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Nickel-cobalt precipitation method for nickel laterite ore acid leaching solution after iron and aluminium removal

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Abstract

The present disclosure provides a nickel-cobalt precipitation method for nickel laterite ore acid leaching solution after iron-aluminium removal. The above precipitation method includes: S100, adding a reducing agent to the post iron-aluminium removal solution; S200, continuously feeding the mixed solution into a reactor to perform an alkali conversion precipitation reaction; S300, continuously performing a dense separation treatment on the precipitation slurry; S400, adding a precipitation inducer to a part of an overflow so as to perform the alkali conversion reaction, then mixing and homogenizing the alkali conversion overflow and the first part of an underflow; or, adding the precipitation inducer to the first part of the underflow so as to perform the alkali conversion reaction; S500, continuously feeding the alkali conversion crystal slurry into the reactor; and S600, filtering and washing the second part of the underflow and obtain a mixed hydroxide precipitate product.

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Drawings

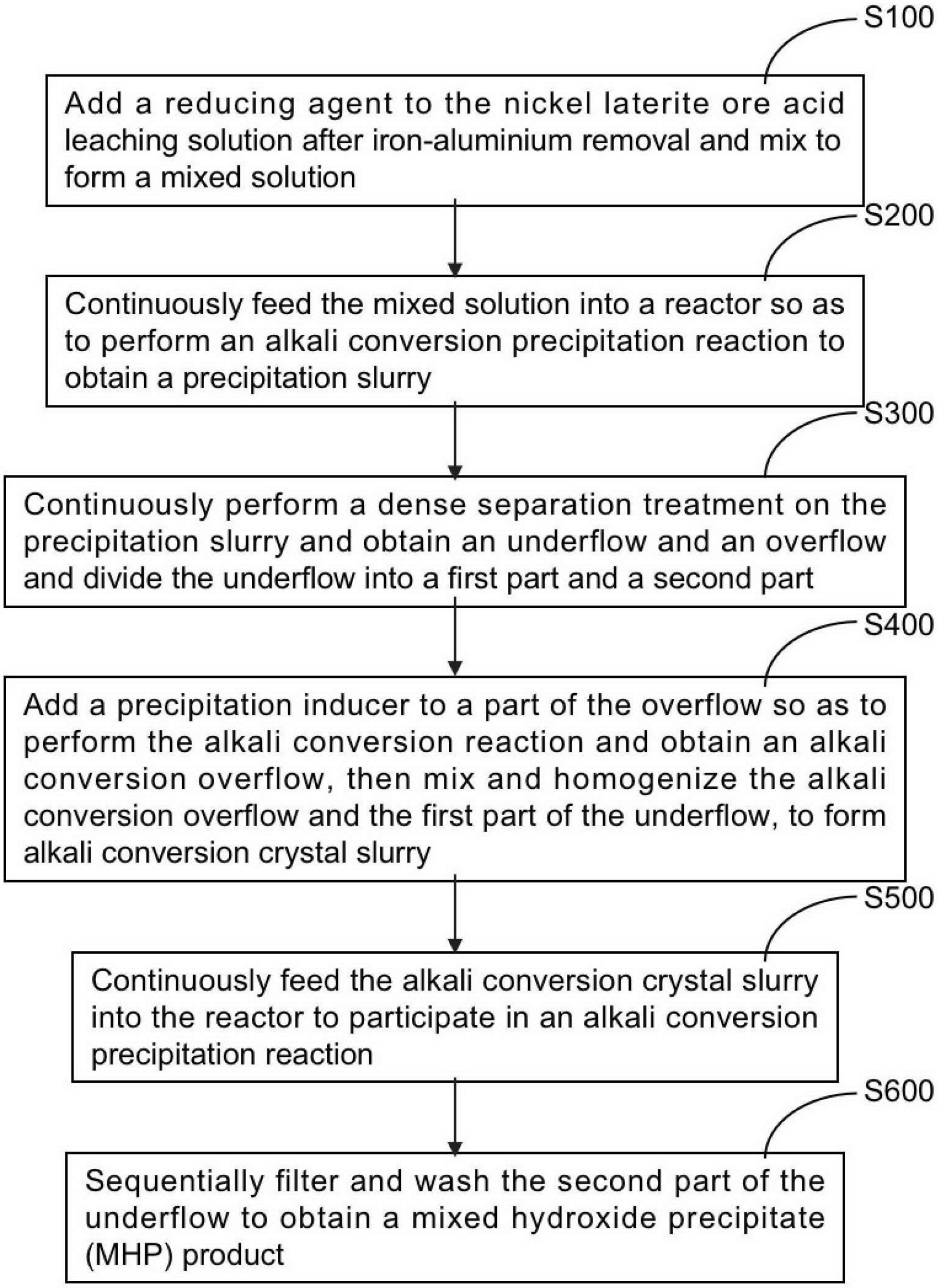


Fig. 1

Nickel-cobalt precipitation method for nickel laterite ore acid leaching solution after iron and aluminium removal

Technical Field

The present disclosure relates to the technical field of nickel laterite ore hydrometallurgy, in particular, to a nickel-cobalt precipitation method for nickel laterite ore acid leaching solution after iron-aluminium removal.

Background

The main processes of nickel laterite ore hydrometallurgy include atmospheric pressure leaching and high pressure leaching, and the commonly used leaching agent is sulfuric acid. Herein the high pressure acid leaching process is used to treat a limonite-type nickel laterite ore, and it has the advantages of short leaching time, low iron leaching rate, high nickel-cobalt leaching rate and recovery rate, low production cost and the like, and becomes a preferred process for most new nickel laterite ore hydrometallurgy projects in recent years. The high pressure leaching process of nickel laterite ore mainly includes working procedures such as high pressure acid leaching, neutralization of leach slurry, countercurrent washing, neutralization and impurity removal, and nickel-cobalt precipitation. The nickel-cobalt precipitation working procedure is aimed at the nickel laterite ore high pressure acid leaching solution after neutralization and impurity removal treatment, it is also known as nickel laterite ore acid leaching solution after iron-aluminium removal.

