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(54) **Titre :** CARBONATE DE COBALT A FAIBLE TENEUR EN METAL ALCALIN, METHODE DE PRODUCTION CONNEXE ET OXYDE DE COBALT OBTENU PAR LADITE METHODE

(54) **Title:** COBALT CARBONATE OF LOW ALKALI METAL CONTENT, METHOD FOR PRODUCING THE SAME AND COBALT OXIDE PRODUCED FROM THE SAME

(57) **Abrégé/Abstract:**

Disclosed are a method for producing a cobalt carbonate of low alkali metal content at a low cost in high productivity, and a cobalt oxide of low alkali metal content and high performance produced from the cobalt carbonate. The method comprises reacting an aqueous cobalt salt solution with a carbonate of an alkali metal to produce the cobalt carbonate; wherein: (1) a reaction temperature is controlled at less than or equal to 25°C, and/or (2) an aqueous solution of the carbonate of an alkali metal containing a hydroxide of the alkali metal in an amount of 5 to 40g/L is used as the carbonate of the alkali metal.

ABSTRACT

Disclosed are a method for producing a cobalt carbonate of low alkali metal content at a low cost in high productivity, and a cobalt oxide of low alkali metal content and high performance produced from the cobalt carbonate. The method comprises reacting an aqueous cobalt salt solution with a carbonate of an alkali metal to produce the cobalt carbonate; wherein:

(1) a reaction temperature is controlled at less than or equal to 25°C, and/or

(2) an aqueous solution of the carbonate of an alkali metal containing a hydroxide of the alkali metal in an amount of 5 to 40g/L is used as the carbonate of the alkali metal.

SPECIFICATION

COBALT CARBONATE OF LOW ALKALI METAL CONTENT, METHOD FOR PRODUCING THE SAME AND COBALT OXIDE PRODUCED FROM THE SAME

BACKGROUND OF THE INVENTION

FIELD OF THE INVENTION

The present invention relates to cobalt carbonate of a low alkali metal content, a method for producing the same and cobalt oxide produced from the same, more specifically a method for producing cobalt carbonate of a low alkali metal content at a low cost in high productivity, and cobalt oxide of a low alkali metal content and high performance produced from the cobalt carbonate.

DESCRIPTION OF THE PRIOR ART

Cobalt oxide has been used as a pigment for potteries and a colorant for glass products. Recently, a demand of the cobalt oxide has been growing rapidly as a ceramic material for electronic device parts, e.g., those for condensers, varistors and thermistors, and as a material for lithium ion batteries and organic synthesis catalysts. When the cobalt oxide is used for the above-mentioned purposes, in particular for the electronic device and the battery, an alkali metal is cited as an impurity which causes various problems, e.g., abnormal sintering, reduced electronic resistance and deteriorated electromagnetic properties, while they are produced or in service. In particular, it is highly desirable to contain no sodium.

It is difficult for a common method used for removing the alkali metal, e.g., by wet washing or evaporation under heating, to remove efficiently and sufficiently the alkali metal from cobalt oxide containing an alkali metal. Therefore, a cobalt salt as a starting material for cobalt oxide is pretreated to reduce alkali metal content. Several methods for producing a cobalt salt of low alkali metal content have been proposed, and the representative ones are described below. They have been contributing to production of cobalt oxide of low alkali metal content, but each involves its own disadvantages.

(1) Precipitation of highly filterable cobalt basic carbonate is produced from an aqueous cobalt salt solution using a sodium-free carbonate of an alkali, e.g., ammonium bicarbonate or ammonium carbonate. This method can produce cobalt oxide of low ammonium content by washing the precipitate, which is highly filterable, and subsequent calcination to reduce ammonium content. However, the method involves disadvantages of high production cost, because ammonium bicarbonate used as a reactant is relatively expensive, and, in particular, high cost for waste solution treatment because ammonium ions are massively present in the solution.

(2) Production of cobalt hydroxide from an aqueous cobalt salt solution using an alkali metal hydroxide, and subsequent removal of impurities, e.g., an alkali metal, from the resulting precipitate by repeated washing (refer to, e.g., Japanese Patent Publication No. 55-62814). This method involves a disadvantage of low productivity resulting from difficulties in washing and filtration on a commercial scale because the precipitate is of very fine cobalt hydroxide particles, very difficult to filtrate and settle.

