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1,158,328

NO DRAWINGS

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COMPLETE SPECIFICATION.

Filler Compositions for Gas Cylinders.

We, COYNE CYLINDER COMPANY, a Corporation organised under the laws of the State of California, United States of America, of 224 Ryan Way, South San Francisco, California, United States of America, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to a porous filler composition for containers adapted for storing gas, such as acetylene, and to a method for preparing such a composition and filling containers with the composition. The filler composition is characterized by high crushing strength together with uniformity of the finished material.

Some gases and particularly acetylene gas, are unstable at pressures in excess of 15 psig. In order to transport acetylene gas, for example, it must be dissolved under pressure in acetone and carried in cylinders of appropriate strength and construction. To prevent violent decomposition of the acetylene gas above the acetone level, the cylinder must be filled with a porous, absorbent material having minute individual pores. The acetone and acetylene gas are carried within these pores. To be effective the filler material must be substantially free of voids and have little or no space between the outer filler surface and the inside walls of the container. If this is not so, space for acetylene gas to collect separated from the acetone carrier would exist creating a decomposition hazard. Further, and at the same time, the filler must have adequate strength and physical resistance so that it does not crack, crumble or settle with normal handling of the acetlyene cylinder. Otherwise decomposition problems could be encountered for similar reasons.

Prior art fillers have been generally made using a cement or a lime-silica mixture as the bonding agent together with asbestos, charcoal and/or other materials to provide the requisite porosity, absorptive capacity, resilience and strengths. To prepare the filled cylinder such a mixture has been suspended in an aqueous slurry and thereafter poured into the cylinder in slurry form. Elevated temperatures and/or pressure are conventionally applied to the cylinders to harden and set the suspended mass. Water is then evaporated leaving a porous, uniform, monolithic filler inside the cylinder. As is understood, porosity of the filler is determined primarily by the quantity of water used in preparing the slurry and also, but to a lesser extent, by the proportion and types of suspended solids in the filler.

It is most important to filler uniformity that the wet slurries be easily flowable into the cylinder without entrapping air and forming voids or pockets within the filler material. It is known that voids can be minimized by applying a vacuum to the cylinder as it is filled and by vibrating the cylinder. Both operations tend to displace air pockets to the top of the cylinder and thence out of the cylinder.

Viscosity of the mix used in filling the cylinder has a great bearing upon the success with which voids and pockets of entrapped air can be avoided. The lower the mix viscosity the easier the filling operation becomes and the more complete is the separation of entrapped gases. On the other hand, as the mix viscosity is reduced, it becomes more difficult to maintain the mix solids in a homogeneous suspension with the result

[Price 4s. 6d.]

that too much settling occurs and parts of the finished filler composition do not perform as required.

Two approaches have been taken to arrive at a suitable filling viscosity with reduced settling problems. Where a blend of ground sand and diatomaceous earth such as herein utilized has been employed, it has been the practice to include suspending agents, such as aluminum sulfate or bentonite to the mix. While these materials may achieve the desired uniformity of composition, they do not contribute to the strength of the filler since they are not bonded into the lime blend matrix. Bentonite tends to positively weaken the structure of the filler. Moreover, gel type suspending agents may plug fittings of the cylinders and other equipment when the water content is driven off and the gel is dried out.

Another approach is to utilize a colloid mill for purposes of achieving a very fine average particle dimension. Where this technique is employed with a mix in which ground sand is used as the sole source of silica, a relatively high crushing strength and product may be achieved. Such a material prepared in this way can approach the requisite uniformity and lack of settling. However, a colloid mill is costly and in any event, unduly complicates the manufacturing process and necessarily increases the cost of the end product.

This invention provides a porous filler composition for gas cylinders, which composition consists essentially of asbestos, lime, and a blend of ground sand and diatomaceous earth in a ratio which provides optimum viscosity and crushing strength to the filler composition, the lime having been hydrated at temperatures of at least 180°F preparatory to combination with the other elements of the filler.

