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Process for the Extraction of Aluminum from Aluminum Ores

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***Abstract*: Aluminium may be recovered from various raw materials including kaolinite , alunite , coal ash and slag , and both raw and spent oil shale by contacting such materials with an aqueous solution of hydrofluoric acid , followed by removal of insoluble residues remaining suspended in solution and the precipitation of hydrated aluminium hydroxide from the clear solution by basification with an alkaline agent**

***Keywords*: aluminium, red mud, Bayers process, fluoride , calcium**

1. Introduction

Aluminium or aluminum is a chemical element with symbol Al and atomic number 13. It is a silvery- white, soft, nonmagnetic and ductile metal in the boron group. By mass, aluminium makes up about 8% of the Earth's crust; it is the third most abundant element after oxygen and silicon and the most abundant element after oxygen and silicon and the most abundant metal in the crust, though it is less common in the mantle below. The chief ore of aluminium metal is so chemically reactive that specimens are rare and limited to extreme reducing environments. It is found combined in over 270 different minerals. Aluminium is remarkable for its low density and its ability to resist corrosion through the phenomenon of passivation. Aluminium and its alloys are vital to the aerospace industry and important in transportation and building industries, such as building facades and window frames.The oxides and sulphates are the most useful compunds of aluminium

Despite its prevalence in the environment, no known form of life uses aluminum salts metabolically, but aluminium is well tolerated by plants and animals. Because of these salts' abundance, the potential for a biological role for them is of continuing interest, and studies continue. In the standard Bayer's process for refining bauxite to produce alumina the dissolution of alumina in the ore to form sodium aluminate is accomplished in autoclaves under high pressure varying from 70-200 pounds per square inch. However some lateritic bauxite ores are readily soluble in caustic making it possible for them to be leached or digested at atmospheric pressure

This study deals with the treatment of one such lateritic bauxite ore batch laboratory test shows that a high alumina extraction in caustic could be accomplished at a coarse grind thus promising low grinding cost and of greater importance a coarse sand residue which could be washed more efficiently thus keeping soluble loss to only a trace. The optimum grind was found to be -3/16" with 51%+32 mesh. However at this coarse grind and at the desirable pulp density for the digestion of approximately 23% solids the heavy specific gravity solids 3.0 to 3.5 had a settling rate as high as 75 feet per minute the problem resolved itself to finding some type of continuous mixture or agitator which would keep the coarse fast settling in homogenous suspensions. During the digestion operation so that all of the bauxite particles would be attacked by the caustic sodium aluminate liquor in this way it would be possible to capitalize on the important advantages of the coarse grind.

2. Literature survey

In the paper "Process of extraction of aluminum values from coal ash" by Robert O. Hill Bernard Raistrick he says that the process for preparing a solution high aluminum content from the combustion residue of a solid carbon-containing fuel having a carbon content of 2% or less by weight and an iron content calculated as Fe2 O3 of 4% or less by weight is described. His process consists of (a) pre-treating said residue with nitric acid at a temperature lower than the next extraction stage which follows and removing the nitric acid solution from the residue and then (b) extracting said treated residue with nitric acid of strength about 30% to 65% by weight strength at about 100° to 225° C., the amount of nitric acid employed in said pre-treatment step and is said extraction step being in excess of the cationic equivalent actually dissolved in each step.

In "Processes extracting aluminum and iron from aluminous ores" by Richard Boudreault Serge Alex Fabienne Biasotto they say that processes and compositions for extracting aluminum and/or iron ions from aluminous ores or a mixture. Their process comprises recovering said aluminum ions from a composition comprising said aluminum ions, said iron ions, an organic solvent and an extracting agent adapted to form an organometallic complex substantially selectively with said iron ions or with said aluminum ions which is soluble in said organic solvent. Another process for extracting aluminum from an ore comprising leaching said ore with an acid so as to obtain a leachate and a solid residue and removing at least a portion of iron ions contained in said leachate by either substantially selectively precipitating said at least a portion of said iron ions in basic conditions in which the pH is of at least 10, or substantially selectively complex said at least a portion of said iron ions with an extracting agent adapted to form organometallic complex. Another process for at least partially separating aluminum ions from iron ions comprised in a composition comprises substantially selectively precipitating at least a portion of said iron ions in basic conditions in which the pH is at least 10.