(3) Precipitation of cobalt basic carbonate produced from an aqueous cobalt salt solution using sodium

carbonate or sodium bicarbonate, and subsequent washing the resulting precipitate with a sodium hydroxide solution and then with water, to produce cobalt basic carbonate of low sodium content (refer to, e.g., Japanese Patent Publication No. 7-196323). This method can remove sodium by washing a water-insoluble double salt e.g., sodium cobaltate dicarbonate ($Na_2[Co(CO_3)_2]\cdot4H_2O$), which is precipitated as a by-product in the case of high carbonate ion content in a solution, with a sodium hydroxide solution to decompose the salt and thereby to produce cobalt carbonate of low sodium content. However, it involves disadvantages of increased costs, e.g., those resulting from an additional step of alkali washing needed before a water-washing step, which is normally adopted, and those associated with use of sodium hydroxide or the like.

As discussed above, cobalt carbonate, e.g., cobalt basic carbonate, is a more preferable starting compound for cobalt oxide of low sodium content than cobalt hydroxide, because it is highly filterable and can be easily treated by water-washing. Moreover, a carbonate of an alkali metal is preferable than a carbonate of an alkali, because it can avoid problems associated with waste water treatment.

Under these circumstances, there are demands for methods for producing cobalt carbonate of low alkali metal content at a low cost in high productivity, and for cobalt oxide of low alkali metal content and high performance produced from the cobalt carbonate. The term cobalt carbonate used in this specification means a cobalt carbonate compound in the broad sense, including cobalt basic carbonate having hydroxide group in addition to carbonate group.

SUMMARY OF THE INVENTION

It is an object of the present invention to provide a method for producing cobalt carbonate of low alkali metal content at a low cost in high productivity in consideration of the problems involved in the conventional techniques. It is another object of the present invention to provide cobalt oxide of low alkali metal content and high performance produced from the cobalt carbonate.

The inventors of the present invention have found, after having extensively studied, in particular, cobalt carbonate for a method for producing cobalt oxide to attain the above objects, that cobalt carbonate of low alkali metal content can be produced at a low cost in high productivity when specific reaction conditions are adopted, and have achieved the present invention.

A first aspect of the present invention provides a method for producing cobalt carbonate of low alkali metal content, which comprises reacting an aqueous cobalt salt solution with a carbonate of an alkali metal to produce the cobalt carbonate under at least one of the following conditions:

(1) a reaction temperature is controlled at 25°C or lower, and

(2) an aqueous solution of an alkali metal carbonate containing of an alkali metal hydroxide in an amount of 5 to 40g/L is used as the carbonate of an alkali metal.

In a first major embodiment of the first aspect, a sodium salt is used as the carbonate of an alkali metal.

In a second major embodiment of the method, sodium bicarbonate is used as the carbonate of an alkali metal.

In a third major embodiment of the method, the reaction temperature is controlled at 10 to 20°C.

In a fourth major embodiment of the method, sodium hydroxide is used as the hydroxide of an alkali metal.

The second aspect of the present invention provides cobalt carbonate of low alkali metal content produced by the method.

A third aspect of the present invention provides cobalt oxide of low alkali metal content produced from the cobalt carbonate.

The method of the present invention can produce cobalt carbonate of low alkali metal content at a low cost in high productivity, and cobalt oxide produced from the cobalt carbonate contains an alkali metal at a low content and exhibits high performance. As such, they have very high industrial values.

DETAILED DESCRIPTION OF THE INVENTION

Cobalt carbonate of low alkali metal content, the method for producing the same and cobalt oxide produced from the same of the present invention are described in more detail below.

The method of the present invention produces cobalt carbonate of low alkali metal content by a reaction of a carbonate of an alkali metal with a cobalt salt in the form of an aqueous solution, wherein (1) a reaction temperature is controlled at 25°C or lower, and/or (2) an aqueous solution of carbonate of an alkali metal containing a hydroxide of alkali metal in an amount of 5 to 40g/L is used as the carbonate of an alkali metal. The cobalt carbonate of low alkali metal content is used for producing cobalt oxide.