The composition may include charcoal, and the ground sand in the blend of ground sand and diatomaceous earth may be 50—90% by weight of the blend.

Alternatively, the composition may include charcoal, and the ground sand in the blend of ground sand and diatomaceous earth may be 60—80% by weight of the blend, the blend to lime ratio being 1.6—2.4 by weight, and the water to solids ratio of the composition being 3.5—3.8. Preferably, the water to solids ratio is 3.64, and the blend to lime ratio is 1.8—2.0.

A filler composition for gas cylinders may be prepared by hydrating lime by adding a first portion of the lime to water at a temperature of at least 180°F maintaining the water close to boiling while adding the balance of lime thereto, and mixing the hydrated lime with asbestos and a blend of ground sand and diatomaceous earth wherein the ratio of ground sand to diatomaceous earth is so selected to provide an optimum viscosity in the mixture for uniformly filling empty cylinders therewith and to provide an optimum crushing strength of the composition after hardening, the blend to lime ratio of the composition being 1.6—2.4 and the water to solids ratio being 3.5—3.8. Preferably the ground sand constitutes 50—90% by weight of the blend.

A filler composition for gas cylinders may also be prepared by hydrating lime at a temperature of at least 180°F, combining the hydrated lime with asbestos and a blend of ground sand and diatomaceous earth to form a slurried mixture, and indurating the slurried mixture, the ratio of ground sand to diatomaceous earth being so selected to provide an optimum viscosity in the mixture for uniformly filling empty cylinders therewith and to provide an optimum crushing strength of the composition after hardening.

A method in accordance with the invention for charging gas cylinders with a porous filler composition comprises hydrating lime by adding a first portion of the lime to water at a temperature of at least 180°F, maintaining the water close to boiling while adding the balance of lime thereto, mixing the hydrated lime with asbestos and a blend of ground sand and diatomaceous earth wherein the ratio of ground sand to diatomaceous earth is so selected to provide an optimum viscosity in the mixture for uniformly filling empty cylinders therewith and to provide an optimum crushing strength of the composition after hardening, the blend to lime ratio of the composition being 1.6—2.4 and the water to solids being 3.5—3.8, filling cylinders with said composition, and indurating the filler in the cylinders, the blend proportions and lime conditions serving to substantially prevent settling of the solids thereby yielding a uniform porous filler composition. The ground sand preferably constitutes 50—90% by weight of the blend. The hydrated lime may be mixed with charcoal, asbestos and the blend.

Still in accordance with this invention is a method for charging gas storage cylinders with a porous filler composition comprising a slurried mixture of lime, asbestos, and a blend of ground sand and diatomaceous earth wherein the ratio of ground sand to diatomaceous earth is so selected to provide an optimum viscosity in the mixture for uniformly filling empty cylinders therewith and to provide an optimum crushing strength of the composition after hardening, which method comprises hydrating said lime at a temperature at least 180°F, combining the hydrated lime with the blend and asbestos to form the slurried mixture to substantially reduce settling of solids in the filled cylinders, and indurating the slurried mixture. Preferably this method is one

wherein the filler composition has a silica to lime weight ratio of 1.5 to 2.0 and a water to solids weight ratio of 3.5 to 3.8, and wherein said lime is hydrated by adding a first portion of the lime to water at a temperature of at least 180°F and thereafter maintaining the water close to boiling while adding the balance of the lime thereto. The filler composition may advantageously include charcoal.

Because of the optimum viscosity, formation of air created voids or pockets within the filler is avoided and a high crushing strength in the end product is thereby made possible.

The new filler achieves a uniformity of composition and strength not exhibited by previously available products. These significant advantages are achieved without the addition of supplementary ingredients such as suspending or dispersion agents and without the necessity for using specialized techniques in the manufacturing process, such as passing the filler components through a colloid mill.

