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PROCESS OF MANUFACTURING POROUS BODIES, DIAPHRAGMS,
FILTERS, AND THE LIKE OF EBONITE
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Fig. I.

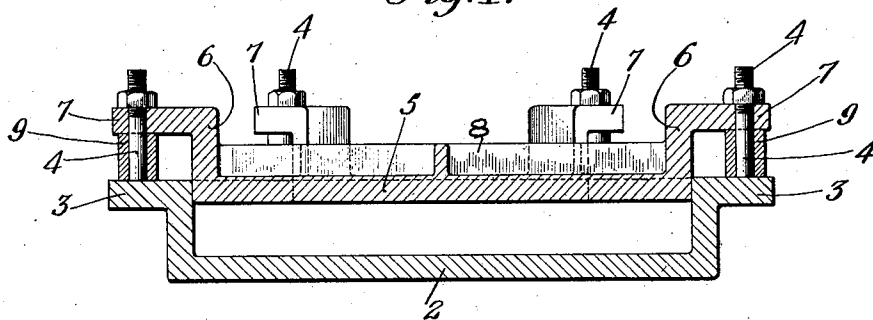
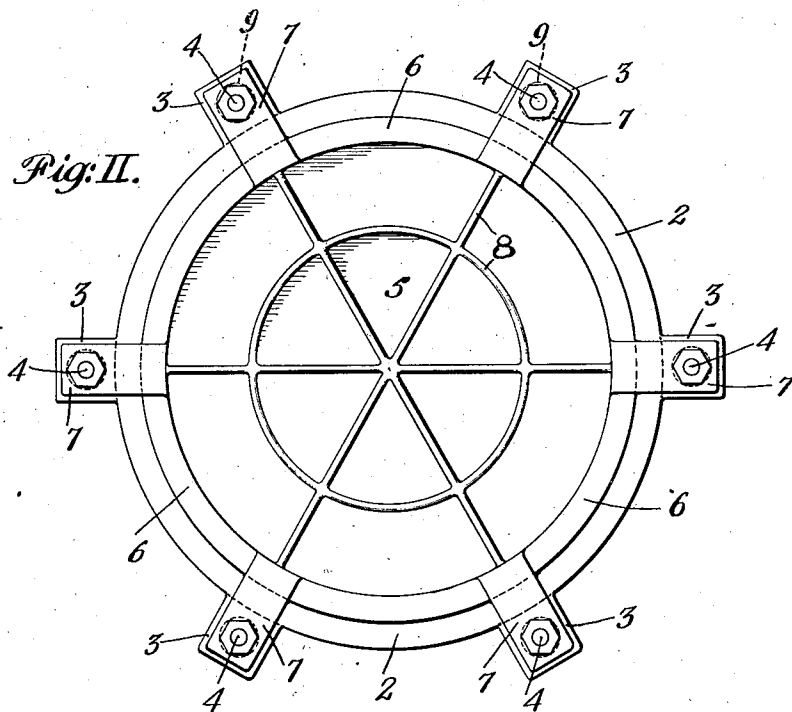


Fig. II.



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PROCESS OF MANUFACTURING POROUS BODIES, DIAPHRAGMS, FILTERS, AND THE LIKE OF EBONITE.

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The present invention relates to the production of diaphragms or filters and of other porous bodies from ebonite.

The different kinds of diaphragms or filters, prepared with the different processes and used up to the present, either do not withstand the action of alkali (e. g. diaphragms of nitrocloth, cement, ceramic diaphragms, which contain free silica) or do not withstand the action of acids or chlorine (e. g. the diaphragms of nitrocloth, cement, asbestos, etc.).

These diaphragms do not also always have the desired physical properties. Some of them become soft in water, swell in the solution and lose their mechanical strength (e. g. sheets of asbestos); others do not swell, but become brittle and lose in this way their mechanical strength; others again cannot be produced of the desired shape, or cannot be produced straight enough for large surfaces (e. g. the ceramic diaphragms, etc.); others again present great difficulties in their manufacture, etc.

Another special feature of the different kinds of diaphragms prepared with the different methods, is that their production is restricted to special porosities and percolations, and that the last can be varied only in comparatively narrow limits. This restricts the field of the useful application of the different kinds of diaphragms.

These diaphragms have further the disadvantage, that they cannot be produced of any size or shape one may choose, that they cannot readily be used in combination with metals and especially not with insulated metals, and that after temporary use they cannot be repaired in case of damage.

