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I. IN THE MATTER OF

[**X**] Application for grant of patent [ ] Application for registration of a utility model

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Application No. given by receiving Office:

II. TITLE

PROCESS FOR RECOVERING VALUABLE METAL FROM RED MUD

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IV. NOTIFICATION

We hereby notify you, pursuant to Rule 26(1), that the above-identified application has been accorded **23.05.2023** (date) as its filing date.

This notification is being sent to****:

Ghana

V. SIGNATURE

06.11.2025

T Bemanya
DIRECTOR GENERAL

(Date)

* A notification Form No. 12 shall be sent to the applicant's representative and to the industrial property office of each designated State.

** Please quote the application number given by the ARIPO Office in all subsequent communications concerning this application.

*** All names and addresses will be displayed on the back of this notification in case of two or more applicants.

**** Indicate all those to whom a notification Form No. 12 is being sent in connection with the above-identified application.

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PROCESS FOR RECOVERING VALUABLE METAL FROM RED MUD

Biblio

(12) PATENT

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(54)Title	PROCESS FOR RECOVERING VALUABLE METAL FROM RED MUD
(57)Abstract	The present disclosure provides a series of processes implemented by combining: a dry process involving reduction roasting and magnetic separation for recovering iron in red mud generated as a by-product in a bauxite processing process; and a wet process for additionally recovering each of aluminum and titanium, respectively, after separation of iron.
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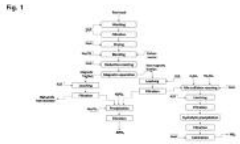
Abstract Figure



【DESCRIPTION】

【Title】

PROCESS FOR RECOVERING VALUABLE METAL FROM RED MUD

【Technical Field】

[1] The present disclosure relates to a process for recovering valuable metals from red mud. More particularly, the present disclosure relates to a series of processes implemented by combining a dry process involving reductive roasting and magnetic separation for recovering iron from red mud generated as a byproduct during bauxite processing, and a wet process for additionally recovering aluminum and titanium, respectively, after separation of iron.

【Background Art】

[2] Red mud is a red-colored byproduct generated in the process of manufacturing alumina from bauxite (i.e., the Bayer process), and is also called bauxite residue or *jeokni* (in Korean). In the Bayer process, to recover alumina from bauxite, caustic soda (NaOH) is added followed by treatment at a high temperature under high pressure. The reaction mechanism by which red mud is generated during the process may be represented in Scheme 1 below.

[3]

[4] [Scheme 1]

[5] $2NaOH + Al_2O_3 \cdot 2H_2O \rightarrow Na_2O \cdot Al_2O_3 + 4H_2O$ + red mud

[6]

[7] As demand for alumina has increased recently, the generation of red mud is also increasing. Typically, red mud is generated in an amount of about 1 to 1.5 tons per ton of alumina produced. With an increase in the demand for alumina, the generation of red mud is also increasing proportionally. The pH of red mud ranges from approximately 9.2 to 12.8, and due to strong alkalinity thereof, red mud is handled as designated waste.

[8] In the past, red mud was disposed of inefficiently, primarily through methods like underground landfill and ocean dumping. However, problems have arisen due to contamination of soil, surface water, and groundwater by corrosive leachate generated from large-scale red mud accumulation, and the cost of disposal is also rising. Furthermore, conventional treatment methods are insufficient to handle the increasing volume of red mud being generated.

[9] Red mud contains significant amounts of valuable metals such as iron (Fe), aluminum (Al), titanium (Ti), silicon (Si), gallium (Ga), and rare earth elements including as scandium (Sc) and yttrium (Y). Therefore, active research is ongoing to recycle red mud by recovering multiple valuable metals therefrom.

[10] For example, in order to recover iron from red mud, a method has been proposed in which a carbonaceous material (e.g., activated carbon, coal, lignite, etc.) as a reducing agent is added to red mud, followed by heat treatment to convert the iron ingredient contained in the form of hematite (Fe_2O_3) in red mud into magnetite (Fe_3O_4), which is then separated by magnetic separation and recovered (dry smelting process) (e.g., Steel Trans. 42 (5), 379-386 (2012), etc.). In addition, technologies for separating/recovering valuable metals by leaching the same through direct acid treatment of red mud have also been developed (U.S. Patent No. 9,023,301, etc.).

[11] However, in the dry smelting process, the loss of titanium and aluminum in slag is induced, while in the wet smelting process, highly corrosive and toxic concentrated inorganic acids are primarily used to directly dissolve the metals in red mud, which may cause secondary environmental contamination.

[12] In this way, the conventional treatment technologies described above have advantages and disadvantages, and it is also practically difficult to implement a single process for individually recovering major valuable metals such as iron, aluminum, titanium, etc. from red mud with high efficiency.

[13] In particular, red mud has a variety of compositions depending on the source (e.g., bauxite processing plant), and furthermore, the treatment of titanium contained in red mud is difficult, rendering the recovery thereof challenging.

[14] Therefore, a method is required to overcome the limitations of conventional technology and to easily separate and recover valuable metals, for example iron, aluminum, titanium, etc., from red mud with high efficiency and purity.

【Disclosure】

【Technical Problem】

[15] An embodiment of the present disclosure is to provide a process capable of recovering iron, aluminum, and titanium, which are major ingredients of red mud, with high purity and high efficiency within a single process.

【Technical Solution】

[16] An embodiment of the present disclosure provides a process for recovering valuable metals from red mud, comprising:

[17] a) providing red mud containing iron, aluminum, and titanium;

[18] b) separating a magnetic portion and a non-magnetic portion from each other through magnetic separation after reductive roasting of the red mud; and

[19] c) liquid leaching each of the magnetic portion and the non-magnetic portion, followed by recovering iron from a residue of the magnetic portion and titanium from a residue of the non-magnetic portion, while recovering aluminum from a leach solution from liquid leaching.

[20]

[21] According to an embodiment, in step c), aluminum may be recovered through precipitation by adding an acid to the leach solution.

[22] According to an embodiment, aluminum may be precipitated at pH adjusted in the range of 3 to 6, and may be recovered as a precipitate in the form of $AlPO_4$ using phosphoric acid (H_3PO_4) as the acid.

[23] According to an embodiment, sodium phosphate (Na_3PO_4) may be additionally added with phosphoric acid, in which the amount of sodium phosphate added may be adjusted such that a molar ratio of aluminum relative to sodium phosphate is 0.5 to 2.

[24] According to an embodiment, the precipitation of aluminum may be performed at 50 to 90°C for 0.1 to 5 hours.

[25] According to an embodiment, in step c), titanium may be recovered in the form of titania (TiO_2).

[26] According to an embodiment, the red mud may contain, on an elemental basis, 5 to 60 wt% of iron (Fe), 3 to 40 wt% of aluminum, 2 to 20 wt% of titanium, and balance.

[27] According to an embodiment, the content of the balance in the red mud may be 25 to 60 wt% on an elemental basis.

[28] According to an embodiment, LOI (loss on ignition) of the red mud may be 9 to 50 wt%.

[29] According to an embodiment, the reductive roasting in step b) may be performed in the presence of a carbon source as a reducing agent and at least one alkali metal salt selected from the group consisting of a sodium salt and a potassium salt as an additive.

[30] According to an embodiment, the carbon source may be at least one selected from the group consisting of coke, coal, charcoal, biochar, agricultural residue, waste paper, and

pulp processing waste, and

[31] the alkali metal salt may be at least one selected from the group consisting of sodium carbonate, sodium sulfate, borax ($Na_2B_4O_7$), potassium carbonate, potassium sulfate, and potassium bicarbonate.

[32] According to an embodiment, the weight ratio of the red mud to the alkali metal salt to the carbon source may be adjusted in the range of 8 to 20 : 2 to 8 : 1.

[33] According to an embodiment, the reductive roasting in step b) may be performed at a temperature adjusted in the range of 600 to 1,200°C

[34] According to an embodiment, the reductive roasting in step b) may be performed for 0.3 to 10 hours.

[35] According to an embodiment, the weight ratio of the magnetic portion to the non-magnetic portion separated in step b) may be 1 to 4 : 1.

[36] According to an embodiment, the liquid-to-solid (L/S) ratio during liquid leaching each of the magnetic portion and non-magnetic portion in step c) may be adjusted in the range of 1 to 50.

