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METHODS OF PREPARING ABSORBENTS FOR LIQUID-OXYGEN EXPLOSIVES AND
RESULTING ABSORBENTS AND EXPLOSIVES

No Drawing.

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My present invention relates to methods of preparing absorbents for liquid oxygen for use in explosives, and to the resulting absorbents and explosives, and aims to devise methods of the general character specified which are simple, easy and economical to practice, which take into proper account the properties of the materials being handled and the desired properties of the resulting absorbents and explosives, and which yield absorbents and explosives having superior characteristics for the intended purpose, and being also characterized by their relative cheapness, their high degree of absorption for liquid oxygen, their high degree of retention of liquid oxygen, and the stiffness and long life of the cartridge, as well as other qualities which will in part be pointed out hereinafter and will in part be obvious to those skilled in the art to which the present invention relates, the resulting explosives and cartridges being characterized by equally desirable characteristics.

In the accompanying specification I shall described an illustrative embodiment of the method of the present invention. In the accompanying specification I shall also describe illustrative embodiments of the absorbents for liquid oxygen, the resulting liquid oxygen explosives, and the final liquid oxygen explosive cartridges of the present invention, particularly those resulting from the practice of the aforesaid illustrative embodiment of the method of the present invention. It is, however, to be clearly understood that my invention is not limited to the specific forms thereof herein described for purpose of illustration only. It is also to be understood that the aforesaid illustrative embodiment of the process of the present invention is not limited in its practice to the production of the aforesaid illustrative embodiments of the products of the present invention, nor are the aforesaid illustrative embodiments of the products of the present invention limited to their production by the aforesaid illustrative embodiment of the method of the present invention.

Referring now to the aforesaid illustrative

embodiment of the method of the present invention, I take, first, a relatively coarse material, preferably one in granular form, and preferably one having by itself a degree of absorption for liquid oxygen at least equal to about 2.6 times its weight. Since this is the theoretically necessary quantity of oxygen required to consume a given quantity of carbon, and since allowance should be made for the loss of liquid oxygen by evaporation or otherwise, I prefer that the relatively coarse, such as granular, absorbent for liquid oxygen, generally in the form of a carbonaceous absorbent having a high carbon content, should have a degree of absorption for liquid oxygen equal to from about 3.5 to about 4 times its weight.

I have discovered that a granular material ideally suited for the purposes of the present invention is leached carbonized lignin residues derived by first carbonizing and then leaching liquors rich in lignin derived from the manufacture of wood pulp by the alkali (soda) process, especially where wood pulp is derived from woods, such as spruce, pine and the like, rich in resins or other forms of lignin. Such liquors are particularly desirable, since they yield a high purity carbon characterized by high density and great absorptiveness and retentiveness for liquid oxygen, as hereinafter described in considerable detail.

These liquors are now carbonized, preferably with controlled access of air or even with the total exclusion of air. This is done in order to prevent too great combustion and consequent loss of carbon values in the final material. Preferably such carbonization takes place in retorts which are sealed against the ingress of air or to which air is admitted in very small amounts only to carry off by combustion or otherwise more volatile carbonaceous materials to thus leave a very open or porous carbonized lignin residue in the retort.

The carbonized lignin residues thus obtained will be found to be in a granular condition, the grains of which vary in size to a considerable degree and are quite uneven in size. The material also will be found to con-

tain in the pores thereof a considerable amount, often as high as ten (10) per cent., of alkaline carbonates, particularly sodium carbonate, where the pulp has been made by the soda process, together with other mineral substances. The carbonized lignin residues thus obtained are now thoroughly leached, first with water, to remove water-soluble mineral substances, and thereafter, if desired, although this step may be omitted, with acidulated water, such as water acidulated with about one-half to one per cent. of hydrochloric acid, to remove acid-soluble mineral substances. The purpose of this treatment or treatments is to reduce to a minimum the mineral constituents of the carbon so as to obtain a carbon characterized by maximum combustibility.

I now take the leached carbonized lignin residues, whether or not the same have been previously subjected to the acid treatment specified above, and submit the same to a slight grinding action to reduce the grains to a more or less uniform size so that the largest grains are not more than about one-quarter of an inch in their longest dimension and so that not more than about twenty-five per cent. of the material will pass through a one hundred mesh screen. Care should be taken during this slight grinding action, the purpose of which is to obtain a more uniform, even-sized material which is very suitable for use in smaller sized cartridges, to see that the material is not too finely ground, which would convert it from a highly absorptive material for liquid oxygen, suitable for the purposes of the present invention, into a material characterized by considerable decolorizing power but a low degree of absorptiveness for liquid oxygen, which would make the material unfit for the purposes of the present invention.

