the bicarbonate as evidenced by consistently smaller amounts of residue (heel) remaining after discharge, and that using calcium carbonate without the stearic acid coating (5), provides a composition substantially less satisfactory than the bicarbonate alone (1).

Example II

Sodium bicarbonate characterized by having particles 23.4% of which are retained on a U.S. Standard sieve No. 200, and 33.2% are retained on a U.S. Standard sieve No. 325, with 40.4% passing through a U.S. Standard sieve No. 325, is combined with calcium carbonate having an average particle size of about 1 micron, and the particles of which are coated with 1% of stearic acid, are blended for a period of about 10 minutes.

The amount of the calcium carbonate is 3% of the total composition in (1) below and 5% in (2) below. The results of using these compositions in a fire extinguisher of the type described for Example I are set forth in the table below:

	(1)	(2)
Quantity Discharged After 8 Seconds (Percent of the Amount Discharged).....	8.0	21.0
Quantity Discharged in 8 Seconds (Percent of the Total Discharged).....	9.20	79.0
Total Discharge Time in Seconds.....	12	16
Heel Left in Cylinder (Percent of Capacity).....	10.9	14.2
Total Quantity Discharged (Percent of Capacity).....	89.1	85.8

It is seen from the above that with this much larger particle size sodium bicarbonate, the amount of coated calcium carbonate employed in the composition of the present invention is desirably not more than about 5%.

Example III

Sodium bicarbonate characterized by a particle size range of that shown in Example I, in the amount of 50% is combined with sodium bicarbonate characterized by a particle size range of that shown in Example II in the extent of 47%, and blended with 3% (by weight of the total composition) of calcium carbonate having an average particle size of the order of 1 micron, the particles of which are coated with 1% of stearic acid.

Another mixture of sodium carbonate characterized by the particle size range of that shown in Example I, in the amount of 75% is combined with the sodium carbonate having a particle size range of that shown in Example II in the amount of 22%, is blended calcium carbonate having an average particle size of about 1 micron, the particles of which are coated with 1% of stearic acid, in the amount of 3%.

These two compositions are blended together in the apparatus referred to hereinabove for a period of about 10 minutes after which they are charged to gas pressurized fire extinguishers of the type described in Example I with the results shown in the table below:

	(1)	(2)
Total Discharge Time in Seconds.....	9	12
Heel Left in Cylinder (Percent of Capacity).....	9.2	10.2
Total Quantity Discharged (Percent of Capacity).....	90.8	89.8

It will be noted that the flowability characteristics of these compositions, with regard to residue (heel) and

quantity discharged, are very nearly identical with (2), (3), and (4) of Example I.

Example IV

Sodium carbonate having a particle size range of that used in Example I in the amount of 50% is combined with sodium bicarbonate having a particle size range of that used in Example II, in the amount of 47%, and the combined material is divided into three equal batches.

(1) In the first batch, calcium carbonate having an average particle size of about 1 micron, the particles of which are coated with 1% of stearic acid by weight of the calcium carbonate, and magnesium stearate are added to the sodium bicarbonate, the amount of the calcium carbonate being 2.7% of the total composition, and the magnesium stearate being 0.3% of the total composition, are blended with the sodium bicarbonate for a period of 10 minutes.

(2) In the second batch, the amount of the calcium carbonate is decreased to 2% of the total composition, and the magnesium stearate is increased to 1% of the total composition.

(3) The amount of the calcium carbonate is increased to 2½%, and the amount of the magnesium stearate is decreased to 0.5%.

These three compositions are placed in pressurized dry fire extinguisher apparatus with the results shown in the table below:

	(1)	(2)	(3)
Total Discharge Time in Seconds.....	11	12	12
Heel Left in Cylinder (Percent of Capacity).....	6.4	8.8	10.7
Total Quantity Discharged (Percent of Capacity).....	93.6	91.2	89.3

The addition of the magnesium stearate increases the bulk density of the sodium bicarbonate from 54 lbs. per cubic foot to 72 lbs. per cubic foot where the bulk density of the material is measured without jarring the container to densify the material contained therein. Jarring the container to remove occluded gases increases the bulk density to 87 lbs. per cubic foot, an increase of about 20%, as compared with commercial composition which, under the same conditions, undergo a change in bulk density of from 24% to 28%.

All of the compositions disclosed in the above examples, except (1) and (5) in Example I, meet the specifications set forth hereinabove as to hygroscopicity, storage characteristics, friability of any lumps which form during storage, water absorption, and ability to extinguish the classes of fires noted above.

In addition, it will be noted that the compositions within the scope of the present invention are not only simple in their make-up, but also in the very simple manner in which the ingredients are combined. Moreover, the effect of the addition to the sodium bicarbonate of the alkaline earth metal carbonates, the particles of which have deposited upon their surfaces a comparatively small amount of fatty acid having more than eight carbon atoms, is in marked contrast to the concept of prior art teachings where the fatty acid material is combined with the mixture of sodium bicarbonate and calcium carbonate, and in excess of the amount of fatty acid-coated calcium carbonate employed in the compositions of the present invention.