The object of the present invention is to produce diaphragms or filters which are free from the above disadvantages.

By the present invention diaphragms or filters are manufactured from ebonite, which is capable of withstanding the action of alkali, acids and chlorine, the ebonite mixtures being prepared in accordance with my earlier U. S. Patent No. 1,022,014 issued April 2, 1908, by which the chemical strength of the diaphragms is secured. The diaphragms can be made of any desired porosity and of any desired percolation properties. As they are made of ebonite, they have also the property of adhering to

iron and to other metals, as well as of combining with metals which are totally or partially insulated either with a non-vulcanized or with a partially vulcanized or with a fully vulcanized ebonite-mixture. This makes it possible for the first time to manufacture electrolytic cells and apparatus for the most varied purposes in a technically rational manner, both from the chemical and from the mechanical point of view. These diaphragms can be repaired in case of damage; they can be freed after use from all substances which may have blocked up their capillaries during electrolysis or filtration, and be used again, by treating the diaphragms with acids or other suitable reagents.

The means or methods used for the manufacture of these diaphragms or filters are of a varied kind; they are nevertheless all based upon the same principle, consisting in the manufacture of the same from particles which have the property, on heating (during vulcanization) to a suitable temperature, of combining easily with one another. Particles with such properties can be prepared either from partially vulcanized ebonite (i. e. ebonite which has been vulcanized only a short time, so that it is still soft or leathery instead of being hard) in form of powder, or from totally vulcanized hard ebonite in the form of fine powder, fine shavings, etc., by covering the same with a thin layer of nonvulcanized or partially vulcanized ebonite. The last has on the one hand the property of combining during vulcanization with the enveloped fine powder, shavings, etc., and on the other hand of causing the particles of the powder, shavings, etc., to combine with one another, while transforming itself at the same time into hard ebonite.

The tendency of the particles thus prepared to easily combine with one another on heating, while they only slightly touch each other, and for the mass to subsequently transform during vulcanization into hard rubber, enables one to manufacture ebonite diaphragms or filters and other porous bodies of the most varied porosities and speeds of percolation.

In order that one manner of doing this may be readily understood, I show in the accompanying drawings a form of mold adapted for my purposes. In these drawings, Fig.

I shows a sectional view of such a mold, and Fig. II a plan thereof.

The numeral 2 designates the main portion of the mold which in this instance is circular. Extending out from the upper edge of the mold 2 are flanges 3 from which project bolts 4. The cover 5 has sides 6 from which extend flanges 7. These flanges 7 are apertured so that they will slide on bolts 4. If desired, the cover 5 may be strengthened by webbing 8.

A quantity of the prepared powder is then taken, sufficient, if compressed so that no interstices would remain, to form a layer in the mold of a thickness considerably less than the total depth of the mold 2. For example, if the mold has a bottom area of one sq. dec. and 50 grams of fine ebonite powder of the specific gravity of 1.25 were used, and the mold were subjected to a pressure of 150 atmospheres, we should get after vulcanization a dense ebonite plate free from pores which would be about 4 mm. thick. Obviously, if the plate 5 is so held that the diaphragm cannot be compressed down to a thickness of 4 mm., a certain degree of porosity will result and this porosity will vary with the thickness of the final plate. In order to control such porosity, I utilize spreader blocks 9 which fit over bolts 4 and by substituting various spreader blocks of different lengths, the degree of porosity can be exactly controlled. For example, if the final plate should be 5, 6, 7, 8, 10 or 12 mm. thick, instead of 4 mm., we would obtain porous plates or diaphragms in which the volume of the air capillaries is equal to 20, 33, 43, 50, 60 and 66% of the total plate or diaphragm. In this way we can get porosities which run through the whole scale of the known porosities for all of the different kinds of diaphragms used up to the present, and it is quite evident that in the same manner still greater and still smaller porosities can be produced. After the proper spreader blocks 9 are in place, the cover is pressed into place and the nuts are then screwed down on bolts 4, after which the mold is placed in a vulcanizer and the material is cured in the usual manner, preferably at a temperature corresponding to steam at 3 atmospheres pressure, though this may have to be varied to suit changing conditions.

