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METHOD OF TREATING ELECTRODES

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This invention relates to carbon electrodes and a method of treating them to make them more durable, and more particularly to such electrodes for use in the electrolysis of aqueous solutions of alkali metal halides.

For the above-mentioned purpose carbon electrodes, which include graphite electrodes, have in the past been impregnated with a drying oil, such as linseed oil, which was then allowed or caused to dry or harden. A common way of drying the oil was to merely set the electrodes aside for several months, but of course that method required a lot of time and space. The reason for treating electrodes with a drying oil is to make them more durable and resistant to the electrolytes in which they are used, but the difficulty with the methods used in the past has been that, in addition to requiring considerable time, the drying oil has not been entirely cured so that full advantage of the benefits of properly treated electrodes could be realized.

It is among the objects of this invention to provide a treated carbon electrode of this character which can be made in a short time, which is more durable and has a longer and more useful life than similar electrodes known heretofore, which does not spall or crumble during electrolysis, which corrodes more slowly and uniformly than heretofore, which materially reduces the fouling and plugging of alkali chlorine cells, and from which the drying oil does not ooze or exude when the electrode is in service.

Another object is to provide a method for making such an electrode by which a uniform cure from batch to batch is obtained.

In accordance with this invention a carbon electrode is impregnated with a drying oil of a character to be described presently, and is then heated at a temperature high enough to harden the oil into a gel which will remain solid or in situ even though the electrode later is heated to a temperature high enough to cause the gelatinized oil to smoke. Such a gel is known as a temperature-irreversible gel. The drying oils that can be used should be those that are susceptible to being cured in air at a high temperature to a state in which they will not ooze out of the electrodes even though the electrodes are later heated to a temperature at which the oils will smoke. The electrodes are not subjected to such a high temperature in service, but it has been found that if the gelatinized oil will not ooze out of the electrodes when heated to a temperature at which they will smoke, the gel is temperature-irreversible and the electrodes

will have the long life and other desirable properties that it is the object of this invention to obtain. The smoke test thus is a convenient way of determining whether or not the electrodes will have the desired properties when placed in service. Suitable oils for this purpose are linseed oil and oiticica oil, or mixtures of the suitable oils.

The electrodes are impregnated with the drying oil in any suitable manner, such as by the vacuum-pressure method or merely by soaking them in a vat of hot or cold drying oil. The amount of drying oil that an electrode should contain is from about 4 to 9 percent by weight of oil. The percentage of oil can be controlled by the impregnating method employed and also by diluting the drying oil with a solvent or by using an emulsion of drying oil and water. For example, the electrodes may be impregnated in an autoclave by subjecting them to a vacuum of about 25 inches of mercury and then introducing the oil in sufficient quantity to completely immerse the electrodes while under vacuum. The vacuum is then released and the electrodes allowed to soak in the drying oil at atmospheric or super-atmospheric pressure until they are saturated, whereupon the oil is removed from the autoclave and a vacuum again applied to remove a portion of the oil from the pores of the carbon. By controlling the conditions, such as the degree of vacuum and the time it is applied, it is possible to produce an impregnated electrode which contains between 6 and 8 percent of drying oil which is the preferred amount for the practice of this invention.

Another method of removing a portion of the oil from the fully impregnated electrode referred to in the preceding paragraph is to subject the saturated electrode to heat applied directly to the autoclave. The action of the heat is to expand the oil and any air occluded in the carbon and thereby force a portion of the oil to the surface where it drains off.

A further manner of securing partial impregnation by a drying oil is to use either a solution of oil in a solvent, or an emulsion of oil and water with a suitable emulsifying agent such as triethanolamine. The carbon electrodes are fully saturated with the oil solution or the oil emulsion after which the solvent or water is evaporated from the electrodes by reduced pressure, by heat or by a combination of both, the amount of oil in the electrodes being controlled by the ratio of oil to solvent or of oil to water. A preferred procedure for producing our elec-

trodes is to impregnate them by the vacuum-pressure method with a solution consisting of about 60 per cent by weight of linseed oil and 40 per cent by weight of acetone. When the electrodes are removed from the impregnating autoclave they will contain from 11 to 15 per cent by weight of linseed oil-acetone solution. These electrodes are then introduced into a vacuum dryer where they are subjected to a vacuum. When the greater portion of the acetone is volatilized low pressure steam is admitted to the steam jacket of the dryer in order to completely remove the acetone which can be received by condensation or adsorption. Upon completion of the vacuum drying the electrodes will contain from 6 to 8 per cent of linseed oil.

