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PROCESS FOR THE TREATMENT OF SILICATES WITH ACIDS IN ORDER TO OBTAIN THEIR SOLUBLE SALTS

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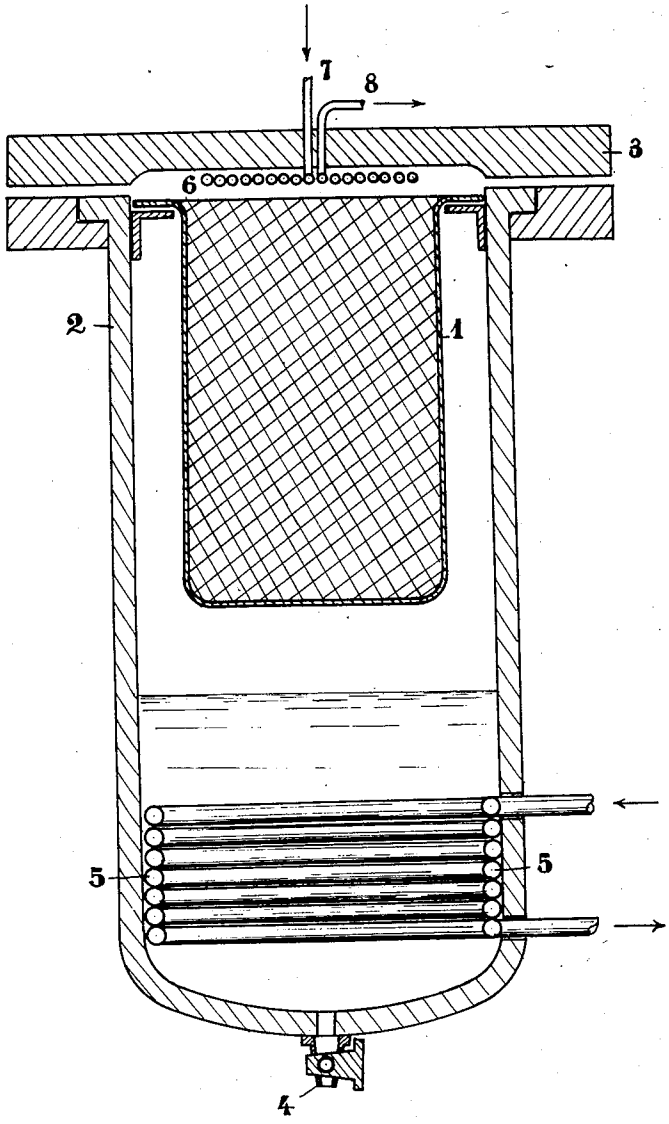


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## PROCESS FOR THE TREATMENT OF SILICATES WITH ACIDS IN ORDER TO OBTAIN THEIR SOLUBLE SALTS

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1 Claim. (Cl. 23—32)

It is known that many silicates, mainly those containing alkali metals (potassium, sodium, lithium) may be attacked at high temperature and under pressure by the vapours of volatile acids, such as hydrochloric or nitric acid. When the attack is effected with the vapours at high temperature of these acids, which may also be mixed with water steam, a liquid condensate is obtained which contains in solution the soluble salts resulting from the reaction, the concentration of the said solution being however slight, which renders in practice the process very costly and consequently unsuitable, as it would require to be repeatedly applied or prosecuted by renewing continuously the vapours of the acid used in order to exhaust completely the minerals subjected to the treatment.

The present invention has for its object a new method for effecting the above said treatment, which allows to obtain with a small expense and in a practical and very convenient way, highly concentrated solutions of the salts resulting from the attack of rocks such as leucite, and other similar silicates, even with the condensable vapours of active liquids, which in consequence of the attack are but slightly charged with the soluble salts formed or dissolved.

The principle on which the working of the process according to the present invention is based, is the following: After the attack at high temperature and pressure of the rock under treatment, either alone or in the presence of other substances capable of facilitating the reaction, and after collecting the liquid condensate or the liquid dropping down mechanically, instead of attacking the rock with the vapour from another liquid, the vapour obtained by re-evaporating the condensed liquid, is used; such vapour being pure as it consists only of vapour of acid and water steam, the dissolved substances in the liquid, in the case considered, being all fixed substances, so that the second attack has the same effect as if fresh vapours and steam were used. The condensed vapour contains other substances dissolved in it which increase the concentration of the liquid, the said operation being continued thus enriching successively the liquid until the complete exhaustion of all the useful components of the rock has been reached.

In practice, it has been found convenient to use a quantity of liquid which is sufficient to extract from the rock and to transfer into the solution all the salts obtainable from the rock under treatment, to maintain the liquid always at the boiling temperature and under pressure, and the charge of rock to be treated at a temperature either equal (when wet vapours are used) or slightly lower, so that the vapour coming in contact with it may condense, both the liquid and the mineral being enclosed in the same closed container, having such dimensions that the liquid may not reach or come in contact with the material placed above it.

Under the said operating conditions, the process can be carried out in a continuous manner, and when good care is taken for thermally insulating the whole apparatus, in order to prevent heat losses, the consumption of combustible will be very small, viz: just the quantity which is sufficient to replace the heat lost owing to the slight decrease of the temperature of the charge under treatment, whenever it is necessary to decrease the temperature of the said charge.

The annexed drawing shows schematically a vertical section of the apparatus used for carrying the invention into practice.