[37] According to an embodiment, the magnetic portion in step c) may contain at least 20 wt% of iron (on an elemental basis).

[38] According to an embodiment, the non-magnetic portion in step c) may contain at least 4 wt% of titanium (on an elemental basis).

[39] According to an embodiment, recovering titanium in step c) may comprise subjecting the non-magnetic portion to mixed sulfation roasting using sulfuric acid and sulfate.

[40] According to an embodiment, the weight ratio of sulfuric acid/sulfate during the mixed sulfation roasting may be adjusted in the range of 0.5 to 4.

[41] According to an embodiment, the sulfate may be at least one selected from the group consisting of sodium sulfate (Na_2SO_4), ammonium sulfate ($(NH_4)_2SO_4$), magnesium sulfate ($MgSO_4$), potassium sulfate (K_2SO_4), and barium sulfate ($BaSO_4$).

[42] According to an embodiment, the non-magnetic portion subjected to mixed sulfation roasting may be leached with sulfuric acid at a temperature of 70 to 150°C to form a titanium-containing leach solution, in which the concentration of sulfuric acid may be up to 10 M and the liquid-to-solid ratio may be adjusted in the range of 2 to 100.

[43] According to an embodiment, a titanium-containing precipitate is formed through titanium hydrolysis carried out by heating the titanium-containing leach solution to a temperature of 70 to 100°C and adjusting pH to less than 3.

[44] According to an embodiment, the titanium-containing precipitate may be

converted into titania by calcination in an oxygen-containing atmosphere at a temperature of 200 to 900°C for 0.5 to 10 hours.

【Advantageous effects】

[45] The process according to embodiments of the present disclosure can recover iron, aluminum, and titanium, respectively, which are valuable metals, from red mud discharged as a byproduct of bauxite processing, with high efficiency through a single process, and is particularly advantageous in terms of the environment and waste reduction because it is configured to be operable with relatively low energy consumption and low reagent usage. In addition, the process according to embodiments of the present disclosure provides the advantage of reducing carbon emissions compared to conventional smelting processes and also effectively suppressing the generation of secondary waste such as slag. Therefore, widespread commercialization is expected in the future.

【Description of Drawings】

[46] FIG. 1 shows a flowchart of a process according to one embodiment;

[47] FIG. 2 shows an X-ray diffraction (XRD) pattern of a red mud sample used in Examples;

[48] FIG. 3 shows an X-ray diffraction (XRD) pattern after reductive roasting performed without adding a reducing agent to the red mud sample (in which the mixing ratio (by weight) of coke to red mud is 1 : 10); and

[49] FIG. 4 shows an X-ray diffraction (XRD) pattern after reductive roasting performed under the addition of a reducing agent (sodium carbonate) to the red mud sample (in which the mixing ratio (by weight) of coke to sodium carbonate to red mud is 1 : 5 : 10).

【Best Mode】

[50] The present disclosure may be realized in its entirety based on the following description. It should be understood that the following description is given of preferred embodiments of the present disclosure, and the present disclosure is not necessarily limited thereto. In addition, the accompanying drawings are provided to aid understanding of the present disclosure, and the present disclosure is not limited thereto, and details regarding individual components may be appropriately understood by the specific intent of the related description described below.

[51] Terms used herein may be defined as follows.

[52] “Red mud” may be understood to mean a red-colored byproduct generated in the process of manufacturing alumina from bauxite.

[53] “Reductive roasting” may mean a process in which a metal oxide is reduced into a metal or a compound of lower oxidation number in a reducing atmosphere.

[54] “Magnetic separation” may broadly mean a process of separating a mixture using magnets, for example a process of applying an electromagnetic field to a mixture containing at least one magnetic ingredient and at least one non-magnetic ingredient to separate the magnetic ingredient from the other ingredient.

[55] “Leaching” may mean a process of removing a material by dissolution in a percolating liquid.

[56] Herein, when a numerical range is specified as a lower limit and/or an upper limit, it may be understood that any sub-combination within the numerical range is also disclosed. For example, a description of “1 to 5” may include 1, 2, 3, 4 and 5, as well as any sub-combination therebetween.

[57] When any component or member is described herein as being “connected” to another component or member, it may be understood that it includes not only cases where it is directly connected to the other component or member, but also cases where it is connected via a further component or member interposed therebetween, unless stated otherwise.

[58] Similarly, the term “contact” may be understood to include not only direct contact, but also contact by the intervention of a further component or member.

[59] When any component is “included,” another component may be further included, unless specified otherwise.

[60] According to an embodiment of the present disclosure, a process is provided for recovering valuable metals, for example iron (Fe), aluminum (Al), and titanium (Ti), from red mud, which is discharged as a byproduct during bauxite processing, through a combination of dry treatment and wet treatment in a physical and chemical manner.

[61] A flowchart for an overall process for individually separating and recovering iron, aluminum, and titanium from red mud according to one embodiment is shown in FIG. 1.

[62] Referring to the above drawing, red mud is provided as a starting material. Red mud, also referred to as bauxite residue, contains typically iron (Fe), aluminum (Al), and titanium (Ti), as well as silicon (Si), sodium (Na), and calcium (Ca), primarily in the form of oxides, with trace amounts of balance elements.

[63] Red mud is typically obtained in the form of fine grains, and before processing, a

washing process (e.g., water washing) may be performed at least once to remove impurities such as various dusts attached to the particle surface other than ingredients inherently contained in red mud and to lower high alkalinity. As an example, the amount of water used in water washing may be adjusted in the range of, for example, about 5 to 30% (w/v), for example about 10 to 25% (w/v), and the water washing temperature may be adjusted in the range of, for example, about 50 to 100°C, for example about 70 to 95°C, for example about 80 to 90°C.

[64] After the water washing as described above, a drying process may be performed, and optionally, a filtration process may be conducted before drying to separate solids, and the filtrate thus obtained may be reused in the water washing process. Moreover, drying may be performed by a typical process known in the art, for example, heat (hot air) drying, reduced pressure drying, etc.

[65] Referring again to FIG. 1, the particle size of red mud may be determined in consideration of the fact that it is a factor affecting the treatment process described later (e.g., filtration, transport, etc.), and may be, for example, in the range of about 40 μm or less, for example about 0.8 to 20 μm, for example about 3 to 10 μm, which may be understood as illustrative.

[66] According to an embodiment, the content of iron (Fe) in red mud may be, for example, in the range of about 5 to 60 wt%, for example about 15 to 50 wt%, for example about 20 to 40 wt%, on an elemental basis. As such, iron (Fe) may be mostly present in the form of hematite (Fe_2O_3).

[67] Also, the content of aluminum (Al) in red mud may be, for example, in the range of about 3 to 40 wt%, for example about 5 to 20 wt%, for example about 7 to 15 wt%, on an elemental basis.

[68] In addition, the content of titanium (Ti) in red mud may be, for example, in the range of about 2 to 20 wt%, for example about 3 to 10 wt%, for example about 5 to 7 wt%, on an elemental basis.

[69] Meanwhile, red mud may contain balance elements in addition to the aforementioned ingredients, and examples of the balance elements may include silicon (Si), calcium (Ca), sodium (Na), carbon (C), oxygen (O), etc., and the types and amounts of the balance elements may vary depending on the source of the red mud, etc.

[70] According to an embodiment, the balance content in red mud may be, for example, in the range of about 25 to 60 wt%, for example about 30 to 55 wt%, for example about 40 to 50 wt%, on an elemental basis.

[71] Also, according to an embodiment, LOI (loss on ignition) of red mud may be, for example, in the range of about 9 to 50 wt%, for example about 20 to 45 wt%, for example about 25 to 40 wt%.

[72]

[73] Reductive roasting

[74] In an embodiment, red mud is roasted in the presence of a carbon source as a reducing agent (reductive roasting). The roasting treatment serves to reduce hematite (Fe_2O_3) into magnetite (Fe_3O_4) according to Scheme 1 below, and through additional reduction, metallic iron (Fe) may be obtained as represented in Schemes 2 and 3 below.