Hand in hand with the different porosities we get also the greatest variation in speeds of percolation, which run through the whole scale of known speeds of percolation, and more. The speed of percolation of a diaphragm or filter by a solution depends, as known, upon the radius of its capillaries. According to the law of Stokes and Neumann the speed of discharge of a liquid through a capillary tube is directly proportional to the 4th power of its radius. The last, i. e. the radius of the capillary tubes

depends in case of diaphragms upon two factors: in the first instance, upon the ratio of the air volume of the capillary tubes of the diaphragm to the total volume of the diaphragm, and secondly, upon the degree of subdivision of the ebonite powder or shavings used for the preparation of the diaphragms. As we are able to get the ebonite powder or shavings of the most varied degree of fineness and also to vary the porosity in very great limits, we are therefore able to vary the radius of the capillary tubes of the diaphragms in very great limits, thus getting diaphragms or filters of the most varied speed of percolation.

While it is obvious that the chemical composition of the partially vulcanized or totally vulcanized ebonite used for the diaphragms can vary within wide limits and can be selected in accordance with the use to which the product is to be put, nevertheless, if it is desired that the diaphragms or filters should stand alkali and acids, as it is requisite for the chemical and the electrolytic industries, the ebonite mixtures used for the diaphragms should be prepared and the mixtures vulcanized in accordance with my Patent No. 1,022,014 above referred to, i. e. the partially vulcanized ebonite powder or shavings or the binding medium consisting of the thin layer of unvulcanized ebonite round the particles or shavings of hard ebonite, must be of a certain composition and must be sufficiently long vulcanized, or "over-vulcanized". The use of vulcanization-accelerators does not alter anything in the essence of vulcanization, as long as they do not have a detrimental influence on the chemical composition of the ebonite.

The covering of the ebonite powder or shavings etc. with a thin layer of nonvulcanized ebonite is carried through in the following manner:

If, e. g., diaphragms capable of withstanding the action of acids and alkali are to be prepared, then a solution of 55% rubber in benzine with 10% graphite and 35% sulphur is prepared; the fine powder or shavings are then introduced into such a solution, then separated by filtration from the same, dried in vacuum with recovery of the solvent and pulverized. This operation is repeated if necessary.

If now the powder or the fine shavings so prepared are vulcanized in a mold the requisite time, the thin covering layer of the nonvulcanized ebonite is transformed into hard vulcanized ebonite, and combines on the one hand with the enclosed ebonite powder or shavings, and on the other hand it causes the particles of the powder or shavings to combine with one another, so that a diaphragm or filter is obtained consisting only of vulcanized ebonite. The characteristic feature of this method consists in this, that

the binding medium (the unvulcanized ebonite) itself is transformed during vulcanization itself into hard ebonite, so that after vulcanization the porous diaphragm no longer contains any dissimilar binding medium, the whole of the diaphragm consisting of vulcanized ebonite only.

The vulcanized ebonite used for the preparation of the ebonite powder or shavings need not have a long vulcanization, since it subsequently undergoes a further vulcanization, while the nonvulcanized ebonite of the top surface of the particles is being vulcanized.

When partially vulcanized ebonite powder is to be used for the manufacture of the diaphragms, the ebonite used for the preparation of the powder has to be only partially vulcanized, and this partial vulcanization is carried on for a longer or a shorter time according to the fineness of the powder which it is desirable to get from it. The finer the powder is to be, the longer this partial vulcanization must last.

On the other hand it is possible, by keeping the time of the partial vulcanization of the ebonite for the powder as short as possible and by vulcanizing subsequently the powder in the mold also as short a time as possible, to get porous pliable, leatherlike diaphragms or filters, which can also be applied for a number of useful purposes.

Another method of preparing ebonite diaphragms or filters consists in the following:

The partially vulcanized ebonite powder, or the vulcanized hard powder or shavings or the like covered with a thin layer of nonvulcanized ebonite, is mixed with a suitable quantity of a powder of a substance like salt etc. which does not combine with the ebonite and which after vulcanization can be removed by extraction with water or another suitable solvent or by some other physical or chemical means. The mixture is compressed in the mold and vulcanized. This method proves, however, to be not only more complicated, but also less satisfactory than the above described much simpler methods. It is found that the mechanical strength of such diaphragms invariably suffer much, the cohesion of the ebonite particles becoming much weaker.

As it is possible to get with the methods described above diaphragms of any thickness, size and shape, of any desired porosity and percolation properties, and as the diaphragms, being made of ebonite, have also the property, that they can be easily made to stick to iron and other metals, as well as to metals which are partially or totally covered either with unvulcanized or with partially vulcanized or with completely vulcanized (hard) ebonite, it is evident that the field of their application is very great, and the manner of their application can also

be of a most varied kind. Thus it is possible e. g. to use very porous diaphragms of great percolation properties as supports for other diaphragms or filtering materials, such as sand, barium sulphate, flocks of asbestos, etc., in which case the latter act as the filtering material taking up all the impurities, while the ebonite diaphragms enable one to use these filters or filtering materials in a more rational manner and for a longer time. The ebonite diaphragms can also be made of such a shape as to securely receive and keep the barium sulphate, flocks of asbestos, etc., and so as to enable one to remove the same from an electrolytic cell in a practical manner.