The use of a blend of ground sand and diatomaceous earth is only one reason for the beneficial nature of the end product. Thus, in addition, discovered that when the lime used in the end product is hydrated by its addition to water maintained at temperatures of at least 180°F and preferably maintained close to boiling throughout the balance of the addition step, a composition having exceptionally low solids settling and high uniformity can be obtained.

The blended sand and diatomaceous earth technique and the control of the water temperature used for the hydration of the lime each independently contribute to the desired goal of reducing the settling of solids in the filled cylinder end product.

The present invention achieves the lack of settling, uniformity of composition of the end product and significantly increased crushing strength while avoiding the necessity of any suspending agents or special equipment or techniques, such as the use of a colloid mill to grind the lime in the preparative process. The end product has all of the desired attributes and can be made at relatively low cost. As previously mentioned these attributes are provided by two modifications in the steps of preparing filler masses. In one aspect of this invention the lime hydrating water temperature is regulated. In the other aspect of the invention blending of a proportion of diatomaceous earth with ground silica is carried out so that a thickening action in the filler mix through absorption of free water in the pores of the diatomaceous earth occurs. In this way suspending agents are dispensed with. Control of the amount of diatomaceous earth blended with the ground sand lends sufficient fluidity to the mix for easily flowing into the cylinders with negligible air entrapment and void formation. At the same time, a sufficient proportion of ground sand is present to provide high crushing strength.

The improvement in filler crushing strength is achieved where the proportion of ground sand or silica is maintained reasonably high. As indicated above, preferably the blend of ground sand to diatomaceous earth or kieselguhr is such that the ground sand is 50—90% by weight of the blend. Although increased crushing strength is proportional to increased concentration of ground sand, it has been found that 100% ground sand is undesirable because the extent of settling and uniformity of the mix is unfavourably disturbed.

Thus, this invention contemplates the use of a blend of the two types of silica noted although over a wide range of proportions depending upon the precise end properties desired. Increasing the concentration of the low density, highly porous diatomaceous earth lowers viscosity while increasing the proportion of the high density non-porous ground sand increases crushing strength. Within the specific proportions above noted, optimum compositions are generally achieved. But in all cases a blend including an effective proportion of each of the ground sand and diatomaceous earth is critical to avoidance of the various stabilizing techniques previously considered necessary.

Heretofore no special criticality has been placed upon the temperature of the water use in the hydration with the result that relatively low temperatures on the order of 120°F have been used. It has now been discovered that the hydrating water to which the lime is added should be at least 180°F when the addition is commenced. As the lime addition proceeds, the temperature of the hydrating water should be maintained close to boiling while the balance of the lime in the formula is added thereto and hydrated. If these temperature limitations are observed, an excellent uniformity of product is achieved As noted the temperature of the lime hydration water has been discovered to be so influential upon the character of the end product that even in the absence of the blend discussed above its incorporation into the manufacturing process will lead to substantially improved products in terms of uniformity and absence of settling of the porous filler mass.

Apart from blending the ground sand and diatomaceous earth, absence of suspending agents and colloidal grinding, and the temperature adjustment in the lime hydration step, the invention is consistent with prior techniques as to ingredients employed, amounts and general preparative steps. The

charcoal can be considered as functioning to provide a swelling action with acetone which further tightens the filler within the cylinder shell.

As indicated above, the water to solids ratio is relatively critical and is preferably maintained at a ratio of 3.5 to 3.8 for proper porosity in the end product. A water to solids ratio of 3.64 appears to be optimum for many purposes. The silica to lime ratio is also important, and is preferably maintained between 1.6 to 2.4 as indicated above with excellent results achieved in the range of 1.8 to 2.0. Where these water to solids and blend to lime ratios are observed, the remaining components can be varied as desired. Thus, the asbestos content can be varied to obtain the desired flexibility in the end product, and as mentioned above, charcoal can be eliminated altogether.

To illustrate the present invention, a filler formula composed of the following elements was formulated.