[75]

[76] [Scheme 1]

[77] $3Fe_2O_3 + C \rightarrow 2Fe_3O_4 + CO$

[78] [Scheme 2]

[79] $Fe_3O_4 + C \rightarrow 3FeO + CO$

[80] [Scheme 3]

[81] $FeO + C \rightarrow Fe + CO$

[82]

[83] In this regard, the carbon source is not particularly limited so long as it has reducing ability. For example, the carbon source may be at least one selected from among coke, coal, charcoal, biochar, agricultural residue, waste paper, pulp processing waste, etc. According to certain embodiments, coke may be used as a carbon source, which may be advantageous because coke has a higher carbon calorific value (32 MJ/kg) than coal (24 MJ/kg) and has no smoking properties.

[84] Meanwhile, in the embodiment, red mud may be subjected to reductive roasting in the presence of an alkali metal salt in combination with a carbon source as a reducing agent. In this regard, according to an embodiment, the alkali metal salt may be at least one selected from the group consisting of a sodium salt and a potassium salt, and for example, may be a sodium salt.

[85] In the embodiment, red mud is subjected to reductive roasting using a carbon source as a reducing agent in the presence of an alkali metal salt (e.g., a sodium salt). As such, reductive roasting may be carried out by placing red mud, a carbon source, and an alkali metal salt in a reduction furnace and raising the temperature. According to an embodiment, a furnace known in the art may be used as a device for reductive roasting, and for example, at least one selected from among a natural gas furnace, an oil furnace, an electric furnace, a

waste oil furnace, a wood burning furnace, a dual fuel furnace, a single-stage furnace, a two-stage furnace, a bell furnace, a box furnace, a forging furnace, a pit furnace, a quenching furnace, a rotary furnace, a tempering furnace, etc. may be used.

[86] When reductive roasting is performed without adding an alkali metal salt, for example a sodium salt, sodium aluminum silicate ($AlNa_{12}SiO_5$), maghemite (Fe_2O_3, γ-Fe_2O_3), perovskite, etc. may be formed, whereas when an alkali metal salt is added, conversion of Fe_2O_3 in the red mud into Fe_3O_4 may be promoted.

[87] In this regard, the sodium salt among alkali metal salts is an additive capable of imparting surface modification properties, and addition thereof may promote reduction during the reductive roasting process. According to an embodiment, examples of the sodium salt may include sodium carbonate, sodium sulfate, borax ($Na_2B_4O_7$), etc., and examples of the potassium salt may include potassium carbonate, potassium sulfate, potassium bicarbonate, etc., and at least one of the types listed above may be used. According to certain embodiments, as the sodium salt, sodium carbonate, may be used because it has a small ionic radius (about 1.02 Å), which may contribute to effectively converting hematite into magnetite during the reductive roasting process.

[88] According to an embodiment, the weight ratio of the red mud to the alkali metal salt to the carbon source during the reductive roasting may be, for example, in the range of about 8 to 20 : about 2 to 8 : 1, for example about 9 to 15 : about 3 to 7 : 1, for example about 10 to 12 : about 4 to 6 : 1. In this regard, if the content ratio of the carbon source is too low or high, reaction may not be sufficient or excessive reaction may occur, and if the content ratio of the alkali metal salt is too low or high, reaction may not be sufficient or excessive reaction may occur. Hence, it may be advantageous to appropriately control the content ratio within the above-mentioned range. However, the range of the content ratio described above is not necessarily limited thereto as it may vary depending on the type of carbon source or alkali metal salt used, the composition of red mud, etc.

[89] According to an embodiment, reductive roasting may be performed under elevated temperature conditions for reduction of iron (Fe) in the red mud, and the reductive roasting temperature may be determined in the range of, for example, about 600 to 1,200°C, for example about 700 to 1,100°C, for example about 800 to 1,000°C, for example about 850 to 950°C. Also, the reductive roasting time may be determined in consideration of reaction efficiency, etc., and may be, for example, in the range of about 0.3 to 10 hours, for example about 0.5 to 6 hours, for example about 0.8 to 3 hours.

[90] Through the reductive roasting, for example, at least about 40 wt%, for example

at least about 50 wt%, for example at least about 55 wt%, for example at least about 60 wt% of Fe_2O_3 in red mud may be reduced into Fe_3O_4 and/or elemental iron (Fe). Accordingly, the roasted red mud is present in the form of a mixture of magnetic and non-magnetic portions.

[91]

[92] Magnetic separation

[93] Referring again to FIG. 1, the red mud subjected to reductive roasting (heat treatment) may be separated into a magnetic portion and a non-magnetic portion, respectively, through magnetic separation based on the principle of magnetic induction. An apparatus used for magnetic separation is not limited to a specific type, and any magnetic separation apparatus known in the art may be used. Specifically, magnetic separation may be based on a mechanism of passing a powder of reductive roasted red mud through a magnetic field to separate a magnetic portion and a non-magnetic portion. In this regard, the magnitude of the magnetic field applied during magnetic selection may be set in consideration of magnetic induction properties, etc., and may be adjusted in the range of, for example, about 1,200 to 10,000 Gauss, for example about 2,000 to 8,000 Gauss, for example about 4,000 to 6,000 Gauss, which may be understood as illustrative.

[94] According to an embodiment, the ratio (by weight) of the magnetic portion to the non-magnetic portion separated by magnetic separation may vary depending on the composition and properties of red mud as the starting material, reductive roasting treatment conditions, etc., and may be, for example, in the range of about 1 to 4 : 1, for example about 1.2 to 3 : 1, for example about 1.4 to 2 : 1, but is not limited thereto.

[95] According to an embodiment, the iron content in the magnetic portion may be higher than that in the initial red mud, and may be, for example, at least about 20 wt%, for example at least about 25 wt%, for example at least about 30 wt%, on an elemental basis. According to certain embodiments, the iron content in the magnetic portion may be, for example, about 40 to 80 wt%, for example about 50 to 75 wt%, for example about 60 to 70 wt%, on an elemental basis.

[96] In contrast, the titanium content in the magnetic portion may be much lower than that in the initial red mud, because the titanium ingredient tends to be concentrated in the non-magnetic portion. In this regard, the titanium content (on an elemental basis) in the magnetic portion may be, for example, about 15 wt% or less, for example about 10 wt% or less, for example about 5 wt% or less, for example about 2 wt% or less. According to certain embodiments, the titanium content (on an elemental basis) in the magnetic portion may be, for example, about 1 wt% or less, for example about 0.8 wt% or less, for example about 0.5

wt% or less.

[97] Meanwhile, the iron content in the non-magnetic portion may be lower than that in the initial red mud, and may be, for example, about 60 wt% or less, for example about 50 wt% or less, for example about 30 wt% or less, for example about 20 wt% or less, on an elemental basis. According to certain embodiments, the iron content in the non-magnetic portion may be, for example, about 15 wt% or less, for example about 10 wt% or less, for example about 8 wt% or less, on an elemental basis.

[98] In contrast, the titanium content in the non-magnetic portion may be higher than that in the initial red mud, and may be, for example, at least about 4 wt% (on an elemental basis), for example about 5 to 40 wt%, for example about 7 to 30 wt%.

[99] Aluminum may be contained in a balanced amount in both the magnetic portion and the non-magnetic portion, and the reason why aluminum is not concentrated in the magnetic portion or the non-magnetic portion by magnetic separation may be deemed to be due to factors such as the magnitude of the external magnetic field, etc. In each of the magnetic and non-magnetic portions, the aluminum content (on an elemental basis) may be determined in the range of, for example, about 3 to 10 wt%, for example about 4 to 8 wt%, for example about 5 to 7 wt%, which may be understood as illustrative. As an example, aluminum in the magnetic and non-magnetic portions may be typically present in the form of sodium aluminate, free alkali metal (e.g., sodium cation), etc., which may be a soluble sodium ingredient that may be dissolved relatively easily during liquid leaching described below.

[100]

[101] Liquid leaching

[102] In the embodiment, each of the magnetic and non-magnetic portions of the red mud separated by magnetic separation is subjected to liquid leaching, so that most of the aluminum contained therein may be removed (i.e., aluminum is dissolved in a leach solution). As such, the leaching medium used for liquid leaching may be an aqueous medium, for example, water. According to certain embodiments, leaching using an aqueous medium without adding an acid or a base (i.e., water leaching) may be performed.

[103] The leaching efficiency of aluminum may be, for example, at least about 80% (on an elemental basis), and, the leaching efficiency of aluminum in the magnetic portion may be, for example, about 90 to 99%, for example about 93 to 96%, while the leaching efficiency of aluminum in the non-magnetic portion may be, for example, about 82 to 92%, for example about 83 to 87%, which may be understood as illustrative.