The nickel-cobalt precipitation working procedure in existing technologies mostly adopts the method of sodium hydroxide precipitation or sulfide precipitation, such as:

A method of iron removal by precipitation and nickel-cobalt enrichment is proposed in patent CN101575676A. Herein a nickel-cobalt enrichment working procedure adopts a vulcanizing agent to perform the sulfide precipitation, and during the nickel-cobalt precipitation enrichment, this patent adopts one or more of the following vulcanizing agents: hydrogen sulfide, hydrogen persulfide, sodium hydrosulfide, sodium sulfide, potassium sulfide, ammonium sulfide, magnesium sulfide and zinc sulfide. The precipitation effect of hydrogen sulfide is the best, but it is highly toxic; the requirements for operation and system are higher and the subsequent treatment for intermediate products of the sulfide precipitation is difficult.

Patent CN102061387A discloses a two-stage nickel precipitation method, in which NaOH solution is added to nickel sulfate solution containing magnesium, adjusting pH to 7.5~8.5. The reaction temperature is 20~80°C; the reaction time is 0.5~3 hours, and the obtained precipitate is separated from the mother solution. The precipitate enters the next working procedure, in which NaOH solution is added to the mother solution, adjusting pH to 9~10. The reaction temperature is 20-80°C; the reaction time is 0.5~3 hours, and the finally obtained precipitate is returned to a stirring and leaching system for circulation after being separated from the mother solution. In this patent, sodium hydroxide or mixed alkali solution is directly used to precipitate nickel and cobalt, and the product is small and difficult to settle, resulting in high water content in the product.

A process of preparing cobalt hydroxide from cobalt sulfate solution is proposed in patent CN101921001A. Firstly magnesium hydroxide slurry is used to perform the first cobalt precipitation on cobalt sulfate solution in a first cobalt precipitation tank, and after the first cobalt precipitation slurry obtained by the previous reaction passes through a first cobalt precipitation buffer tank, 30%~50% of the obtained slurry is returned to the first cobalt precipitation tank, while the remaining slurry is subjected to pressure filtration, and the filter cake obtained is sent to a drying working procedure. The cobalt hydroxide after being dried is the product, and magnesium hydroxide is used to perform second cobalt precipitation on the filtrate. This technology directly uses magnesium hydroxide as precipitating agent, but its alkalinity is limited. Although the generation of local excess alkalinity is avoided, the activity of magnesium hydroxide is reduced after slurrying, resulting in the adverse effect of a low cobalt precipitation rate.

In a word, the process of nickel-cobalt precipitation by hydrogen sulfide method is widely used, and it has the advantages of high precipitation rate, low precipitation pH, and nickel-cobalt can be recovered without purification of the acidic leach solution. However, hydrogen sulfide is highly toxic, so there exist many safety and environmental hazards, and the subsequent treatment process for the intermediate product obtained by precipitation is also extremely cumbersome. The process of nickel-cobalt precipitation by using sodium hydroxide is simple, and the investment in device is low. However, strong alkaline sodium hydroxide is directly used as the precipitant, which may cause the mixed hydroxide precipitate (MHP) product small in particle diameter, and poor in sedimentation ability, resulting in a tough liquid-solid separation, a high water content in the filter cake, and further difficult subsequent transportation and treatment. In addition, directly using magnesium hydroxide as the precipitant, may easily lead to a low precipitation rate due to its limited alkalinity and lower activity.

In view of this, the present disclosure is proposed.

Summary

The main purpose of the present disclosure is to provide a nickel-cobalt precipitation method for nickel laterite ore acid leaching solution after iron-aluminium removal, as to solve problems in existing technologies that while nickel and cobalt are precipitated, the environmental protection is poor; the nickel-cobalt hydroxide is smaller in particle diameter; the filter cake is high in water content, or the precipitation rate is low.

In order to achieve the above purpose, according to one aspect of the present disclosure, a nickel-cobalt precipitation method for nickel laterite ore acid leaching solution after iron-aluminium removal is provided. The positive ions in the post iron-aluminium removal solution contain nickel ions, cobalt ions, magnesium ions and manganese ions. The working processes include: S100, adding a reducing agent to the post iron-aluminium removal solution and mixing, to form a mixed solution; S200, continuously feeding the mixed solution into a reactor so as to perform an alkali conversion precipitation reaction and obtain a precipitation slurry; S300, continuously performing a dense separation treatment on the precipitation slurry and obtain an underflow and an overflow, and dividing the underflow into a first part and a second part; S400, adding a precipitation inducer to a part of the overflow so as to perform the alkali conversion reaction and obtain an alkali conversion overflow, then mixing and homogenizing the alkali conversion overflow and the first part of the underflow, to form alkali conversion crystal slurry; or, adding the precipitation inducer to the first part of the underflow so as to perform the alkali conversion reaction and form the alkali conversion crystal slurry. Herein the precipitation inducer is a mixed solution of water and one or more of the following substances: sodium hydroxide, calcium hydroxide, calcium oxide, potassium hydroxide, magnesium oxide and magnesium hydroxide; S500, continuously feeding the alkali conversion crystal slurry into the reactor to participate in an alkali conversion precipitation reaction; and S600, sequentially filtering and washing the second part of the underflow and obtain a mixed hydroxide precipitate (MHP) product.

Further, the reducing agent is selected from one or more of the following substances: sodium pyrosulfite, sodium sulfite, hydrogen peroxide, sulfur dioxide, and sodium bisulfite; preferably the reducing agent is sodium pyrosulfite.

Further, the temperature of the reducing agent added to the post iron-aluminium removal solution is 50°C ~70°C, and the mixing time is 0.5~3.0 h; preferably the addition amount of reducing agent is 0.05~10 kg/m^3 relative to the post iron-aluminium removal solution, more preferably 0.1~2 kg/m^3.

Further, the total mass concentration of the precipitation inducer is 1~45%, preferably 18~25%; preferably, the precipitation inducer is a mixed solution of water and at least two of the following substances: sodium hydroxide, calcium hydroxide, calcium oxide, potassium hydroxide, magnesium oxide, and magnesium hydroxide; more preferably, at least one solute in the precipitation inducer is sodium hydroxide or potassium hydroxide, and at least one solute is calcium hydroxide, calcium oxide, magnesium oxide, or magnesium hydroxide; and further preferably, the solute of the precipitation inducer is sodium hydroxide, potassium hydroxide and magnesium oxide, with a weight ratio of (3~4):(3~4):(2~4).

Further, based on the theoretical mole number of hydroxyls required for nickel-cobalt precipitation in the mixed solution, the mole number of hydroxyls in the precipitation inducer is 0.7~1.2 times the theoretical mole number, preferably 0.8~0.9 times.

Further, the temperature of the alkali conversion precipitation reaction is 50~70°C, and the time is 0.5~5 h.