In the paper" Process for treating red mud to recover metal" by Robert J. Barnett Michael B. Mezner they say that their method for processing red mud to recover iron, aluminum, silicon and titanium metal values therefrom the method comprising the steps of adding the red mud to a digester containing an acid to provide a mixture of acid and red mud and heating the mixture to dissolve soluble compounds of at least one of iron, aluminum, silicon and titanium to provide a digest containing dissolved salts of the soluble compounds and to provide a gas component. Thereafter, the digest is treated with water to dissolve water soluble salts therein to provide slurry comprised of liquid containing water and the dissolved soluble salts and a solid component comprised of silica. The solid component is separated from the liquid and the pH of the liquid is adjusted to form an aluminate and an iron-containing precipitate. The iron-containing precipitate is separated from the liquid to provide an iron-depleted liquid whose pH is adjusted to precipitate aluminum trihydrate which is separated from the iron-depleted liquid to provide an aluminum trihydrate-depleted liquid. The pH of the aluminum trihydrate-depleted liquid is adjusted to form a precipitate comprised of at least one remaining salt in the aluminum trihydrate-depleted liquid to provide a salt-depleted liquid. The salt precipitate is separated from the salt-depleted liquid thereby recovering the metal values from the red mud.

3. Proposed method

The present relates to metallurgy in general and to processes for the recovery of aluminum from its ores particular.

It is specifically incorporated by reference herein, and to which reference may be made for a full understanding of the background of the present invention, the shortcomings of the Bayer process were enumerated. Among these shortcomings is the fact that that process is only capable of extracting aluminum with maximum efficiency from hydrated aluminum oxide ore materials such as gibbsite and boehimite. Thus potential aluminum sources such as the kaolins or goethite are generally viewed as being unsuitable as starting materials in the Bayer process. In addition, aluminum material containing in excess of 5% silica is generally considered unsuitable for use in the Bayer process due to the cost of the digestive removal of the silica. Finally, the very small particle size found in many bauxite ores results in the formation of "red mud" which is only difficultly separated from the alkaline aluminate liquor.

For these and other reasons my prior patent disclosed and claimed the use of various naturally occurring acids such as citric, tartaric and salicylic acids, in a process wherein the aluminum containing ore is treated with a solution containing one or more of such acids in order that the aluminum present in the ore may be complexed by the acid anions are carried into solution. This extraction is followed by filtration to remove the insoluble matter from the solution containing the complexed aluminum, and the filtrate is subsequently treated with a basic reagent whereby precipitated and removed from solution.

Despite the success of such process, there remains a need for a process which will accomplish a similarly efficient removal of aluminum from ores which are presently not amenable to the Bayer process, and also do so even more economically than my prior process.

4. Process

It has now been discovered that aluminum may be efficiently and economically extracted and recovered from raw materials such as kaolinite, alunite, coal ash and slag, and raw and spent oil shale by a hydrofluoric acid extraction process. Broadly stated, the process of the present invention comprises the following four steps:

1. Extracting the aluminum containing raw-material for a predetermined period of time with an aqueous solution of hydrofluoric acid to form an aqueous solution containing aluminum ions in association with from zero to six fluoride ions;
2. Removing the insoluble residue from the aluminum containing solution, such as by filtration etc;
3. Recovering a precipitate consisting essentially of aluminum hydroxide from the filtrate from step (2), by the controlled addition of an alkaline agent and the removal of any non-aluminum hydroxide precipitates; and
4. Isolating aluminum in a useful form, i.e. by filtration and calcination of the precipitate to form alumina.

In an alternative embodiment of my process, the addition of the alkaline agent in step (2) may be stopped just short of the point at which aluminum hydroxide precipitates, the solution filtered, and evaporated to dryness. By so doing, a solid aluminum will be deposited, and the vapor driven off will contain a significant percentage of the hydrofluoric acid originally used as the extracting, which may be recycled for subsequent use.The recovery of HF may be maximized by adding a stoichiometric excess of an inorganic acid of lower volatility than HF such as H2 SO4 or H3 PO4. By so doing, the acid counter ion, i.e., SO4 -2 or, PO4 -3, will replace the fluoride ion, allowing virtually all the HF present to vaporize on heating. The process is facilitated and accelerated by the use of fine particle sizes in the raw materials. In addition, because silicon, which is present as an impurity in a significant amount of most aluminum containing raw materials, is converted to SiF4 gas by the reagent solution, under certain conditions silicon may be separated and recovered as a byproduct from the solution. Finally, the present process is capable of functioning at room temperature, rather than at the elevated temperatures required by the Bayer process, and, as such, represents an improvement over the Bayer process and my prior process. A complete understanding of my process will be gained by those skilled in the art from the following discussion with reference to the drawing.