[104] According to an embodiment, the liquid-to-solid (L/S) ratio during leaching of

each of the magnetic and non-magnetic portions may be, for example, in the range of about 50 or less, for example about 1 to 20, for example about 3 to 15, for example about 5 to 12.

[105] In addition, liquid leaching may be performed under elevated temperature conditions, and the leaching temperature may be adjusted in the range of, for example, about 50 to 100°C, for example about 70 to 98°C, for example about 85 to 95°C. Also, the water leaching time may be, for example, in the range of about 15 minutes to about 8 hours, for example about 0.5 to 4 hours, for example about 1 to 2 hours.

[106] Through the liquid leaching process, a leach solution (e.g., an aqueous leach solution) containing aluminum ions (or compounds), and residues of the magnetic and non-magnetic portions may be formed. As in the illuminated embodiment, a solid-liquid separation technique known in the art, such as filtration, centrifugation, sedimentation, decantation, evaporation, etc., may be employed to obtain the leach solution and the residue of each of the magnetic and non-magnetic portions.

[107] In this regard, the concentration of aluminum in the leach solution for each of the magnetic and non-magnetic portions may be determined in the range of, for example, about 3 to 20 g/L, for example about 4 to 18 g/L, for example about 5 to 15 g/L, but is not limited thereto.

[108] According to an embodiment, the iron content in the residue of the magnetic portion after leaching may be significantly increased compared to before leaching, and may be, for example, in the range of at least about 20 wt% (on an elemental basis), for example at least about 25 wt%, for example about 30 to 70 wt%, based on the residue of the magnetic portion. In particular, the relative amount (on an elemental basis) of iron in the residue of the magnetic portion may be, for example, at least about 80 wt%, for example at least about 85 wt%, for example at least about 90 wt%, compared to the iron content in the initial red mud, resulting in high-grade iron.

[109] In contrast, respective contents of iron and titanium in the residue of the non-magnetic portion may not significantly change compared to before leaching, and compared to the non-magnetic portion before leaching, the rate of change in the iron content may be, for example, about 12% or less (for example about 10% or less, for example about 6% or less), and the rate of change in the titanium content may be, for example, about 15% or less (for example about 10% or less, for example about 8% or less).

[110] According to an embodiment, respective contents of aluminum and alkali metal (e.g., sodium) in the residue of each of the magnetic and non-magnetic portions may be significantly decreased, and the rate of change in the aluminum content may be, for example,

at least about 80% (for example at least about 85%, for example at least about 90%), and the rate of change in the alkali metal content may be, for example, at least about 80% (e.g., at least about 85%, for example at least about 90%), compared to before liquid leaching, which may be understood as illustrative.

[111]

[112] Recovery of aluminum

[113] In the embodiment, recovering aluminum from the leach solution of each of the magnetic and non-magnetic portions or a combination (mixture) thereof is performed.

[114] To this end, aluminum may be precipitated by adding an acid to the leach solution (e.g., an aluminum-containing aqueous leach solution). An example of the acid used for precipitation of aluminum may include at least one selected from among phosphoric acid (H_3PO_4), sulfuric acid (H_2SO_4), hydrochloric acid (HCl), nitric acid (HNO_3), etc., and, for example, phosphoric acid may be used.

[115] Specifically, the formation of a precipitate by the reaction between aluminum and phosphoric acid in the leach solution may be as follows. First, aluminum ions react with phosphoric acid to form soluble intermediates (compounds), such as $AlH_2PO_4^{2+}$, $AlHPO^{4+}$, etc., which are subsequently converted into insoluble $AlPO_4$ while releasing hydrogen ions.

[116] According to an embodiment, when adding phosphoric acid to aluminum, sodium phosphate (Na_3PO_4) may be additionally added in combination with phosphoric acid. As such, the amount of sodium phosphate added may be determined in consideration of the molar concentration of aluminum in the solution. For example, the molar ratio of aluminum relative to sodium phosphate (Al/Na_2PO_4) may be adjusted in the range of about 0.5 to 2, for example about 1 to 1.5. According to certain embodiments, sodium phosphate may be added in a stoichiometric amount.

[117] According to an embodiment, during the precipitation reaction of aluminum in the leach solution, an acid may be added such that the pH is in the range of, for example, about 3 to 6, for example about 3.5 to 5.5, for example about 4 to 5. Furthermore, an acid may be added in the form of an aqueous solution, in which case the acid concentration may be adjusted in the range of, for example, about 5 to 20% (v/v), for example about 7 to 15% (v/v), for example about 9 to 12% (v/v), but the present disclosure is not limited thereto.

[118] Also, the temperature during the precipitation of aluminum may be determined in the range of, for example, about 50 to 90°C, for example about 55 to 85°C, for example about 60 to 80°C, and the precipitation time may be adjusted in the range of, for example, about 0.1 to 5 hours, for example about 0.5 to 4 hours, for example about 1 to 3 hours.

[119] Through the process described above, most of the aluminum in the leach solution may be precipitated, and the aluminum precipitation efficiency in the leach solution may be, for example, at least about 95 wt%, for example at least about 99 wt%, for example at least about 99.9 wt%. After aluminum is precipitated in this way, the solid $AlPO_4$ may be recovered using a solid-liquid separation process known in the art. Such a solid-liquid separation process may be at least one selected from among, for example, filtration, centrifugation, decantation, evaporation, etc., and for example, filtration may be employed.

[120] Also, the purity of the $AlPO_4$ precipitate may be, for example, at least about 99%, for example at least about 99.5%, for example at least about 99.8%, for example at least about 99.9%.

[121]

[122] Separation/recovery and utilization of iron

[123] The residue of the magnetic portion obtained after liquid leaching contains a high concentration of iron, and in the embodiment, the residue of the magnetic portion may be subjected to a recovery process known in the art, for example, a smelting process to form metallic iron, which is then separated from slag and recovered. Since details of this process are well known in the art, a description of the details is omitted.

[124]

[125] Recovery of titanium

[126] The residue of the non-magnetic portion obtained after liquid leaching contains a significant amount of titanium, and titanium in the residue may be recovered by converting the same into titania.

[127] To this end, in the embodiment, the residue is subjected to mixed sulfation roasting. In this regard, mixed sulfation roasting may be conducted to convert titanium into a form that is easily leachable in subsequent processes.

[128] According to an embodiment, mixed sulfation roasting may be performed using sulfuric acid (H_2SO_4) and sulfate, by mixing the residue of the non-magnetic portion with sulfuric acid and sulfate followed by roasting under elevated temperature conditions. In this regard, the sulfate may be at least one selected from among sodium sulfate (Na_2SO_4), ammonium sulfate ($(NH_4)_2SO_4$), magnesium sulfate ($MgSO_4$), potassium sulfate (K_2SO_4), barium sulfate ($BaSO_4$), etc. It may be advantageous to use sodium sulfate having good reaction efficiency. In this way, titanium in the residue of the non-magnetic portion may be converted into a water-soluble form through mixed sulfation roasting. As such, during mixed sulfation roasting, sulfuric acid may be used in a concentrated form (e.g., about 98% or more).

[129] According to an embodiment, the weight ratio of sulfuric acid/sulfate during the mixed sulfation roasting may be adjusted in the range of, for example, about 0.5 to 4, for example about 1.5 to 2.5, for example about 1 to 2.

[130] According to an embodiment, the ratio (by weight) of the sulfuric acid and sulfate to the residue of the non-magnetic portion may be adjusted in consideration of the relative ratio with the reactants, and may be, for example, about 0.5 to 3 : 1, for example about 0.7 to 2 : 1, for example about 0.9 to 1.5 : 1.

[131] In addition, the mixed sulfation roasting temperature may be adjusted to a level sufficient to break down the refractory matrix of the titanium compound in the residue of the non-magnetic portion, and may be, for example, in the range of about 180 to 400°C, for example about 200 to 300°C, or about 220 to 270°C. Also, the mixed sulfation roasting time may be adjusted in the range of, for example, about 0.5 to 5 hours, for example about 1 to 4 hours, for example about 1.5 to 3 hours, which may be understood as illustrative.