Further, the weight ratio of the first part of the underflow to the second part of the underflow is 1:(1~15), preferably 1:(8~12).

Further, in step S400, the precipitation inducer is added to a part of the overflow which accounts for 0.5~10% of the total weight of the overflow.

Further, in step S400, the temperature of the alkali conversion reaction is 60~70°C, and the time is 1~5 min.

Further, positive ion components in the nickel laterite ore acid leaching solution after iron-aluminium removal are as follows: 2.5~5 g/L of nickel ions, 0.2~0.5 g/L of cobalt ions, 3~15 g/L of magnesium ions, and 1.5~5 g/L of divalent manganese ions.

The present disclosure provides a nickel-cobalt precipitation method for nickel laterite ore acid leaching solution after iron-aluminium removal, which effectively solves the problems of poor environmental protection, small nickel-cobalt hydroxide particle diameter, difficult nickel-cobalt hydroxide settlement, a high water content in filter cake, or poor precipitation rate in existing technologies. Through the aforementioned nickel-cobalt precipitation method, the additives used are more environment-friendly, the precipitation rate is high, the morphology of the nickel-cobalt hydroxide is better, the particle diameter is larger, and the sedimentation ability is good, which is beneficial to the subsequent liquid-solid separation working procedure, resulting in reduced water content in MHP product.

Brief Description of the Drawings

Drawings constituting a part of the present application are used to provide a further understanding of the present disclosure, and the exemplary embodiments of the present disclosure and descriptions thereof are used to explain the present disclosure, and do not constitute an improper limitation to the present disclosure. In the drawings:

Fig. 1 shows a flow chart of a nickel-cobalt precipitation method for nickel laterite ore acid leaching solution after iron-aluminium removal according to an embodiment of the present disclosure.

Fig. 2 shows a flow chart of a nickel-cobalt precipitation method for nickel laterite ore acid leaching solution after iron-aluminium removal according to another embodiment of the present disclosure.

Detailed Description of the Embodiments

It should be noted that embodiments in the present application and features of the embodiments may be combined in the case without conflict. The present disclosure is described in detail below with reference to the drawings and in combination with the embodiments.

As described in the background section, in existing technologies, while nickel and cobalt are precipitated from the nickel laterite ore acid leaching solution after iron-aluminium removal, the environmental protection is poor, the nickel-cobalt hydroxide is small in particle diameter, the filter cake is high in water content, or the precipitation rate is low.

In order to solve the above problems, the present disclosure provides a nickel-cobalt precipitation method for nickel laterite ore acid leaching solution after iron-aluminium removal. The positive ions in the post iron-aluminium removal solution contain nickel ions, cobalt ions, magnesium ions and manganese ions. Herein, as shown in Fig. 1 and Fig. 2, the working processes include: S100, adding a reducing agent to the post iron-aluminium removal solution and mixing, to form mixed solution; S200, continuously feeding the mixed solution into a reactor so as to perform an alkali conversion precipitation reaction, to obtain a precipitation slurry; S300, continuously performing a dense separation treatment on the precipitation slurry, to obtain an underflow and an overflow and dividing the underflow into a first part and a second part; S400, as shown in Fig. 1, adding a precipitation inducer to a part of the overflow so as to perform the alkali conversion reaction to obtain an alkali conversion overflow, then mixing and homogenizing the alkali conversion overflow and the first part of the underflow to form the alkali conversion crystal slurry; or, as shown in Fig. 2, adding the precipitation inducer to the first part of the underflow so as to perform the alkali conversion reaction and form the alkali conversion crystal slurry. Herein, the precipitation inducer is a mixed solution of water and one or more of the following substances: sodium hydroxide, calcium hydroxide, calcium oxide, potassium hydroxide, magnesium oxide and magnesium hydroxide; S500, continuously feeding the alkali conversion crystal slurry into the reactor to participate in the alkali conversion precipitation reaction; and S600, sequentially filtering and washing the second part of the underflow and obtain the MHP product.

In the above method provided by the present disclosure, before the nickel-cobalt precipitation is performed, the reducing agent is added to it in advance, so that the post iron-aluminium removal solution may be kept in a reducing environment, preventing divalent manganese ions from being subsequently oxidized to a high valence and further precipitated due to activity loss. On the one hand, divalent manganese ions may be effectively converted into manganese hydroxide to participate in the subsequent alkali conversion precipitation reaction; on the other hand, the manganese content in the final MHP product may also be effectively reduced (high-valent manganese ions may be converted into precipitate and enter MHP, and the divalent manganese ions may enter alkali conversion crystal slurry by the subsequent alkali conversion reaction and further participate in the alkali conversion precipitation reaction).

After the reducing agent is added, the mixed solution is continuously fed into the reactor to perform the alkali conversion precipitation reaction. In this process, the present disclosure does not use sulfide or strong alkali sodium hydroxide as in traditional precipitation reactions, nor directly uses the magnesium hydroxide with lower activity. The precipitating agent used in the present disclosure is the alkali conversion crystal slurry formed after the underflow or overflow is subjected to the alkali conversion reaction with the addition of a precipitation inducer. As mentioned above, the slurry is obtained after the underflow or overflow produced by continuous precipitation is subjected to the alkali conversion reaction with the addition of a precipitation reducer. Specifically, the precipitation inducer is added to a part of the overflow obtained in the previous working procedures so as to perform the alkali conversion reaction to obtain the alkali conversion overflow which is later mixed and homogenized with the first part of the underflow to form the alkali conversion crystal slurry; or, the precipitation inducer is added to the first part of the underflow obtained in the previous working procedure so as to perform the alkali conversion reaction and form the alkali conversion crystal slurry.

As mentioned above, in addition to the nickel ions and cobalt ions to be precipitated, the mixed solution of the post iron-aluminium removal solution and the reducing agent also contains a large number of magnesium ions and divalent manganese ions. Correspondingly, the underflow and the overflow generated in the previous working procedure also contain a large number of magnesium ions and divalent manganese ions. Adding the aqueous solution of one or more of the following substances: sodium hydroxide, calcium hydroxide, calcium oxide, potassium hydroxide, magnesium oxide and magnesium hydroxide to a part of the overflow or the first part of the underflow as the precipitation inducer may convert these magnesium ions and divalent manganese ions into magnesium hydroxide and manganese hydroxide. Meanwhile, since the present disclosure uses the precipitation inducer to prepare the alkali conversion crystal slurry at the same time as carrying out the processes of continuous alkali conversion precipitation and continuous dense separation, the alkali conversion crystal slurry with high activity may be returned to the previous working procedure in time and participate in the alkali conversion precipitation reaction, and it is also guaranteed that the alkali conversion crystal slurry has a high precipitation activity. In addition, the alkali conversion crystal slurry also contains a part of the nickel-cobalt hydroxide (from the first part of the underflow) generated in the previous working procedure, it may also play the role of the seed crystal template in the process of alkali conversion precipitation reaction, promoting the precipitation and crystal growth of nickel-cobalt hydroxide.