The impregnated electrodes, regardless of the method by which they have been impregnated, are then placed in a suitable oven where there is free circulation of air all around them. In this oven they are heated at a temperature high enough and for a time long enough to cure the drying oil by causing it to set or harden into a temperature-irreversible gel, i. e., one that will remain in situ and not ooze out of the electrodes even though they are later heated to a temperature high enough to cause the gelatinized oil to smoke. In the case of electrodes impregnated with linseed oil as referred to above they are preferably gradually heated to a temperature of 260° C. and maintained at that temperature for about 15 hours to convert the oil from a liquid state to a substantially completely hardened or gel state. In effecting this transition of the oil both the chemical and physical properties are appreciably altered. For example, with linseed oil the acid value is greatly increased while the iodine value is decreased. Linseed oil can be cured in this manner when heated to a temperature between 200 and 350° C. by varying the heating time, a longer heating period being required for the lower temperatures. The temperature range for properly curing oiticica oil is between about 225 and 350° C. However, the maximum temperature at which an oil is cured should be limited by volatilization of the oil because, otherwise, all of the oil might be volatilized out of the electrodes. We prefer to limit ourselves to a temperature which will not result in a loss of more than 1 per cent of the weight of the electrode.

Electrodes prepared in accordance with this invention have a much longer service life and their performance in conventional types of chlorine cells is much better than electrodes treated by other known methods. Laboratory tests conducted in an electrolytic cell for the electrolysis of sodium chloride solution show a very pronounced improvement in the life of our electrodes. Thus, electrodes treated with linseed oil in accordance with this invention showed an average loss or reduction in size of $\frac{1}{1000}$ of an inch per thousand hours as compared with $\frac{4}{1000}$ for untreated electrodes and $\frac{3}{1000}$ for electrodes treated with linseed oil by the methods known heretofore. Any

of the varieties of linseed oil can be used in practicing this invention, either alone or with the addition of dryers such as lead, manganese or cobalt compounds.

According to the provisions of the patent statutes, we have explained the principle and mode of practicing our invention and have described what we now consider to represent its best embodiment. However, we desire to have it understood that, within the scope of the appended claims, the invention may be practiced otherwise than as specifically described.

We claim:

1. The method of treating a carbon electrode, comprising impregnating it with linseed oil and then curing the oil by heating the electrode in air at a temperature above 200° C. but below a temperature at which an appreciable amount of the oil will volatilize, whereby to convert the oil to a temperature-irreversible gel.

2. The method of treating a carbon electrode, comprising impregnating it with linseed oil and then heating the electrode in air at a temperature between 200° C. and 350° C. to cure it whereby to convert the oil into a temperature-irreversible gel, said oil constituting between 4 per cent and 9 per cent of the weight of the electrode.

3. The method of treating a carbon electrode, comprising impregnating it with linseed oil and then heating the electrode in air at a temperature between 200° C. and 350° C. long enough to set the oil into a temperature-irreversible gel.

4. The method of treating a carbon electrode, comprising impregnating it with boiled linseed oil and then heating the electrode in air at about 260° C. for about 15 hours to cure the oil and thereby harden it into a stable gel that is temperature-irreversible.

5. The method of treating a carbon electrode, comprising impregnating it with oiticica oil and then heating the electrode in air at a temperature between 225° C. and 350° C. long enough to set the oil into a temperature-irreversible gel.

6. The method of treating a carbon electrode, comprising impregnating it with a solution consisting of about 60 per cent by weight of linseed oil and about 40 per cent by weight of volatile solvent, volatilizing the volatile solvent from the electrode whereby the oil therein constitutes about 6 to 8 per cent of the weight of the electrode, and heating the electrode in air at a temperature of about 260° C. until the oil is converted from a fluid state to a temperature-irreversible gel.

7. The method of treating a carbon electrode comprising impregnating it with a drying oil from the class consisting of linseed and oiticica oils, and then curing the oil by heating the electrode in air at a temperature above 200° C. but below a temperature at which an appreciable amount of the oil will volatilize whereby to convert the oil into a temperature-irreversible gel.

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