[132] In the embodiment, titanium in a water-soluble form in the roasted residue may be leached by adding an aqueous medium or an acid solution (e.g., an aqueous sulfuric acid solution) after mixed sulfation roasting. As such, the concentration of the sulfuric acid solution may be adjusted in the range of, for example, up to 10 M, for example about 1 to 5 M, for example about 1.5 to 3 M. Also, the liquid-to-solid ratio during the leaching process may be adjusted in the range of, for example, about 2 to 100, for example about 3 to 20, for example about 5 to 10. In addition, leaching may be performed at a temperature of, for example, about 70 to 150°C, for example about 90 to 140°C, for example about 100 to 130°C, for about 0.2 to 5 hours, for example about 0.5 to 4 hours, for example about 0.8 to 3 hours, which may be understood as illustrative. As such, the leaching efficiency of titanium may be, for example, at least about 90%, for example at least about 95%, for example at least about 99%.

[133] Through the leaching process, a leach solution containing a significant amount of titanium (e.g., titanium sulfate) may be formed, and the titanium concentration in the leach solution may be, for example, in the range of at least about 3 to 10 g/L, for example at least about 4 to 9 g/L, for example about 6 to 8 g/L.

[134] Referring again to FIG. 1, a hydrolysis process may be performed to precipitate titanium in the titanium-containing leach solution. The pH during the hydrolysis process may be adjusted taking into account the extent of precipitation, and the pH may be, for example, in the range of less than about 3, for example about 1 to 2, for example about 1.2 to 1.8. As such, hydrolysis reaction may be carried out under elevated temperature conditions, and the

hydrolysis temperature may be adjusted in the range of, for example, about 70 to 100°C, for example about 75 to 95°C, for example about 85 to 92°C.

[135] Through the hydrolysis process, the titanium compound (e.g., titanium sulfate) in the leach solution may be precipitated in the form of metatitanic acid according to Scheme 2 below.

[136]

[137] [Scheme 2]

[138] $TiOSO_4 + 2H_2O \rightarrow H_2TiO_3\downarrow + H_2SO_4$

[139]

[140] According to an embodiment, at least about 90 wt%, for example at least about 92 wt%, for example at least about 95 wt% of titanium in the leach solution may be converted into a precipitate.

[141] Meanwhile, after titanium precipitation involving hydrolysis reaction, the metatitanic acid precipitate may be recovered using a solid-liquid separation process known in the art. Such a solid-liquid separation process may be at least one selected from among, for example, filtration, centrifugation, decantation, evaporation, etc., and for example, filtration may be employed.

[142] The metatitanic acid precipitate thus obtained may be optionally dried, and may then be converted into titania through heat treatment in an oxygen-containing atmosphere, e.g., calcination.

[143] According to an embodiment, the calcination temperature may be, for example, in the range of about 200 to 900°C, for example about 400 to 800°C, for example about 500 to 700°C. Also, the calcination time may be adjusted in the range of, for example, about 0.5 to 10 hours, for example about 1 to 5 hours, for example about 2 to 4 hours.

[144] The purity of the titania converted through the calcination treatment may be, for example, at least 95%, for example at least about 99%, for example at least about 99.5%. Also, the resultant titania may be in a rutile form.

[145] As described above, according to the present disclosure, valuable metals including iron, aluminum, and titanium contained in red mud as a byproduct of bauxite processing may be separated and recovered by a single treatment process. The present disclosure is noteworthy in that it provides a method of recovering each of at least three valuable metals with high efficiency and high purity, compared to conventionally known dry or wet processes.

【Mode for Disclosure】

[146] The present disclosure may be more clearly understood by the following examples, which are merely set forth to illustrate the present disclosure and are not to be construed as limiting the scope of the present disclosure.

[147]

[148] **Examples**

[149]

[150] **A. Materials**

[151] The materials used in examples are as shown in Table 1 below.

[152]

[153] [Table 1]

Material	Manufacturer
Na_2CO_3	Sigma-Aldrich
Coke powder	
H_3PO_4	
Na_3PO_4	
H_2SO_4	
Na_2SO_4	

[154]

[155] **B. Analysis method**

[156] The analysis performed in examples was as follows.

[157]

[158] - XRD pattern was analyzed using X'pert PRO PANalytical.

[159] - The composition was analyzed by inductively coupled plasma-induced emission spectroscopy using iCAP 7400 Duo (Thermo Scientific, USA).

[160]

[161] Example 1

[162] In this example, red mud discharged as a byproduct from a commercial alumina manufacturer was obtained and used as a starting material. To achieve homogenization and eliminate excess alkali removable by washing, 1 kg of red mud cake was washed with water to a bulk density of 20% (w/v) at 90°C for 1 hour in a glass beaker under mechanical stirring at 300 rpm. This was repeated for three different batches, followed by hot air drying overnight at 105°C, and a sufficient amount of sample was collected. The dried red mud was ground by hand, mixed properly, and prepared into a uniform powder to be used in the subsequent set of experiments. Weight loss was measured to be 42.02% from the initial cake used in water washing. After aqua regia decomposition, wet chemical analysis (compositional

analysis) was performed using ICP (inductively coupled plasma)-spectroscopy (ICP-OES, iCAP 7400 Duo, Thermo Scientific), and the results are shown in Table 2 below.

[163]

[164] [Table 2]

Major element	Fe	Al	Na	Si	Ti	C	Balance (including oxygen)
(wt%)	24.9	9.4	8.5	6.2	3.5	2.3	45.2

[165]

[166] According to the table above, red mud contains the major ingredients of iron, aluminum, sodium, silicon, titanium and carbon, as well as multiple balance elements, and the balance elements were analyzed to contain trace amounts of calcium, magnesium, yttrium, niobium, strontium, etc., in addition to oxygen.

[167] Also, the physical properties of red mud were evaluated using XRD (X-ray diffraction) analysis, and the results are shown in FIG. 2.

[168] Referring to the above drawing, cancrinite [$Na_7(Al_5Si_7O_{24})CO_3.3H_2O$], gibbsite [$Al(OH)_3$], anatase [$TiO_2$], quartz [$SiO_2$], hematite [$Fe_2O_3$], and sodalite [$Na_6(Al_6Si_6O_{24})2NaF.xH_2O$] were found to be the major mineral phases in red mud.

[169]

[170] Example 2

[171] 50 g of red mud powder and 25 g of Na_2CO_3 powder were mixed with coke powder at various weight ratios (coke powder: varied in the range of 1 to 5 g).

[172] In another experiment, 50 g of red mud and 5 g of coke powder were mixed with Na_2CO_3 powder at various weight ratios (Na_2CO_3 powder: varied in the range of 0 to 25 g).

[173] Reductive roasting was performed in an electric furnace at a heating rate of 10 °C/min at 900°C in an inert atmosphere. As such, the roasting was performed for 1 hour after reaching 900°C.

[174] The phase transformation of reductive roasted samples was analyzed using XRD, and the results are shown in FIGs. 3 and 4.

[175] Referring to FIG. 3, it was confirmed that sodium aluminum silicate, maghemite, and perovskite phases were formed by reductive roasting performed without adding Na_2CO_3.

[176] In contrast, according to FIG. 4, for the sample roasted in the presence of 5 g of coke and 25 g of Na_2CO_3, it was confirmed that hematite was converted into magnetite, and the other major element was found to be a sodium compound.

[177]

[178] Example 3

[179] Red mud was subjected to reductive roasting at 900°C for 1 hour as in Example 2, resulting in a 27.5% weight loss based on the initial red mud. Thereafter, 58 g of the reductive roasted sample was ground in a mortar and pestle, followed by magnetic separation.

[180] Respective fractions of magnetic and non-magnetic portions obtained from the magnetic separation were 64.7% and 35.3%. The compositions of the two portions are shown in Table 3 below.

[181]

[182] [Table 3]

Classification	Content (wt%)	Element (wt%)					
		Fe	Al	Na	Si	Ti	C
Magnetic portion	64.7	30.3	6.9	22.1	5.3	0.3	Not found
Non-magnetic portion	35.3	5.6	5.8	17.7	5.4	7.2	Not found

[183]

[184] Referring to the table above, it was confirmed that >91% of the iron content in the tested sample was separated through magnetic separation. However, the iron grade resulting from magnetic separation was found to be somewhat low due to the presence of other elements.