The reasons for the above aspects make the present disclosure effectively solve the problems of poor environmental protection, small nickel-cobalt hydroxide particle diameter, difficult nickel-cobalt hydroxide settlement, high water content in filter cake, or poor precipitation rate in existing technologies. Through the aforementioned nickel-cobalt precipitation method, the additives used are more environment-friendly, the precipitation rate is high, the morphology of the nickel-cobalt hydroxide is better, the particle diameter is larger, and the sedimentation ability is good, which is beneficial to the subsequent liquid-solid separation working procedure, resulting in reduced water content in MHP product.

In a preferred embodiment, in order to maintain a better precipitation activity of the alkali conversion crystal slurry and prevent it from ageing after a long period of storage, the alkali conversion crystal slurry is returned to the previous step S200 to participate in the alkali conversion precipitation reaction within 5 minutes, more preferably within 3 minutes.

In order to better maintain the reducing environment of the solution system, in a preferred embodiment, the above reducing agent is selected from one or more of the following substances: sodium pyrosulfite, sodium sulfite, hydrogen peroxide, sulfur dioxide, and sodium hydrogen sulfite; preferably the reducing agent is sodium pyrosulfite. By using the above reducing agent, the purpose of preventing the oxidation of divalent manganese ions may be achieved, and the impact on the nickel-cobalt precipitation reaction may be avoided. More preferably, the temperature of the reducing agent added to the post iron-aluminium removal solution is 50°C ~70°C, and the mixing time is 0.5~3.0 h; and preferably the addition amount of reducing agent is 0.05~10 kg/m^3 relative to the post iron-aluminium removal solution, more preferably 0.1~2 kg/m^3. Controlling the mixing process conditions and the addition amount of the reducing agent within the ranges mentioned above may fully exert those effects. Correspondingly, the alkali conversion crystal slurry may maintain a more suitable activity, and the manganese content in the MHP product is lower. In actual operation, the mixing process is preferably performed under stirring.

The above precipitation inducer is added in the form of an aqueous solution or slurry, in order to make the alkali conversion reaction more stable and the activity of the formed alkali conversion crystal slurry to be higher, in a preferred embodiment, the total mass concentration of precipitation inducer is 1~45%, preferably 18~25%. Controlling the concentration within this range, on the one hand, can make the alkali conversion reaction more stable; on the other hand, can make the concentration of the alkali conversion crystal slurry more suitable, so that the alkali conversion precipitation reaction is more efficient and stable.

Preferably, the precipitation inducer is a mixed solution of water and at least two of the following substances: sodium hydroxide, calcium hydroxide, calcium oxide, potassium hydroxide, magnesium oxide and magnesium hydroxide. More preferably, at least one solute in the precipitation inducer is sodium hydroxide or potassium hydroxide, and at least one solute is calcium hydroxide, calcium oxide, magnesium oxide or magnesium hydroxide. Using these precipitation inducers can make the precipitation activity of the alkali conversion crystal slurry more suitable, it may not only make nickel and cobalt be precipitated more stably, but also help nickel-cobalt hydroxide to be precipitated with a better particle morphology, and an easier liquid-solid separation is easier and make the final product have lower water content and lower manganese content. Further preferably, the solute of the precipitation inducer is sodium hydroxide, potassium hydroxide and magnesium oxide, and the weight ratio between these three is (3~4):(3~4):(2~4). Compared with other precipitation inducers such as calcium oxide, calcium hydroxide or sodium hydroxide, using the compound-type precipitation inducer with the above weight ratio, can make the effect of the formed alkali conversion crystal slurry better; and the compound-type seed crystal template has better promoting effects on improving the morphology, precipitation rate, particle size and the like of the nickel-cobalt hydroxide in the alkali conversion precipitation process.

In order to make nickel ions and cobalt ions in the mixed solution to be more fully precipitated, in a preferred embodiment, based on the theoretical mole number of hydroxyls required for the nickel-cobalt precipitation in the mixed solution, the mole number of hydroxyls in the precipitation inducer is 0.7~1.2 times the theoretical mole number. In actual operation, in order to further reduce the impurity content of the MHP product, such as magnesium and manganese; preferably the mole number of hydroxyls in the precipitation inducer is 0.8~0.9 times the theoretical mole number. In this way, a part of the direct yield of the nickel-cobalt hydroxide may be lost, but the product is purer.

In order to make magnesium ions and divalent manganese ions in part of the overflow or the first part of the underflow be more fully converted into magnesium hydroxide and manganese hydroxide, in a preferred embodiment, the temperature of the alkali conversion precipitation reaction is 50~70°C, and the time is 0.5~5 h. Under these conditions, the finally formed alkali conversion crystal slurry has a better nickel-cobalt precipitation activity and has a better promoting effect on the nickel-cobalt precipitation process in the mixed solution.

The active components in alkali conversion crystal slurry mainly are nickel-cobalt hydroxide, magnesium hydroxide and manganese hydroxide. In order to make the alkali conversion crystal slurry have a higher precipitation activity, in a preferred embodiment, the weight ratio of the first part of the underflow to the second part of the underflow is 1: (1~15), preferably 1:(8~12). More preferably, in step S400, the precipitation inducer is added to a part of the overflow which accounts for 0.5~10% of the total weight of the overflow. Further preferably, in step S400, the temperature of the alkali conversion reaction is 60~70°C; the time is 1~5 min.

In the actual operation, the second part of the underflow may be directly filtered and washed to obtain MHP product with a low manganese content. While a part of the overflow participates in the alkali conversion reaction, after it is subjected to the alkali conversion reaction, it is mixed and homogenized with the first part of the underflow to form the alkali conversion crystal slurry with a better precipitation activity, and the remaining part of the overflow may directly perform second-stage nickel-cobalt precipitation. While the overflow does not participate in alkali conversion reaction, it may be directly drawn out to later perform the second-stage nickel-cobalt precipitation (returned to the previous working procedure such as nickel laterite ore high pressure acid leaching working procedure).