Supposing, by way of example, that it is desired to treat the leucite with nitric acid so as to obtain potassium and aluminium nitrates, the operation is carried out as follows:

The leucite is firstly ground or granulated and then placed in a container 1 perforated at the bottom and uncovered at the top. The said container is then introduced into the higher portion of an autoclave formed by a vertical tube 2 capable of withstanding the pressure. The height of the autoclave must be sufficient to allow that, upon having poured into it the liquid required for the reaction, which collects at the bottom and which in the present case is nitric acid, in a quantity slightly less than that indicated by theory, the said liquid does not reach the vessel containing the charge of leucite, which is placed into the upper portion of the tube 2. The tube must be made with a material capable to withstand the action of the reagents used and of the reaction products, or it must be eventually lined with a material which is not attacked by the said reagents and reaction products, such as for example grit sandstone.

The autoclave is provided in its lower part with a discharge cock 4 for the exit of the saturated liquid. The fresh liquid is introduced from the top cover 3 through a suitable inlet.

After the autoclave has been charged as described, it is closed, and the liquid, which is collected at its bottom, is heated in any convenient way, such for example, by means of an electric

resistance, or by steam circulation in a steam pipe 5 or by direct fire.

On the head of the autoclave, in proximity to the charge of leucite, a slight cooling is produced, for example by means of cold water circulating through a pipe having an inlet 7 and an outlet 8, which causes the condensation of the vapour, which condensed vapour drops through the underlying layer of leucite from which it drops again to the bottom of the autoclave where it rejoins the boiling liquid.

In practical working the cooling above mentioned is not required when the steam is already saturated with moisture as is the case in the apparatus now described.

As nitric acid boils at a temperature which is lower than the boiling point of water, there will be produced, under pressure and at high temperature, vapours rich in nitric acid and water, which will pass through the mass of leucite from the bottom to the top or conversely and the salts formed remain dissolved in the condensed liquid, falling to the bottom of the autoclave where the nitric acid is collected.

If the temperature of the attack is kept above 100° C., the silica is no longer dissolved and does not assume the gelatinous form which causes the obstructions occurring in the masses of the attack when operating with no pressure apparatuses. In addition, the heat, developed during the reaction of the attack in a closed apparatus, may be utilized wholly or in part for the heating required after starting the operation.

Moreover, if the temperature is kept higher than 125° C., the following additional advantage is obtained: it is known that iron nitrate decomposes at a temperature of 125° C., while the aluminium nitrate requires for its decomposition a temperature of at least 140° C. Therefore, by working at an intermediate convenient temperature, between the two limits above mentioned, it will thus be possible to eliminate the greater portion of the iron present in the form of iron oxide which precipitates with the insoluble residue.

The above described result could not be obtained without operating the attack under pressure and with the acid in the state of vapour, because, if the attack is effected with the liquid acid, the boiling temperature is a function of the concentration of the solution in acid and in salts, which varies continuously and consequently the temperature cannot be regulated. The boiling action agitates the mass, while with the operation made under pressure with the acid in vapour form, the desired effect of dissolving rapidly the potash and the alumina is obtained directly and in a very simple manner, by operating at such a temperature that neither the iron nor the silica may pass into solution.

If a small quantity of iron, coming from the leucite or from the eventual corrosion of the material of the apparatus, should pass into the solution, there may be added in the solution, kept under pressure, a precipitant for the iron, such for example potassium ferrocyanide, or any other suitable reagent. The precipitate is separated from the liquid by filtration, for example by passing the solution through a layer of silica formed with the exhausted leucite, which latter thus serves no longer to eliminate the silica, which with the process described is produced in the insoluble form, but it eliminates the iron which has passed in the solution in the form of nitrate.

The iron precipitate may on the other hand be eliminated by using any other of the methods of filtration generally known, or by means of a centrifugal machine, or by any other method known and used in the industry for separating solid substances held in suspension in liquids.

When the attack has been completed, the exhausted leucite is removed from the hot apparatus, and another quantity of fresh leucite contained in another container similar to that above described, is introduced into the apparatus, the autoclave is then closed, and the heat is maintained by producing vacuum in it, so that the water in excess may be distilled up to the amount which is considered necessary, passing the water vapour through the leucite. By operating in this way the last traces of free acid which may still be present in the solution become fixed, while the distilled water exhausted is free from acid and salts.

The leucite contained in the container is thus brought to the convenient temperature required to start a fresh treatment; the autoclave is then emptied by withdrawing from it the concentrated solution of potassium and aluminium nitrates, free from iron and from silica, from which the said two nitrates can be separated by any known methods, subsequently admitting a fresh charge of acid into the autoclave.

The operation carried out as above described allows to effect the rapid attack of the leucite, to obtain the complete fixing of the acid without any loss, and also the complete purification of the liquid which is free from silica and from iron, as well as its concentration by eliminating from it the pure water without the use of pumps or of other costly and complicated distilling apparatuses.

I claim:

The process of treating silicates with acid vapours at high temperature for extracting therefrom the corresponding salts, comprising charging the silicate into a cage having a perforated bottom and open at the top, disposed in the upper portion of a closed container, while charging the liquid, consisting of an aqueous solution of acid, into the lower portion of the said container and heating it in order to obtain wet saturated acid vapour, permitting the same to pass through the mineral, which is at a slightly lower temperature, the vapour thus condensed and saturated with the soluble salt abstracted from the mineral dropping into the hot liquid at the bottom of the container, the cycle above described being continued until the material is completely exhausted or the underlying liquid has reached saturation of the said substances, including the further step of introducing at the end of the treatment above specified a fresh charge of material into the cage to replace the exhausted charge, permitting the apparatus to cool thereby causing the distillation of the liquid therein contained, thereby leaving the liquid free from all traces of free acid and allowing the vapours from said distillation to pass through the said fresh charge of material, thereby utilizing the acid distilled from the liquid, and finally withdrawing the concentrated liquid free from acid, iron and silica from said container.

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