[185]

[186] Example 4

[187] In order to improve the iron grade, 37.5 g of the sample of the magnetic portion obtained through magnetic separation in Example 3 was leached in an aqueous medium (water) under conditions of a pulp density of 10% (w/v) (liquid-to-solid ratio: 10) and a temperature of 90°C for 1 hour. Afterwards, the solid collected through filtration using Whatman No. 42 filter paper (pore size: 2.5 μm) was dried overnight under hot air conditions to obtain an aqueous leaching residue. The analysis results showed that the obtained residue had a weight loss of about 48.6 wt% compared to the sample before water leaching. This weight loss is deemed to be due to the dissolution of soluble aluminum compounds such as sodium aluminate and other free alkali metals (primarily sodium).

[188] After water leaching of the magnetic portion, elemental analysis of the residue was performed, and the results are shown in Table 4 below.

[189]

[190] [Table 4]

Major element	Fe	Al	Na	Si	Ti
Content (wt%)	58.5	<0.7	<0.2	<0.2	<0.6

[191]

[192] According to the table above, the elemental analysis results for the aqueous leaching residue of the magnetic portion showed that the iron grade significantly increased from less than 25% to 58.5%. In particular, the recovered high-grade iron accounted for about 91 wt% of the total iron content in the initial red mud fed to the reductive roasting process.

[193]

[194] Example 5

[195] The sample of the non-magnetic portion obtained in Example 3 (containing 5.8 wt% aluminum) was subjected to water leaching under the same conditions as in Example 4 (a pulp density of 10% (w/v), a temperature of 90°C, and 1 hour). Afterwards, the solid collected through filtration using Whatman No. 42 filter paper (pore size: 2.5 μm) was dried overnight under hot air conditions to obtain an aqueous leaching residue. The analysis results showed that the obtained residue had a weight loss of about 17.1 wt% compared to the sample before water leaching. This weight loss is deemed to be due to the dissolution of soluble aluminum compounds such as sodium aluminate and other free alkali metals (primarily sodium).

[196] After water leaching of the non-magnetic portion, elemental analysis of the residue was performed, and the results are shown in Table 5 below.

[197]

[198] [Table 5]

Major element	Fe	Al	Na	Si	Ti
Content (wt%)	5.9	1.2	0.8	5.6	7.8

[199]

[200] According to the table above, the elemental analysis results for the aqueous leaching residue of the non-magnetic portion showed that the titanium grade increased from 3.5% to 7.8%.

[201]

[202] Example 6

[203] Aluminum was recovered by mixing 360 ml of the leach solution (containing 6.8 g/L aluminum) obtained in Example 4 and 165 ml of the leach solution (containing 5.93 g/L aluminum) obtained in Example 5.

[204] The pH of the mixed solution was adjusted to 4.7 under stirring conditions using a 10% (v/v) H_3PO_4 solution, and then Na_3PO_4 was added in a stoichiometric amount, followed by precipitation reaction for 1 hour in a solution heated to 70°C, forming a white precipitate of $AlPO_4$.

[205] The slurry containing the precipitate was filtered under high temperature conditions, and the separated precipitate was washed twice with hot water and dried to obtain $AlPO_4$. Elemental analysis was performed on the aqueous leach solution of the magnetic portion, the aqueous leach solution of the non-magnetic portion, the filtrate after precipitation, and the precipitate after drying, and the results are shown in Table 6 below.

[206]

[207] [Table 6]

Classification	Volume/weight	Al concentration	Absolute amount of Al	Precipitation efficiency	Product purity
Leach solution of magnetic portion	360 ml	6.80 g/L	2.45 g	-	-
Leach solution of non-magnetic portion	165 ml	5.93 g/L	0.98 g	-	-
Filtrate after precipitation	505 ml	0.06 g/L	0.03 g	99.12%	-
$AlPO_4$ precipitate after drying	15.35 g	22.1%	3.39 g	98.83%	99.89%

[208]

[209] According to the table above, 15.3 g of $AlPO_4$ precipitate was obtained with a precipitation efficiency of 99.12%. Also, chemical analysis results showed that the final product (i.e., $AlPO_4$) having a purity of 99.1% was obtained with a recovery efficiency of approximately 83% relative to the aluminum content in the initial red mud.

[210]

[211] Example 7

[212] 17 g of the titanium-containing residue obtained through water leaching of the non-magnetic portion (containing 7.8 wt% Ti) in Example 5 was roasted with a concentrated sulfate mixture (H_2SO_4 + Na_2SO_4) (mixed sulfation roasting). As such, a sulfate source was prepared at a H_2SO_4–to-Na_2SO_4 ratio of 2 : 1 (by weight). The sulfate mixture was placed in a porcelain crucible, and a paste was prepared with the titanium residue at a mixture-to-residue ratio of 1 : 1.

[213] Afterwards, the crucible was roasted for 2 hours in an electric furnace preheated to 240°C, and then the crucible was cooled inside the electric furnace.

[214] The cooled crucible was washed using 170 ml of 2 M H_2SO_4 solution in a 250 ml leaching reactor, maintaining a pulp density of 10% (w/v) (liquid-to-solid ratio: 10). The slurry was stirred for 1 hour at a boiling temperature of 120°C under a closed system equipped

with a condenser. Afterwards, the slurry was cooled to room temperature (about 25°C) and a filtrate was recovered.

[215] 200 ml of the filtrate (containing 6.02 g/L Ti) was heated to 90°C, after which the pH in the solution was adjusted to 1.5 for titanium hydrolysis.

[216] After precipitation for 1 hour, a titanium precipitate was recovered with a precipitation efficiency of 95% through filtration, and the precipitate was calcined in an electric furnace at 600°C for 2 hours, thereby recovering titania (2.2 g) having a purity of >99.5%.

[217] Simple modifications or changes of the present disclosure may be easily used by those skilled in the art, and all such modifications or changes may be considered to be included in the scope of the present disclosure.

【CLAIMS】

【Claim 1】 A process for recovering valuable metals from red mud, comprising:

a) providing red mud containing iron, aluminum, and titanium;

b) separating a magnetic portion and a non-magnetic portion from each other through magnetic separation after reductive roasting of the red mud; and

c) liquid leaching each of the magnetic portion and the non-magnetic portion, followed by recovering iron from a residue of the magnetic portion and titanium from a residue of the non-magnetic portion, while recovering aluminum from a leach solution from liquid leaching.

【Claim 2】 The process of claim 1, wherein, in step c), aluminum is recovered through precipitation by adding an acid to the leach solution.

【Claim 3】 The process of claim 2, wherein aluminum is precipitated at pH adjusted in a range of 3 to 6, and is recovered as a precipitate in form of $AlPO_4$ using phosphoric acid (H_3PO_4) as the acid.

【Claim 4】 The process of claim 3, wherein sodium phosphate (Na_3PO_4) is additionally added with phosphoric acid, in which an amount of sodium phosphate added is adjusted such that a molar ratio of aluminum relative to sodium phosphate is 0.5 to 2.

【Claim 5】 The process of claim 2, wherein precipitation of aluminum is performed at 50 to 90°C for 0.1 to 5 hours.

【Claim 6】 The process of claim 1, wherein, in step c), titanium is recovered in form of titania (TiO_2).

【Claim 7】 The process of claim 1, wherein the red mud contains, on an elemental basis, 5 to 60 wt% of iron (Fe), 3 to 40 wt% of aluminum, 2 to 20 wt% of titanium, and balance.

【Claim 8】The process of claim 7, wherein a content of the balance in the red mud is 25 to 60 wt% on an elemental basis.

【Claim 9】The process of claim 1, wherein LOI (loss on ignition) of the red mud is 9 to 50 wt%.

【Claim 10】The process of claim 1, wherein the reductive roasting in step b) is performed in presence of a carbon source as a reducing agent and at least one alkali metal salt selected from the group consisting of a sodium salt and a potassium salt as an additive.

【Claim 11】The process of claim 10, wherein:

the carbon source is at least one selected from the group consisting of coke, coal, charcoal, biochar, agricultural residue, waste paper, and pulp processing waste, and

the alkali metal salt is at least one selected from the group consisting of sodium carbonate, sodium sulfate, borax ($Na_2B_4O_7$), potassium carbonate, potassium sulfate, and potassium bicarbonate.

【Claim 12】The process of claim 10, wherein a weight ratio of the red mud to the alkali metal salt to the carbon source is adjusted in a range of 8 to 20 : 2 to 8 : 1.

【Claim 13】The process of claim 1, wherein the reductive roasting in step b) is performed at a temperature adjusted in a range of 600 to 1,200°C.