The aforementioned precipitation method provided by the present disclosure may be performed continuously. After the fresh post iron-aluminium removal solution continuously enters the continuous reduction reaction, it continuously performs the alkali conversion precipitation reaction with the alkali conversion crystal slurry returned from the subsequent working procedure. The obtained precipitation slurry continuously enters a liquid-solid separation stage to form the underflow and the overflow. A part of the underflow and a part of the overflow (optionally) continue to participate in the alkali conversion reaction and continue to form the alkali conversion crystal slurry which is later returned to the previous working procedure to participate in the alkali conversion precipitation reaction. In the early operation stage of this method, the mixed solution may firstly perform the sodium hydroxide precipitation, and after the initial underflow and overflow are formed, prepare the alkali conversion crystal slurry through the preparation process described above as the precipitant after a stable operation is achieved.

The above precipitation method of the present disclosure is aimed at the nickel-cobalt precipitation of the nickel laterite ore acid leaching solution after iron-aluminium removal, especially suitable for the solution in which the positive components are as follows: 2.5~5 g/L of Ni^{2+}, 0.2~0.5 g/L of Co^{2+}, 3~15 g/L of Mg^{2+}, and 1.5~5 g/L of Mn^{2+}.

The present application is further described in detail below with reference to specific embodiments, and these embodiments should not be construed as limiting the scope of protection claimed by the present application.

The object to be treated in the following embodiments is the nickel laterite ore acid leaching solution after iron-aluminium removal (aqueous solution). Its negative ions are sulfate ions, and the positive ion components are as follows:

Types of positive ions	Ni^{2+}	Co^{2+}	Mg^{2+}	Mn^{2+}
Concentration	3~5 g/L	0.2~0.5 g/L	5~8 g/L	2~4 g/L

Embodiment 1

0.1 kg/m^3 of sodium pyrosulfite is added to the above post iron-aluminium removal nickel laterite ore acid leaching solution, stirred and mixed at 70°C for 3.0 h, to form a mixed solution.

The mixed solution and alkali conversion crystal slurry prepared by the back-end working procedure are continuously convectively added into a reactor to perform an alkali conversion precipitation reaction, the reaction time is 0.5 h, the reaction temperature is 60°C, and a precipitation slurry is obtained.

The precipitation slurry is continuously sent to a thickener for dense separation, to obtain an underflow and an overflow; a precipitation inducer with a total mass concentration of 20% is prepared, and it is a slurry formed by dissolving sodium hydroxide, potassium hydroxide and magnesium oxide (4:4:2) in water; 5% of the overflow is continuously fed into an alkali conversion reactor, the precipitation inducer is added to it for an alkali conversion reaction, the theoretical mole number of hydroxyls required for nickel-cobalt precipitation in the mixed solution is used as a benchmark, and the mole number of the hydroxyls in the precipitation inducer is 0.9 times the theoretical mole number. The temperature of the above alkali conversion reaction is 70°C, and the reaction time is 1 min. The formed alkali conversion overflow is mixed and homogenized with a part of the underflow (the weight ratio to the remaining underflow is 1:10), to form an alkali conversion crystal slurry. The alkali conversion crystal slurry is continuously returned to the alkali conversion precipitation step within 3 min, to perform the nickel-cobalt alkali conversion precipitation reaction.

The remaining underflow is filtered and washed successively, to obtain an MHP product.

After being tested, the nickel-cobalt precipitation rate is 85%, the particle diameter D50 of MHP is 45~55 μm, the water content is 55%, and the product contains 39% of nickel and 4% of manganese.

Embodiment 2

0.1 kg/m^3 of sodium pyrosulfite is added to the above post iron-aluminium removal nickel laterite ore acid leaching solution, stirred and mixed at 70°C for 3.0 h, to form a mixed solution.

The mixed solution and alkali conversion crystal slurry prepared by the back-end working procedure are continuously convectively added into a reactor to perform an alkali conversion precipitation reaction, the reaction time is 0.5 h, the reaction temperature is 60°C, and a precipitation slurry is obtained.

The precipitation slurry is continuously sent to a thickener for dense separation, to obtain an underflow and an overflow; a precipitation inducer with a total mass concentration of 20% is prepared, and it is a slurry formed by dissolving sodium hydroxide, potassium hydroxide and magnesium oxide (4:4:2) in water; a part of the underflow (the weight ratio to the remaining underflow is 1:10) is continuously fed into an alkali conversion reactor, the precipitation inducer is added to it for an alkali conversion reaction, to obtain an alkali conversion crystal slurry. Herein, the theoretical mole number of hydroxyls required for nickel-cobalt precipitation in the mixed solution is used as a benchmark, and the mole number of the hydroxyls in the precipitation inducer is 0.9 times the theoretical mole number. The temperature of the above alkali conversion reaction is 70°C, and the reaction time is 1 min. The alkali conversion crystal slurry is continuously returned to the alkali conversion precipitation step within 3 min, to perform the nickel-cobalt alkali conversion precipitation reaction. The remaining underflow is filtered and washed successively, to obtain an MHP product.

After being tested, the nickel-cobalt precipitation rate is 85%, the particle diameter D50 of MHP is 40~55 μm, the water content is 55%, and the product contains 39% of nickel and 4% of manganese.

Embodiment 3

The differences between this embodiment and Embodiment 1 are as follows: the weight ratio of sodium hydroxide, potassium hydroxide and magnesium oxide is 3:3:4, and the total mass concentration of the precipitation inducer is 25%.

After being tested, the nickel-cobalt precipitation rate is 86%, the particle diameter D50 of MHP is 55~60 μm, the water content is 54%, and the product contains 41% of nickel and 3.5% of manganese.

Embodiment 4

The differences between this embodiment and Embodiment 1 are as follows: the weight ratio of sodium hydroxide, potassium hydroxide and magnesium oxide is 3.5:3.5:3, and the total mass concentration of the precipitation inducer is 18%.

After being tested, the nickel-cobalt precipitation rate is 84%, the particle diameter D50 of MHP is 40~45 μm, the water content is 53%, and the product contains 40% of nickel and 3.2% of manganese.

Embodiment 5

The differences between this embodiment and Embodiment 1 are as follows: the solute of the precipitation inducer is sodium hydroxide, and the total mass concentration of the precipitation inducer is 15%.

After being tested, the nickel-cobalt precipitation rate is 82%, the particle diameter D50 of MHP is 20~30 μm, the water content is 60%, and the product contains 38% of nickel and 4% of manganese.