I have found that in general the density of the material when poured as freely into a container as it would be into the cartridge wrapper, should be not less than about 0.20. I have also found that the degree of absorptiveness of the material for liquid oxygen should be not less than about 2.6 times its own weight, and preferably about 3.0 times its own weight, or even over, so as to be suitable for use under actual field conditions and to take care of any liquid oxygen that might be evaporated up to the time that the cartridge is actually exploded.

It may here be stated, that by carefully controlling the carbonization of the lignin liquors or residues in the retorts, grains of the proper size may be and often are obtained without any grinding action whatsoever. In any event, the grinding action should be carefully controlled and regulated to yield a carbon characterized by a high degree of absorptiveness and a comparatively low degree of decolorizing power, rather than a low degree

of absorptiveness and a high degree of decolorizing power. I have found that the properties of absorptiveness and decolorizing power vary generally in approximately inverse proportions, absorptiveness depending on a comparatively large grain and being characterized by a comparatively low degree of decolorizing power, decolorizing power being apparently dependent upon small grains of a low degree of absorptiveness.

I now fill the external voids between the granules or other relatively coarse particles of the leached carbonized lignin residues or equivalent liquid oxygen absorbent with the relatively fine particles of a finely divided liquid oxygen absorbent. I prefer for this purpose that the relatively fine particles of finely divided liquid oxygen absorbent have by itself the property of absorbing at least 2.6 times its weight of liquid oxygen, this being the theoretically required amount of liquid oxygen to consume a given amount of carbon, since in the case of the relatively fine liquid oxygen absorbent, as in the case of the relatively coarse liquid oxygen absorbent, the first mentioned absorbent consists of carbonaceous material containing a very high percentage of carbon.

I have discovered that carbon black serves the desired purpose of the present invention ideally and satisfies the conditions as to liquid oxygen absorption already referred to above. In fact, this material absorbs from about 3.5 to about 4 times its weight in liquid oxygen, a very desirable property since it takes care of the liquid oxygen which is often lost through evaporation or otherwise. These two absorbents may be mixed, if desired, in the cartridges themselves. That is, the cartridge blank may first be packed with the granular material, after which the finely divided absorbent may be added by being poured in to fill up the external voids between the coarser particles of absorbent.

While the coarse and fine absorbents may be mixed in various proportions by weight or by volume, depending upon the percentage of voids between the coarser particles of absorbent material, and depending also upon the densities of the coarse and fine absorbent materials, I prefer generally to use a lesser quantity of the finely divided absorbent material and a larger quantity of the coarser absorbent material. For example, I have discovered that a cartridge embodying 60% of the granular leached carbonized lignin residues and 40% of the carbon black is very economical and remarkably efficient.

The material obtained as above described may now be packed into suitable cartridge containers. I prefer to use a container which is completely and readily combustible and which has proper insulating properties to prevent too ready absorption of heat with consequent evaporation of the liquid oxygen.

For this purpose I prefer to use a cartridge container which has a cotton fibre base, such as certain red fibre papers which may be readily obtained in the open market. This paper may readily be formed into containers of the desired size which are then packed with the granular leached carbonized lignin residues obtained as above described, after which the liquid oxygen is added, the cartridge sealed, stored and finally put in place, and exploded by a percussion cap or otherwise.

It may here be stated that whereas the density of the granular leached carbonized lignin residues, as already indicated above, is about 0.20, the density of the carbon black when poured freely into a container is generally about 0.15. It may further be stated that the density of the complex mass of absorbent materials, for example, where such complex mass consists of 60% its weight of granular leached carbonized lignin residues and 40% its weight of carbon black, is about 0.27. It may here further be stated that the degree of absorption of the complex mass for liquid oxygen should not be less than about 2.6 times its weight of liquid oxygen; the degree of absorption, for reasons already indicated above, being preferably from about 3.5 to about 4 times its weight. It is to be pointed out here that the complex absorbent mass obtained as above described has this latter desirably high degree of absorption for liquid oxygen.

The material obtained as above described is characterized by its unusually high density, its remarkable uniformity, its unusually high degree of absorption for liquid oxygen, and its high degree of retention of liquid oxygen. These properties ideally suit it for use as an absorbent for liquid oxygen and ideally suit the resulting explosives and cartridges for eminently satisfactory use under actual field conditions for blasting and similar purposes. Tests in the field indicate that this material is not only highly satisfactory for the indicated purposes, but is also very cheap and very easy to prepare.