【Claim 14】The process of claim 1, wherein a weight ratio of the magnetic portion to the non-magnetic portion separated in step b) is 1 to 4 : 1.

【Claim 15】The process of claim 1, wherein a liquid-to-solid (L/S) ratio during liquid leaching each of the magnetic portion and non-magnetic portion in step c) may be adjusted in the range of 1 to 50.

【Claim 16】The process of claim 1, wherein the magnetic portion in step c) contains at least 20 wt% of iron (on an elemental basis).

【Claim 17】The process of claim 1, wherein the non-magnetic portion in step c) contains at least 4 wt% of titanium (on an elemental basis).

【Claim 18】The process of claim 1, wherein recovering titanium in step c) comprises subjecting the non-magnetic portion to mixed sulfation roasting using sulfuric acid and sulfate.

【Claim 19】The process of claim 18, wherein the sulfate is at least one selected from the group consisting of sodium sulfate (Na_2SO_4), ammonium sulfate ($(NH_4)_2SO_4$), magnesium sulfate ($MgSO_4$), potassium sulfate (K_2SO_4), and barium sulfate ($BaSO_4$).

【Claim 20】The process of claim 18, wherein a weight ratio of sulfuric acid/sulfate during the mixed sulfation roasting is adjusted in a range of 0.5 to 4.

【Claim 21】The process of claim 18, wherein the non-magnetic portion subjected to mixed sulfation roasting is leached with sulfuric acid at a temperature of 70 to 150°C to form a titanium-containing leach solution, in which a concentration of sulfuric acid is up to 10 M and a liquid-to-solid ratio is adjusted in a range of 2 to 100.

【Claim 22】The process of claim 21, wherein a titanium-containing precipitate is formed through titanium hydrolysis carried out by heating the titanium-containing leach solution to a temperature of 70 to 100°C and adjusting pH to less than 3.

【Claim 23】The process of claim 22, wherein the titanium-containing precipitate is converted into titania by calcination in an oxygen-containing atmosphere at a temperature of 200 to 900°C for 0.5 to 10 hours.

【ABSTRACT】

The present disclosure provides a series of processes implemented by combining: a dry process involving reduction roasting and magnetic separation for recovering iron in red mud generated as a by-product in a bauxite processing process; and a wet process for additionally recovering each of aluminum and titanium, respectively, after separation of iron.

Fig. 1

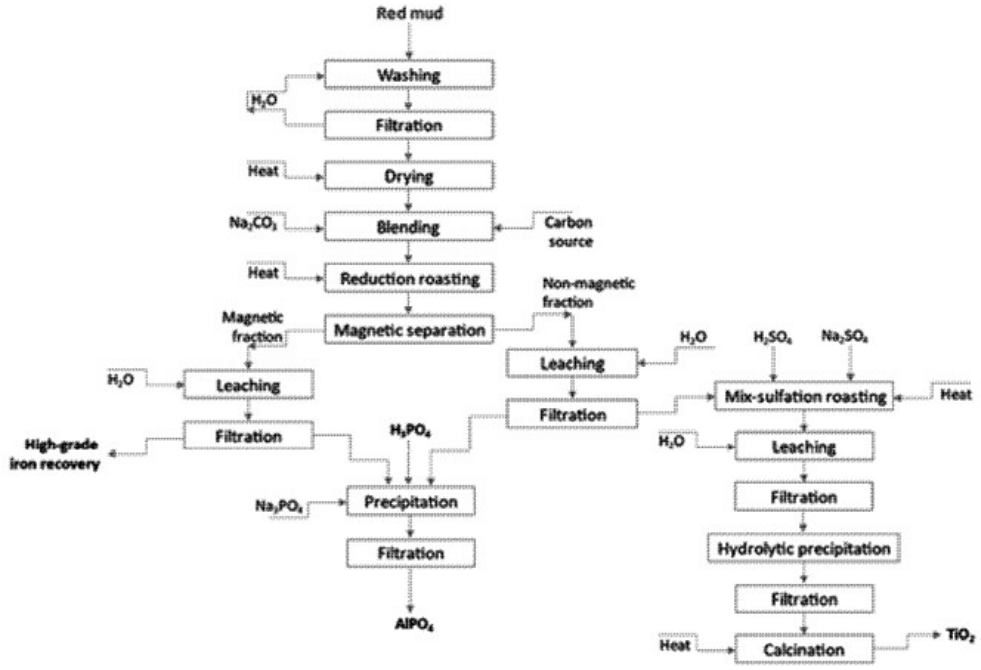


Fig. 1

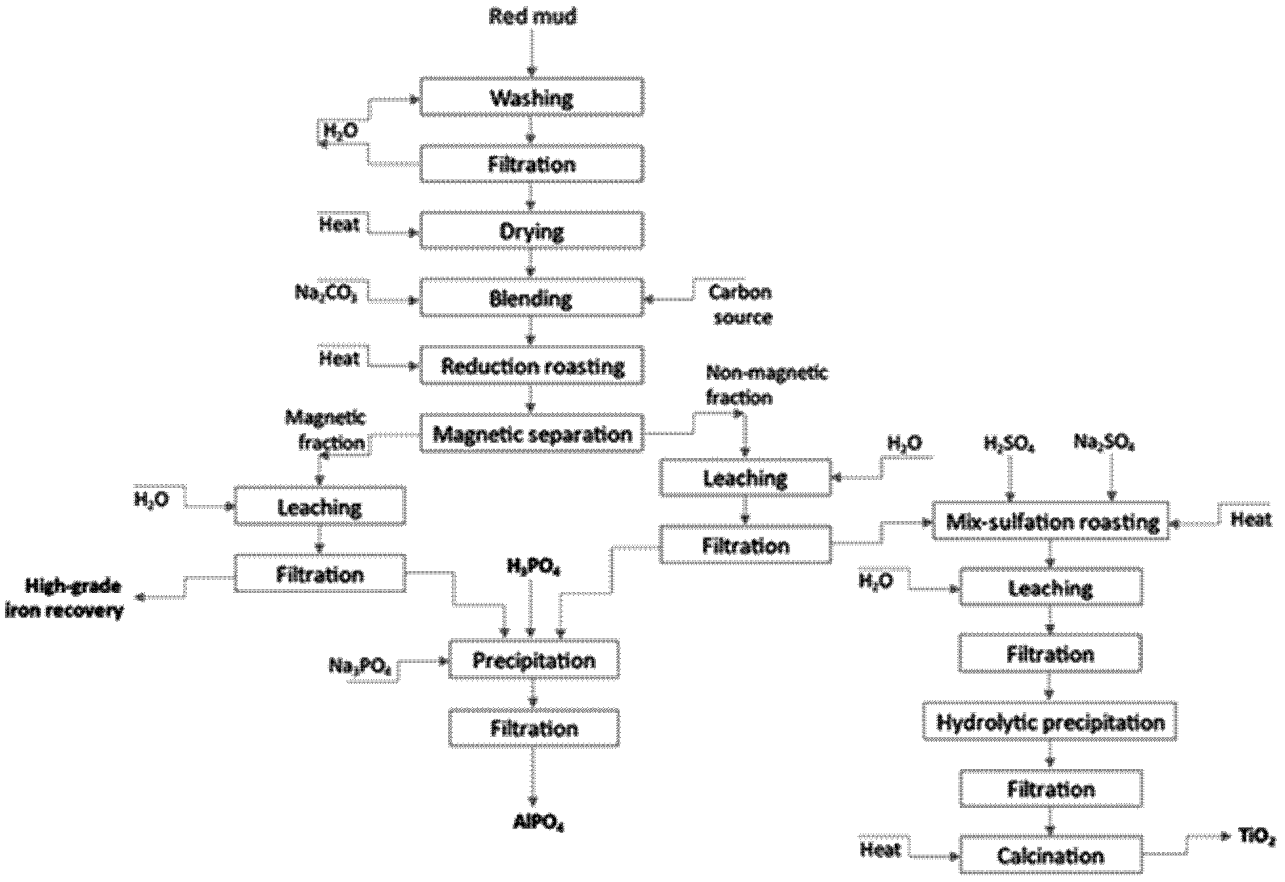

Red mud
Washing
H_2O
Filtration
Heat
Drying
Na_2CO_3
Blending
Carbon source
Heat
Reduction roasting
Non-magnetic fraction
Magnetic fraction
Magnetic separation
H_2O
Leaching
Filtration
High-grade iron recovery
H_3PO_4
Na_3PO_4
Precipitation
Filtration
$AlPO_4$
H_2O
H_2SO_4
Na_2SO_4
Leaching
Filtration
Mix-sulfation roasting
Heat
H_2O
Leaching
Filtration
Hydrolytic precipitation
Filtration
Heat
Calcination
TiO_2