Embodiment 6

The differences between this embodiment and Embodiment 1 are as follows: the precipitation inducer is calcium hydroxide solution, and the mass concentration is 20%.

After being tested, the nickel-cobalt precipitation rate is 84%, the particle diameter D50 of MHP is 25~35 μm, the water content is 57%, and the product contains 38% of nickel and 4% of manganese.

Embodiment 7

The difference between this embodiment and Embodiment 1 is only that the reducing agent is sodium bisulfite.

After being tested, the nickel-cobalt precipitation rate is 85%, the particle diameter D50 of MHP is 55~65 μm, the water content is 56%, and the product contains 39% of nickel and 4.2% of manganese.

Embodiment 8

The differences between this embodiment and Embodiment 1 are as follows: the weight ratio between the underflow added to the alkali conversion crystal slurry and the remaining underflow is 1:8; the overflow added to the alkali conversion crystal slurry is 10% of the total weight of the overflow.

After being tested, the nickel-cobalt precipitation rate is 86%, the particle diameter D50 of MHP is 50~55 μm, the water content is 54%, and the product contains 38% of nickel and 3.8% of manganese.

Embodiment 9

The differences between this embodiment and Embodiment 1 are as follows: the weight ratio between the underflow added to the alkali conversion crystal slurry and the remaining underflow is 1:12; the overflow added to the alkali conversion crystal slurry is 0.5% of the total weight of the overflow.

After being tested, the nickel-cobalt precipitation rate is 83%, the particle diameter D50 of MHP is 45~50 μm, the water content is 56%, and the product contains 40% of nickel and 3.9% of manganese.

Embodiment 10

The differences between this embodiment and Embodiment 1 are as follows: the temperature of adding the reducing agent is 50°C, and the mixing time is 0.5 h; the temperature of the alkali conversion reaction is 60°C, and the time is 5 min; the temperature of the alkali conversion precipitation reaction is 50°C, and the time is 5 h.

After being tested, the nickel-cobalt precipitation rate is 86%, the particle diameter D50 of MHP is 45~55 μm, the water content is 55%, and the product contains 41% of nickel and 3.8% of manganese.

Embodiment 11

The difference between this embodiment and Embodiment 1 is that the theoretical mole number of hydroxyls required for nickel-cobalt precipitation in the mixed solution is used as a benchmark, and the mole number of the hydroxyls in the precipitation inducer is 0.8 times the theoretical mole number.

After being tested, the nickel-cobalt precipitation rate is 83%, the particle diameter D50 of MHP is 50~60 μm, the water content is 56%, and the product contains 42% of nickel and 3.5% of manganese.

Embodiment 12

The difference between this embodiment and Embodiment 1 is that the theoretical mole number of hydroxyls required for nickel-cobalt precipitation in the mixed solution is used as a benchmark, and the mole number of the hydroxyls in the precipitation inducer is 0.8 times the theoretical mole number.

After being tested, the nickel-cobalt precipitation rate is 87%, the particle diameter D50 of MHP is 55~60 μm, the water content is 54%, and the product contains 38% of nickel and 4.2% of manganese.

Embodiment 13

The difference between this embodiment and Embodiment 1 is that the alkali conversion crystal slurry is continuously returned to the alkali conversion precipitation step within 5 min, to perform the nickel-cobalt alkali conversion precipitation reaction.

After being tested, the nickel-cobalt precipitation rate is 81%, the particle diameter D50 of MHP is 40~45 μm, the water content is 58%, and the product contains 39% of nickel and 4% of manganese.

Contrast example 1

0.1 kg/m^3 of sodium pyrosulfite is added to the above post iron-aluminium removal nickel laterite ore acid leaching solution, stirred and mixed at 70°C for 3.0 h, to form a mixed solution.

The mixed solution directly performs a precipitation reaction with a pre-prepared 20% sodium hydroxide aqueous solution, the reaction time is 0.5 h, and the reaction temperature is 60°C, to obtain a precipitation slurry.

The precipitation slurry is continuously sent to a thickener for dense separation, to obtain an underflow and an overflow; and the underflow is filtered and washed successively, to obtain an MHP product.

After being tested, the nickel-cobalt precipitation rate is 78%, the particle diameter D50 of MHP is 10~20 μm, the water content is 70%, and the product contains 39% of nickel and 5.8% of manganese.

Contrast example 2

0.1 kg/m^3 of sodium pyrosulfite is added to the above post iron-aluminium removal nickel laterite ore acid leaching solution, stirred and mixed at 70°C for 3.0 h, to form a mixed solution.

The mixed solution directly performs a precipitation reaction with a pre-prepared 20% magnesium hydroxide aqueous solution, the reaction time is 0.5 h, and the reaction temperature is 60°C, to obtain a precipitation slurry.

The precipitation slurry is continuously sent to a thickener for dense separation, to obtain an underflow and an overflow; and the underflow is filtered and washed successively, to obtain an MHP product.

After being tested, the nickel-cobalt precipitation rate is 76%, the particle diameter D50 of MHP is 15~25 μm, the water content is 65%, and the product contains 37% of nickel and 4.7% of manganese.

Contrast example 3

A difference from Embodiment 1 is that the step of adding a reducing agent is eliminated.

After being tested, the nickel-cobalt precipitation rate is 80%, the particle diameter D50 of MHP is 30~40 μm, the water content is 60%, and the product contains 35% of nickel and 6.1% of manganese.

The above are only preferred embodiments of the present disclosure and are not intended to limit the present disclosure. For those skilled in the art, the present disclosure may have various modifications and changes. Any modifications, equivalent replacements, improvements and the like made within the spirit and principle of the present disclosure shall be included within the scope of protection of the present disclosure.

Claims

1. A nickel-cobalt precipitation method for nickel laterite ore acid leaching solution after iron-aluminium removal, in which the positive ions comprise nickel ions, cobalt ions, magnesium ions and manganese ions, wherein the precipitation method comprises:

   S100, adding a reducing agent to the nickel laterite ore acid leaching solution after iron-aluminium removal and mixing, to form a mixed solution;

   S200, continuously feeding the mixed solution into a reactor so as to perform an alkali conversion precipitation reaction, to obtain a precipitation slurry;

   S300, continuously performing a dense separation treatment on the precipitation slurry, to obtain an underflow and an overflow; and dividing the underflow into a first part and a second part;

   S400, adding a precipitation inducer to a part of the overflow so as to perform the alkali conversion reaction to obtain an alkali conversion overflow, then mixing and homogenizing the alkali conversion overflow and the first part of the underflow, to form alkali conversion crystal slurry; or, adding the precipitation inducer to the first part of the underflow so as to perform the alkali conversion reaction and form the alkali conversion crystal slurry, wherein the precipitation inducer is a mixed solution of water and one or more of the following substances: sodium hydroxide, calcium hydroxide, calcium oxide, potassium hydroxide, magnesium oxide and magnesium hydroxide;

   S500, continuously feeding the alkali conversion crystal slurry into the reactor to participate in an alkali conversion precipitation reaction; and

   S600, sequentially filtering and washing the second part of the underflow, to obtain a mixed hydroxide precipitate product.