The process of the present invention comprises extracting aluminum from aluminum contain raw material separating aqueous solutions containing the dissolved aluminum from the insoluble residue, precipitating the aluminum from the solution and calcining the precipitate to yield a final aluminum product. It is further contemplated that the process of the present invention may also be utilized for the recovery of silicon as a by-product. In the preferred embodiment of my invention, an aluminum ore is ground to a uniform particle size, preferably not greater than 2mm. The vessel in which at least the interior is fabricated from stainless steel, a synthetic material such as tetrafluoroethylene, polyethylene or the like, or any other material which is resistant to attack by hydrofluoric acid, the vessel is also preferably equipped with means for agitation such as paddle wheels, vibrators or the like, as is well known to those skilled in the art. Hydrofluoric acid of from about 2% to about 52% preferably from about 25% to about 30%, by weight is added to the vessel in sufficient quantity to extract the aluminum from the ore preferably in a volume to weight ratio of about 4:1 (liters HF.sub.(aq) to kg ore). The resulting mixture is agitated gently in order to promote the extraction reaction for a sufficient period of time to accomplish the extraction, preferably not more than two hours. As will be evident to those skilled in the art, the rate of extraction will be a function both of reagent concentration and of contact time. Thus a wide but coordinated variation in both conditions is contemplated for the process.

The temperature of the extraction step is controlled at not greater than 100° C, and preferably at not greater than 30° C, by choice of reagent concentration, rate of addition and/or amount of agitation. As is disclosed herein below, the exothermic nature of the extraction reaction results in generally better extraction rates at low temperatures. Naturally, any conventional cooling means may be utilized to control solution temperature if desired.

Upon completion of the extraction step, the insoluble residue is separated from the aluminum-containing solution by any one of the conventional methods, ie, filtration, centrifugation, decantation, etc. The separated residue is then preferably washed with a small additional amount of HF extraction solution and the washings combined with the first solution. The remaining residue is then preferably washed with water, and the washings recycled to an HF recovery operation as further described herein below whereas the, HF-free residue is discarded.

As is well known to those skilled in the art, many aluminum-containing materials also contain other hydrolysable elements such as iron and silicon oxides. Since these elements will also be dissolved by hydrofluoric acid solutions, they must separated from the dissolved aluminum prior to its precipitation. The process is conventionally employed for such separation utilizes the differences in the acidities of the hydrated cations by sequentially increasing the solution pH with an alkaline agent and removing the precipitated hydrated metallic oxides. Thus hydrated iron oxide will precipitate at pH 4-5 whereas hydrated aluminum hydroxide will precipitate between pH 7 and 9. Thus the pH of the solution may be raised to 6.0-6.5, the solution filtered to remove the precipitated iron hydroxide, and the pH raised to not more than 10 to precipitate the aluminum. Care must be taken to maintain the solution pH at not greater than 10 to avoid redissolution of the amphoteric hydrates aluminum hydroxide. The preferred alkaline agent would be a moderately concentrated alkali metal hydroxide such as 6 M NaOH, but any alkaline agent may be used so long as its pH is not less than 10 and it will not interfere with subsequent process steps.

The hydrated aluminum hydroxide precipitate is separated from the solution, once again, by any conventional method consistent with the process. The clear solution, containing fluoride ions and the cation of the alkaline agent, is routed to a fluoride recovery operation, whereas the solid aluminum compound is routed to a calcining operation for the production of alumina. The fluoride recovery operation referred to herein above may comprise any conventional operation capable of recovering fluoride in a useful form from its aqueous solutions, such as by the precipitation of calcium fluoride by the addition of an aqueous solution of a soluble calcium salt. From the recovered fluoride, hydrogen fluoride may be generated for reuse in the extraction, ie, by treatment of the residue with concentrated sulfuric acid thereby improving the economics of the process.

In one embodiment of my process, the extraction conditions be predetermined to be consistent with the generation of silicon tetra fluoride from the silicious materials contained in the ore. Under such conditions the extraction is preferably run in a closed vessel equipped with vapor withdrawal apparatus enabling the operator to withdraw the SiF_4 from the extraction vessel and to treat it in a conventional manner in a subsequent operation. One such operation may comprise hydrolysis to form HF and silica and the subsequent recycling of the HF solution.

5. Conclusion

Finally, we conclude that this has the significant advantage that it operates on aluminum-containing raw materials irrespective of how small their particle size or their crystalline state. Thus many waste materials such as coal ash and slag, lignite ash and spent oil shale which may have fine particle size or which may have become amorphous or glassy during prior treatment will function excellently in this process.

While this process has been described with respect to a certain specific embodiment, it will be appreciated that many modifications and changes may be made by those skilled in the art without departing from the spirit of the method. It is intended, therefore, by the appended claims to cover all such modifications and changes as fall within the true spirit and scope of the process.

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