Fig. 2

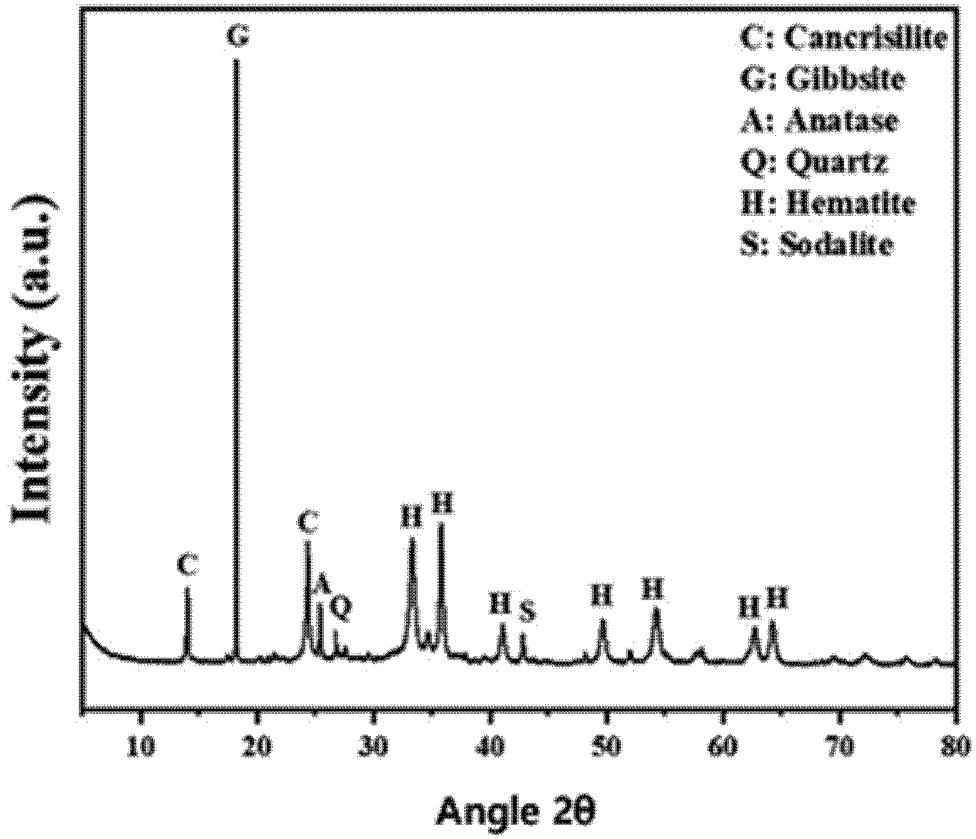

C: Cancrisillite
G: Gibbsite
A: Anatase
Q: Quartz
H: Hematite
S: Sodalite
Intensity (a.u.)
Angle 2θ
10
20
30
40
50
60
70
80

Fig. 3

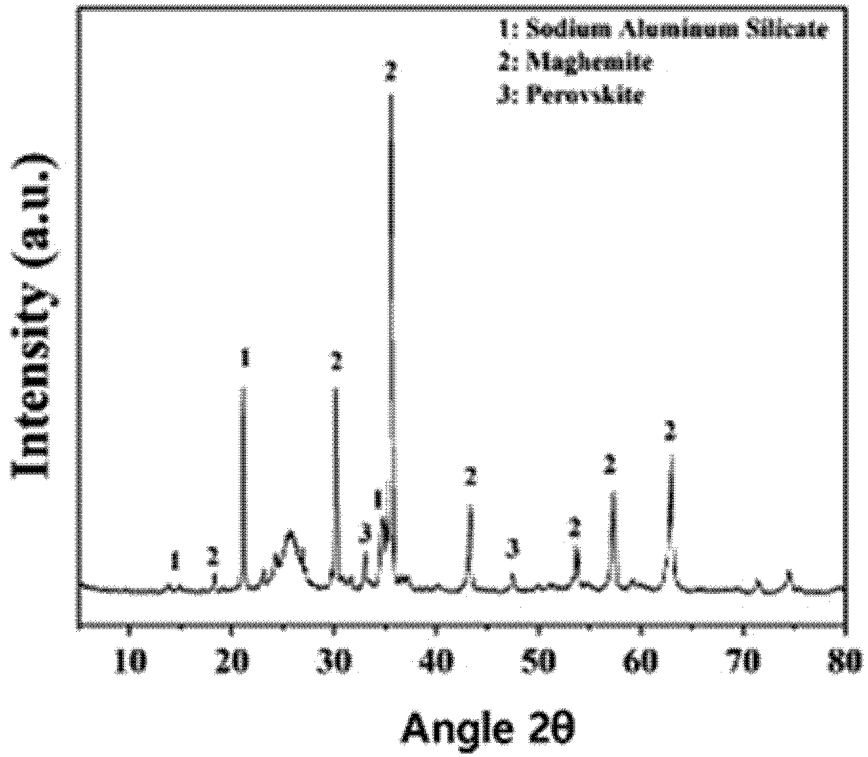
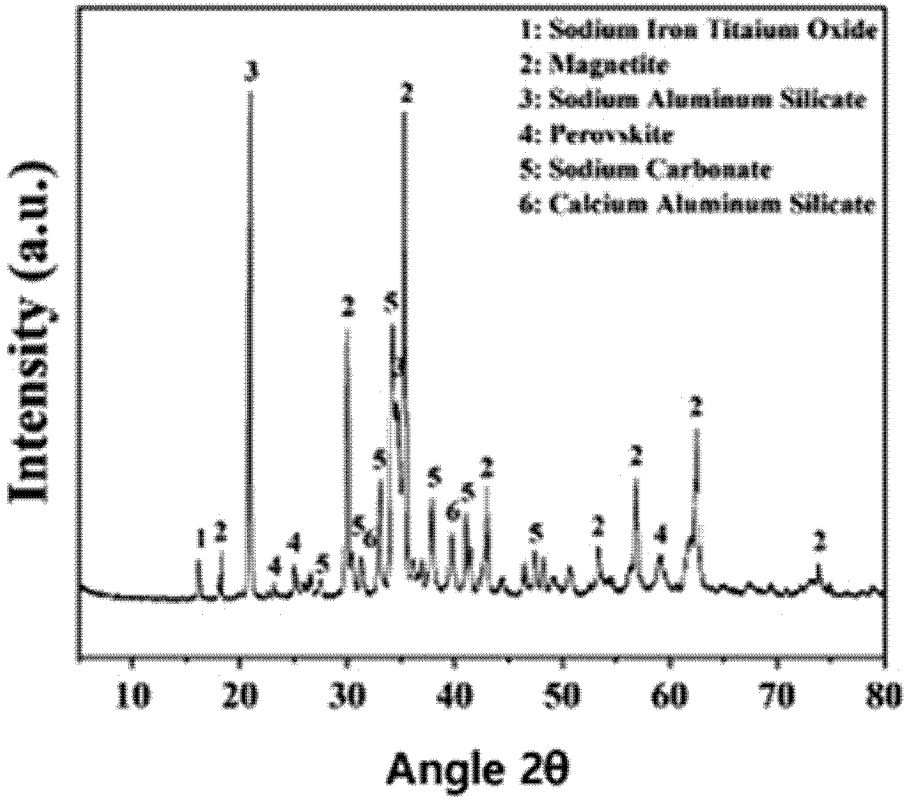

1: Sodium Aluminum Silicate
2: Maghemite
3: Perovskite
Intensity (a.u.)
Angle 2θ
10
20
30
40
50
60
70
80

Fig. 4


1: Sodium Iron Titaium Oxide
2: Magnetite
3: Sodium Aluminum Silicate
4: Perovskite
5: Sodium Carbonate
6: Calcium Aluminum Silicate
Intensity (a.u.)
Angle 2θ
10
20
30
40
50
60
70
80