2. The nickel-cobalt precipitation method for the nickel laterite ore acid leaching solution after iron-aluminium removal according to claim 1, wherein the reducing agent is selected from one or more of the following substances: sodium pyrosulfite, sodium sulfite, hydrogen peroxide, sulfur dioxide, and sodium bisulfite.

3. The nickel-cobalt precipitation method for the nickel laterite ore acid leaching solution after iron-aluminium removal according to claim 1, wherein the temperature of the reducing agent added to the nickel laterite ore acid leaching solution after iron-aluminium removal is 50°C ~70°C, and the mixing time is 0.5~3.0 h.

4. The nickel-cobalt precipitation method for the nickel laterite ore acid leaching solution after iron-aluminium removal according to claim 3, wherein the additional amount of the reducing agent is 0.05~10 kg/m^3 relative to the nickel laterite ore acid leaching solution after iron-aluminium removal.

5. The nickel-cobalt precipitation method for the nickel laterite ore acid leaching solution after iron-aluminium removal according to any one of claims 1 to 4, wherein the total mass concentration of the precipitation inducer is 1~45%.

6. The nickel-cobalt precipitation method for the nickel laterite ore acid leaching solution after iron-aluminium removal according to claim 5, wherein the precipitation inducer is a mixed solution of water and at least two of the follows: sodium hydroxide, calcium hydroxide, calcium oxide, potassium hydroxide, magnesium oxide, and magnesium hydroxide.

7. The nickel-cobalt precipitation method for the nickel laterite ore acid leaching solution after iron-aluminium removal according to claim 5, wherein at least one solute in the precipitation inducer is sodium hydroxide or potassium hydroxide, and at least one solute is calcium hydroxide, calcium oxide, magnesium oxide, or magnesium hydroxide.

8. The nickel-cobalt precipitation method for the nickel laterite ore acid leaching solution after iron-aluminium removal according to claim 5, wherein the solute of the precipitation inducer is sodium hydroxide, potassium hydroxide and magnesium oxide, and the weight ratio between these three is (3~4):(3~4):(2~4).

9. The nickel-cobalt precipitation method for the nickel laterite ore acid leaching solution after iron-aluminium removal according to claim 5, wherein the theoretical mole number of hydroxyls required for the nickel-cobalt precipitation in the mixed solution is used as a benchmark, the mole number of hydroxyls in the precipitation inducer is 0.7-1.2 times the theoretical mole number.

10. The nickel-cobalt precipitation method for the nickel laterite ore acid leaching solution after iron-aluminium removal according to claim 9, wherein the temperature of the alkali conversion precipitation reaction is 50~70°C, and the time is 0.5~5 h.

11. The nickel-cobalt precipitation method for the nickel laterite ore acid leaching solution after iron-aluminium removal according to any one of claims 1 to 4, wherein the weight ratio of the first part of the underflow to the second part of the underflow is 1: (1~15).

12. The nickel-cobalt precipitation method for the nickel laterite ore acid leaching solution after iron-aluminium removal according to claim 11, wherein in step S400, the precipitation inducer is added to a part of the overflow which accounts for 0.5~10% of the total weight of the overflow.

13. The nickel-cobalt precipitation method for the nickel laterite ore acid leaching solution after iron-aluminium removal according to any one of claims 1 to 4, wherein in step S400, the temperature of the alkali conversion reaction is 60~70°C, and the time is 1~5 min.

14. The nickel-cobalt precipitation method for the nickel laterite ore acid leaching solution after iron-aluminium removal according to any one of claims 1 to 4, wherein the positive ion components in the nickel laterite ore acid leaching solution after iron-aluminium removal are as follows: 2.5~5 g/L of nickel ions, 0.2~0.5 g/L of cobalt ions, 3~15 g/L of magnesium ions, and 1.5~5 g/L of divalent manganese ions.