Similarly, the diaphragms can be so constructed that their surfaces are especially suited for the reception and keeping other finely divided substances, such as barium sulphate, Portland cement, colloids, etc. In this way it is possible e. g. to fill up the capillaries only in the uppermost layer of the diaphragm with such substances, while the rest of the diaphragm remains as before of a great porosity and of a small electrical resistance. In this way diaphragms of very small speed of percolation can be obtained. This may be illustrated by the following example:

A 7 mm. thick diaphragm was prepared from fine powder, made from vulcanized hard ebonite and covered with a very thin layer of nonvulcanized ebonite, of a porosity=43%: it had an absolute coefficient of percolation (for water at 13° C.) $k=0.206$. A paste of fine natural barium sulphate was then prepared and rubbed into the pores of the top surfaces of the diaphragm with a piece of cloth, which operation required only very little time. The absolute coefficient of percolation became about 1000 times smaller, k having become equal to 0.0001907.

For the reasons given above the field of application of the diaphragms and filters is very great. Not only will they be of great use for the numerous electrolytic systems for production of alkali and chlorine, of metals, of gases such as hydrogen and oxygen, of the numerous substances used extensively in the dye-industry (obtained either by electrolytic oxidation or by electrolytic reduction), of perchlorates etc., which all use diaphragms, but they will be also employed with great advantage for many other purposes of the electrolytic and chemical industries, such as for purification of solutions used for electrolysis, for separation of solids from liquids, for preparation of pure salts by means of partial solution, for preparation of table salt from rock salt etc. There is also another great field of application of the porous diaphragms or bodies, namely in electric batteries.

Having now particularly described and

ascertained the nature of my said invention and in what manner the same is to be performed I declare that what I claim is:—

1. The process of manufacturing porous ebonite bodies which comprises bringing partially cured ebonite particles, like powder, shavings and the like into such a volume as will cause the adjacent particles to sufficiently contact and combine under the action of heat to form a body of connected particles, but will prevent them thereafter being compacted into a non-porous solid mass, and then vulcanizing them into an integral body of ebonite.

2. The process of manufacturing porous ebonite bodies which comprises bringing ebonite particles, like powder, shavings and the like, at least the surface portions of such particles being only partially cured, into such a volume as will cause the adjacent particles to sufficiently contact and combine under the action of heat to form a body of connected particles, but will prevent them from being compacted into a non-porous mass, and thereafter vulcanizing them into an integral body.

3. A process of making porous ebonite bodies which comprises pressing particles, like powder, shavings and the like, of partially cured ebonite, into a mold having a greater volume than the aggregate volume of such particles, and vulcanizing the same until a porous body of leatherlike consistency is obtained.

4. A porous body comprising a distended mass of ebonite particles integrally united by vulcanization and finely divided material impregnating interstices between such united particles.

5. A filter body comprising a porous body comprising a distended mass of ebonite par-

ticles integrally united by vulcanization and other subdivided filtering material supported thereby.

6. A process of making a porous ebonite body which comprises pressing particles like powder, shavings, and the like of partially cured ebonite into a mass of contacting particles having a greater volume than the sum of the volumes of the individual particles and vulcanizing the mass.

7. A process of making a porous ebonite body which comprises pressing ebonite particles like powder, shavings, and the like whose surface portions are not fully vulcanized and are capable of vulcanization, into a mass of contacting particles having a greater volume than the sum of the volumes of the individual particles and vulcanizing the mass.

8. The process of manufacturing porous ebonite bodies which comprises bringing ebonite particles, whose surface portions at least are only partially cured, into such volume that they are in contact, but have interstitial spaces, and vulcanizing the mass.

9. A porous ebonite body comprising initially partially cured ebonite particles brought into such volume that the adjacent particles are in contact but are not compacted into a non-porous solid mass, the particles being permanently united by vulcanization.

10. A porous ebonite body comprising ebonite particles whose surface portions are initially only partially cured, the particles occupying such volume that adjacent particles are in contact, but are not compacted into a non-porous solid mass, the particles being permanently united by vulcanization.

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