	Weight percent
Asbestos	18.5
Charcoal	12.5
Blend	44.0
Quick lime	25.0
	100.0

In accordance with the invention, the blend content of the formula was prepared from ground sand (95% minus 200 mesh) and diatomaceous earth wherein the ground sand constituted 70% by weight of the two silicas.

A filler is prepared in accordance with the following steps. The charcoal is soaked under vacuum to saturate it with water prior to addition to the mix. Quick lime is added to water maintained over 180°F. Sufficient water is used in the hydrating of the lime to prepare a 14.5% hydrated lime suspension. As the lime is added to the water, the temperature of the water is maintained close to boiling. The presoaked charcoal, hydrated lime suspension and 25 more gallons of water are then combined with the blend and abestos. Total water used in the process is as follows:—

	Gallons	*Pounds*
Charcoal saturation	2	16
Quick lime water	18	150
Free Water	25	208
	45	374

Ratio of water to solids in the filler was 3.64.

The solids are added to the free water in the order shown and mixed for one-half hour before filling into cylinder shells. Filling of the cylinder shells is accomplished in conventional manner with vacuum and vibration as desired.

For comparison, another cylinder was filled with a composition under the procedure described above with the exception that instead of the blend, a composition in which the "blend" was 100% diatomaceous earth was used. After the filled cylinders were indurated, hardened, and set in conventional fashion the fillers were compared. The filler prepared from the blend of ground sand and diatomaceous earth had a smooth surface and a crushing strength of 425 psi. The filler prepared from 100% diatomaceous earth had a rough surface and a crushing strength of only 325 psi.

WHAT WE CLAIM IS:—

1. A porous filler composition for gas cylinders, which composition consists essentially of asbestos, lime and a blend of ground sand and diatomaceous earth in a ratio which provides optimum viscosity and crushing strength to the filler composition, the lime having been hydrated at temperatures of at least 180°F preparatory to combination with the other elements of the filler.

2. A composition according to claim 1 including charcoal, and in which the ground sand in the blend of ground sand and diatomaceous earth is 50—90% by weight of the blend.

3. A composition according to claim 1 including water and charcoal, and in which the ground sand in the blend of ground sand and diatomaceous earth is 60—80% by weight of the blend, the blend to lime ratio being 1.6—2.4 by weight, and the water to solids ratio of the composition being 3.5—3.8.

4. A composition according to claim 3 wherein the water to solids ratio is 3.64, and the blend to lime ratio is 1.8—2.0.

5. A method for preparing a filler composition for gas cylinders, which method comprises hydrating lime by adding a first portion of the lime to water at a temperature of at least 180°F, maintaining the water close to boiling while adding the balance of the lime thereto, and mixing the hydrated lime with asbestos and a blend of ground sand and diatomaceous earth wherein the ratio of ground sand to diatomaceous earth is so selected to provide an optimum viscosity in the mixture for uniformly filling empty cylinders therewith and to provide an optimum crushing strength of the composition after hardening, the blend to lime ratio of the composition being 1.6—2.4 and the water to solids ratio being 3.5—3.8.

6. A method according to claim 5 wherein the ground sand constitutes 50—90% by weight of the blend.

7. A method for preparing a porous filler composition for gas cylinders, which method

charcoal can be considered as functioning to provide a swelling action with acetone which further tightens the filler within the cylinder shell.

As indicated above, the water to solids ratio is relatively critical and is preferably maintained at a ratio of 3.5 to 3.8 for proper porosity in the end product. A water to solids ratio of 3.64 appears to be optimum for many purposes. The silica to lime ratio is also important, and is preferably maintained between 1.6 to 2.4 as indicated above with excellent results achieved in the range of 1.8 to 2.0. Where these water to solids and blend to lime ratios are observed, the remaining components can be varied as desired. Thus, the asbestos content can be varied to obtain the desired flexibility in the end product, and as mentioned above, charcoal can be eliminated altogether.

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2. A composition according to claim 1 including charcoal, and in which the ground sand in the blend of ground sand and diatomaceous earth is 50—90% by weight of the blend.