The process described above is very simple to practice, making use for one of its ingredients of an otherwise waste material, and requires simple apparatus and few manipulations for its successful practice.

The absorbent itself possesses the many advantageous characteristics already specified above. In addition thereto, the use of the absorbent materials of the present invention yields a comparatively stiff cartridge which may therefore be more readily and safely handled. Finally, the combination of carbon black with the granular leached carbonized lignin residues is particularly valuable since the carbon black acts to counteract, to some extent at least, the naturally hygroscopic character of the granular leached car-

bonized lignin residues, such hygroscopic character otherwise tending to cause this absorbent to absorb a considerable amount of moisture which would tend to lessen the degree of absorption of the material for liquid oxygen and render it less efficient than it otherwise might be. The carbon black, not being hygroscopic, counteracts this tendency of the granular leached carbonized lignin residues.

Finally, the explosive itself and the cartridge embodying the same are characterized by the ease and safety with which they may be prepared, transported and kept, their long life in storage, and their great efficiency in use under actual field conditions. Other advantages of the methods and products of the present invention will readily occur to those skilled in the art to which the same relates.

It may here be stated that lamp black may be used with almost equal success to carbon black for the finely divided absorbent for liquid oxygen. It may here also be stated that for the larger cartridges I prefer to use cotton cloth bags instead of red fibre papers or their equivalents. Finally, it may here be stated that instead of mixing the coarse and fine carbonaceous absorbents for liquid oxygen in the containers themselves, I prefer to mix these materials in considerable quantity outside of the cartridge containers and thereafter put a predetermined amount of the mixed materials into the cartridge containers.

What I claim as my invention is:

1. The method of preparing a liquid oxygen absorbent, which comprises filling the external voids between the relatively coarse particles of a mass of liquid oxygen absorbent material, comprising leached carbonized lignin residues, with relatively fine particles of a liquid oxygen absorbent material to form a complex liquid oxygen absorbent material.

2. The method of preparing a liquid oxygen absorbent, which comprises filling the external voids between the relatively coarse particles of a mass of liquid oxygen absorbent material, comprising leached carbonized lignin residues, with relatively fine particles of a liquid oxygen absorbent material, comprising carbon black, to form a complex liquid oxygen absorbent material.

3. The method of preparing a liquid oxygen absorbent, which comprises filling the external voids between the relatively coarse particles of a mass of liquid oxygen absorbent material, comprising leached carbonized lignin residues, with a lesser proportion, by weight, of relatively fine particles of a liquid oxygen absorbent material, comprising carbon black, to form a complex liquid oxygen absorbent material.

4. The method of preparing a liquid oxygen absorbent, which comprises filling the external voids between the relatively coarse particles of a granular mass of liquid oxygen ab-

sorbent material, comprising leached carbonized lignin residues, with a lesser proportion, by weight, of relatively fine particles of a liquid oxygen absorbent material to form a complex liquid oxygen absorbent material.

5 5. An absorbent for liquid oxygen for use in explosives, comprising a complex body embodying a relatively coarse absorbent for liquid oxygen comprising leached carbonized
10 lignin residues having the external voids between the particles thereof filled with particles of relatively fine absorbent material for liquid oxygen.

6. An absorbent for liquid oxygen for use
15 in explosives, comprising a complex body embodying a relatively coarse granular absorbent for liquid oxygen comprising leached carbonized lignin residues having the external voids between the particles thereof filled with
20 particles of relatively fine absorbent material for liquid oxygen comprising carbon black.

7. An absorbent for liquid oxygen for use in explosives, comprising a complex body embodying a relatively coarse absorbent for liquid
25 oxygen comprising leached carbonized lignin residues having the external voids between the particles thereof filled with particles of relatively fine absorbent material for
30 liquid oxygen comprising carbon black.

8. A liquid oxygen absorbent comprising a complex body embodying a relatively coarse absorbent for liquid oxygen comprising
35 leached carbonized lignin residues having the external voids between the particles thereof filled with particles of relatively fine absorbent material for liquid oxygen comprising carbon black, said complex body having a degree of absorption for liquid oxygen equal
40 to at least about 2.6 times the weight of such body.

9. A liquid oxygen absorbent comprising a complex body embodying a relatively coarse granular absorbent for liquid oxygen comprising
45 leached carbonized lignin residues having the external voids between the particles thereof filled with particles of relatively fine absorbent material for liquid oxygen, said complex body having a degree of absorption for liquid oxygen equal to at least about
50 2.6 times the weight of such body.

In testimony, whereof, I have signed my name to this specification this 28th day of August, 1925.

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