Drawings

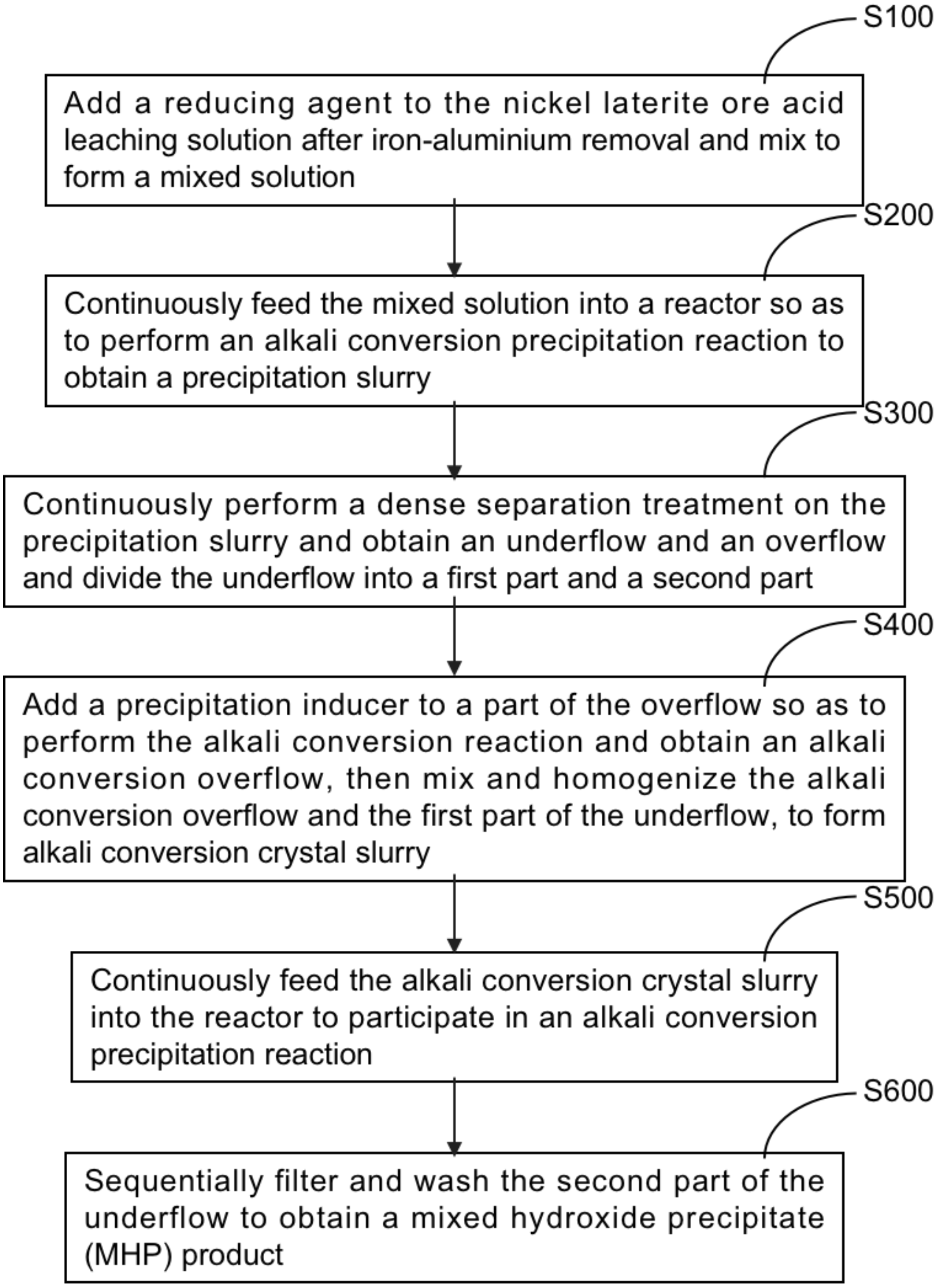
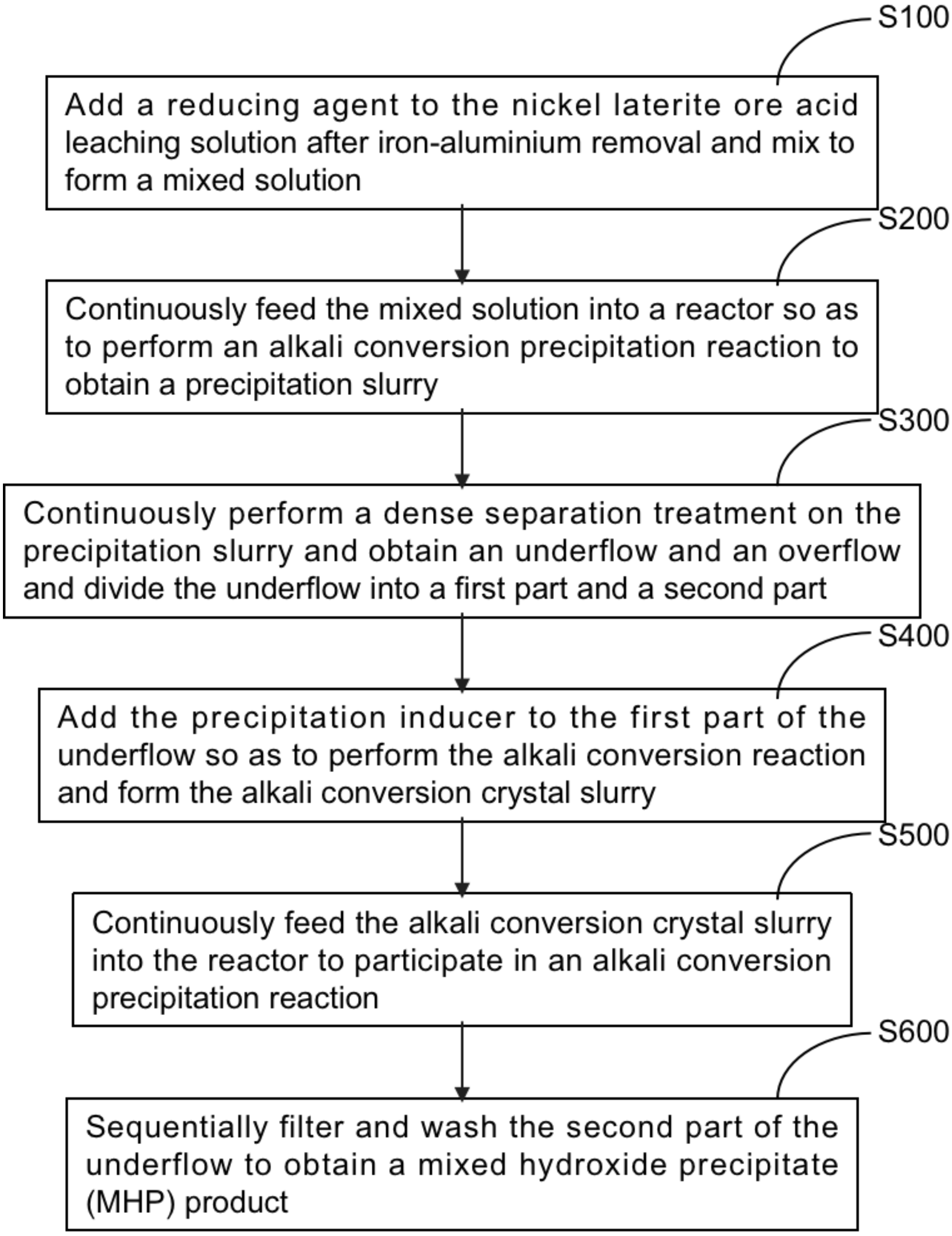


Fig. 1



Add a reducing agent to the nickel laterite ore acid leaching solution after iron-aluminium removal and mix to form a mixed solution

Continuously feed the mixed solution into a reactor so as to perform an alkali conversion precipitation reaction to obtain a precipitation slurry

Continuously perform a dense separation treatment on the precipitation slurry and obtain an underflow and an overflow and divide the underflow into a first part and a second part

Add the precipitation inducer to the first part of the underflow so as to perform the alkali conversion reaction and form the alkali conversion crystal slurry

Continuously feed the alkali conversion crystal slurry into the reactor to participate in an alkali conversion precipitation reaction

Sequentially filter and wash the second part of the underflow to obtain a mixed hydroxide precipitate (MHP) product

Fig. 2