3. A composition according to claim 1 including water and charcoal, and in which the ground sand in the blend of ground sand and diatomaceous earth is 60—80% by weight of the blend, the blend to lime ratio being 1.6—2.4 by weight, and the water to solids ratio of the composition being 3.5—3.8.

4. A composition according to claim 3 wherein the water to solids ratio is 3.64, and the blend to lime ratio is 1.8—2.0.

5. A method for preparing a filler composition for gas cylinders, which method comprises hydrating lime by adding a first portion of the lime to water at a temperature of at least 180°F, maintaining the water close to boiling while adding the balance of the lime thereto, and mixing the hydrated lime with asbestos and a blend of ground sand and diatomaceous earth wherein the ratio of ground sand to diatomaceous earth is so selected to provide an optimum viscosity in the mixture for uniformly filling empty cylinders therewith and to provide an optimum crushing strength of the composition after hardening, the blend to lime ratio of the composition being 1.6—2.4 and the water to solids ratio being 3.5—3.8.

6. A method according to claim 5 wherein the ground sand constitutes 50—90% by weight of the blend.

7. A method for preparing a porous filler composition for gas cylinders, which method

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comprises hydrating lime at a temperature of at least 180°F, combining the hydrated lime with asbestos and a blend of ground sand and diatomaceous earth to form a slurried mixture, and indurating the slurried mixture, the ratio of ground sand to diatomaceous earth being so selected to provide an optimum viscosity in the mixture for uniformly filling empty cylinders therewith and to provide an optimum crushing strength of the composition after hardening.

8. A method for charging gas storage cylinders with a porous filler composition, which method comprises hydrating lime by adding a first portion of the lime to water at a temperature of at least 180°F, maintaining the water close to boiling while adding the balance of lime thereto, mixing the hydrated lime with asbestos and a blend of ground sand and diatomaceous earth wherein the ratio of ground sand to diatomaceous earth is so selected to provide an optimum viscosity in the mixture for uniformly filling empty cylinders therewith and to provide an optimum crushing strength of the composition after hardening, the blend to lime ratio of the composition being 1.6—2.4 and the water to solids ratio being 3.5—3.8, filling cylinders with said composition, and indurating the filler in the cylinders, the blend proportions and lime hydrating conditions serving to substantially prevent settling of the solids thereby yielding a uniform porous filler composition.

9. A method according to claim 8 wherein the ground sand constitutes 50—90% by weight of the blend.

10. A method according to claim 8 or claim 9 wherein the hydrated lime is mixed with charcoal, asbestos and the blend.

11. A method for charging gas storage cylinders with a filler composition comprising a slurried mixture of lime, asbestos, and a blend of ground sand and diatomaceous earth wherein the ratio of ground sand to diatomaceous earth is so selected to provide an optimum viscosity in the mixture for uniformly filling empty cylinders therewith and to provide an optimum crushing strength of the composition after hardening, which method comprises hydrating said lime at a temperature at least 180°F, combining the hydrated lime with the blend and asbestos to form the slurried mixture to substantially reduce settling of solids in the filled cylinders, and indurating the slurried mixture.

12. A method according to claim 11 wherein the filler composition has a silica lime weight ratio of 1.5 to 2.0 and a water to solids weight ratio of 3.5 to 3.8, and wherein said lime is hydrated by adding a first portion of the lime to water at a temperature of at least 180°F and thereafter maintaining the water close to boiling while adding the balance of the lime thereto.

13. A method according to claim 11 or claim 12 wherein the filler composition includes charcoal.

14. A porous filler composition according to claim 1 substantially as herein described.

15. A method according to claim 5 for preparing a filler composition for gas cylinders substantially as herein described.

16. A method according to claim 7 for preparing a porous filler composition for gas cylinders substantially as herein described.

17. A method according to claim 8 substantially as herein described.

18. A method according to claim 11 substantially as herein described.

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