While there have been described various embodiments of the invention, the methods and products described are not intended to be understood as limiting the scope of the invention as it is realized that changes therewithin are possible and it is further intended that each element recited in any of the following claims is to be understood as referring to all equivalent elements for accomplishing substantially the same results in substantially the same or equivalent manner, it being intended to cover the in-

vention broadly in whatever form its principle may be utilized.

What is claimed is:

1. The method of obtaining a dry, pulverulent, free-flowing fire extinguishing composition which includes combining with finely divided particles of an alkali metal bicarbonate, finely divided particles of an alkaline earth metal carbonate, the surfaces of which have a coating of a fatty acid of more than eight carbon atoms deposited thereon, said coating amounting to 0.5% to 6% of said alkaline earth metal carbonate, the coated particles of alkaline earth metal carbonate amounting to 0.5% to 5% of the weight of said alkali metal bicarbonate, and mixing said coated alkaline earth metal carbonate with said alkali metal bicarbonate until a substantially uniform dispersion of the coated alkaline earth metal carbonate particles in the alkali metal bicarbonate particles is obtained.

2. The method of claim 1 which also includes dispersing in said alkali metal bicarbonate particles from 0.5% to 1% of an alkaline earth metal salt of a fatty acid having more than eight carbon atoms, whereby the bulk density of the composition is increased.

3. The method of claim 1 wherein said alkali metal bicarbonate is sodium bicarbonate, said alkaline earth metal carbonate is calcium carbonate having an average particle size within the range of 0.5-10 microns, and said coating of fatty acid on said particles of calcium carbonate is stearic acid, said coating being applied by ball milling a mixture of stearic acid and calcium carbonate in the amounts indicated to a substantially constant minimum oil absorption value.

4. The method of claim 1 wherein the mass of said bicarbonate particles also has dispersed therein an alkaline earth metal salt of stearic acid.

5. The method of claim 2 wherein the alkaline earth metal salt of a fatty acid is magnesium stearate.

6. The method of claim 3 which also includes dispersing in said sodium bicarbonate particles from 0.5 to 1% of magnesium stearate whereby the bulk density of the composition is increased.

7. The method of claim 3 wherein the sodium bicar-

bonate employed is characterized by a U.S. Standard sieve analysis within the following ranges:

	Percent
Retained on a No. 100 sieve	0.1-3
Through a No. 100 sieve and retained on a No. 200 sieve	3-25
Through a No. 200 sieve and retained on a No. 325 sieve	25-35
Through a No. 325 sieve	40-70

8. The method of claim 3 wherein said bicarbonate also has dispersed therein between 0.5% and 1% of magnesium stearate based on the weight of the total composition.

9. The method of claim 6 wherein said calcium carbonate has an average particle size within the range of 0.2 to 5 microns.

10. A dry, pulverulent, free-flowing, fire extinguishing composition, containing an alkali metal bicarbonate as the principal fire extinguishing component, said bicarbonate having particles of an alkaline earth metal carbonate dispersed therein in amounts of 0.5% to 5% by weight of the total composition, the surfaces of said particles of alkaline earth metal carbonate having deposited thereon a coating of a fatty acid having more than eight carbon atoms, said coating amounting to 0.5% to 6% of the weight of the carbonate particles.

11. A dry, pulverulent, free-flowing, fire extinguishing composition containing sodium bicarbonate as the principal fire extinguishing component, said bicarbonate having particles of calcium carbonate uniformly dispersed therein in amounts of 0.5% to 5% by weight of the total composition, the surfaces of the particles of said calcium carbonate having deposited thereon a coating of stearic acid, said coating amounting to 0.5% to 6% of the weight of said carbonate particles.

References Cited in the file of this patent

UNITED STATES PATENTS

1,793,420	Block	Feb. 17, 1931
1,891,673	Gilleo	Dec. 20, 1932
2,776,942	Sylvester et al.	Jan. 8, 1957

UNITED STATES PATENT OFFICE
CERTIFICATE OF CORRECTION

Patent No. 2,912,379

November 10, 1959

John H. McCracken et al

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, Example II, in the Table, second column thereof, second item, for "9.20" read -- 92.0 --.

Signed and sealed this 26th day of April 1960.

(SEAL)

Attest:

KARL H. AXLINE
Attesting Officer

ROBERT C. WATSON
Commissioner of Patents

UNITED STATES PATENT OFFICE
CERTIFICATE OF CORRECTION

Patent No. 2,912,379

November 10, 1959

John H. McCracken et al

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, Example II, in the Table, second column thereof, second item, for "9.20" read ~~92.0~~ 92.0.

Signed and sealed this 26th day of April 1960.

(SEAL)

Attest:

KARL H. AXLINE
Attesting Officer

ROBERT C. WATSON
Commissioner of Patents