1. Method for producing cobalt carbonate

The method of the present invention produces cobalt carbonate by the reaction of a carbonate of an alkali metal with a cobalt salt in the form of an aqueous solution, while satisfying at least one of the conditions (1) and (2):

(1) a reaction temperature is controlled at 25°C or lower, and

(2) an aqueous solution of carbonate of an alkali metal containing a hydroxide of an alkali metal at a concentration of 5 to 40g/L is used as the carbonate of an alkali metal.

More specifically, the method of the present invention produces cobalt carbonate by the reaction of a carbonate of an alkali metal with a cobalt salt in the form of an aqueous solution, wherein (A) a reaction temperature is controlled at 25°C or lower, (B) an aqueous solution of carbonate of an alkali metal containing a hydroxide of an alkali metal at 5 to 40g/L is used as the carbonate of an alkali metal, or (C) an aqueous solution of carbonate of an alkali metal containing a hydroxide of an alkali metal at 5 to 40g/L is used as the carbonate of an alkali metal and, at the same time, reaction temperature is controlled at 25°C or lower.

Each of controlling reaction temperature at 25°C or lower, and using an aqueous solution of carbonate of an alkali metal containing a hydroxide of an alkali metal at 5 to 40g/L as the carbonate of an alkali metal has an essential meaning for the method of the present invention for producing cobalt carbonate. Each leads to a solution for producing cobalt carbonate of low alkali metal content in high productivity on a commercial scale, which is a technical theme for the conventional techniques.

By contrast, the conventional method for producing cobalt carbonate cannot give cobalt carbonate of low alkali metal content by a simple procedure for the following technical problems. For example, some of the conventional methods react a cobalt salt, e.g., cobalt chloride, nitrate or sulfate, in the form of aqueous solution with sodium carbonate or bicarbonate incorporated in the solution to produce cobalt carbonate in the form of precipitate. The resulting cobalt carbonate contains sodium at 1 to 4% by weight, which cannot be removed by water-washing.

The high content of sodium conceivably results from a water-insoluble double salt of alkali metal, e.g., sodium cobaltate dicarbonate ($Na_2[Co(CO_3)_2]\cdot 4H_2O$) partly formed in the system to contaminate the product precipitate of cobalt carbonate. The reaction mechanism for producing the double salt conceivably involves the Co ion which forms the Co-carbonate complex ion in a solution of high carbonate ion concentration and also forms a double salt of the complex ion with a monovalent cation, e.g., that of sodium.

It is therefore essential to allow the cobalt carbonate producing reaction as the major reaction to preferentially proceed while controlling the double salt producing reaction, for which each of controlling reaction temperature at 25℃ or lower, and using an aqueous solution of carbonate of an alkali metal containing a hydroxide of alkali metal at a given content as the carbonate of an alkali metal is effective.

The method for controlling the reaction for the above production method is not limited. For example, the reaction can be controlled by incorporating an aqueous solution containing a cobalt salt at a given content with an aqueous solution containing a carbonate of an alkali metal at a given content in a given mixing ratio, or by controlling addition rate of an aqueous solution of carbonate of an alkali metal in such a way to control the reaction solution at a given pH level.

For the former approach, content of the carbonate of an alkali metal in the aqueous solution of cobalt salt is not limited, but preferably 1.0 to 1.5 chemical equivalents required for transforming cobalt in the aqueous solution into $CoCO_3$ in order to recover cobalt at a high rate. For the latter approach, pH level of the reaction solution is not limited, but preferably 6.5 to 8.0 in order to recover cobalt at a high rate.

The carbonate of an alkali metal for the above production method is not limited. For example, sodium or potassium is used as the alkali metal. In particular, sodium carbonate and sodium bicarbonate are preferable for their inexpensiveness. Sodium bicarbonate is more preferable, because it is low in alkalinity, and hence excellent in pH controllability for the reaction to keep sodium content at a low level more easily.

(1) Production Method (A)

Production Method (A) involves a procedure to control reaction temperature at 25℃ or lower, wherein reaction temperature is controlled at 25℃ or lower, preferably 10 to 20℃, more preferably 14 to 18℃. More specifically, decreasing reaction temperature retards the double salt producing reaction more than the cobalt carbonate producing reaction as the major reaction. Therefore, it is decreased to room temperature or below to control production of the double salt. The reaction system operating at above 25℃ cannot give cobalt carbonate of sufficiently low alkali metal content for electronic device and battery materials because of its insufficient effect of controlling the double salt producing reaction. Decreasing reaction temperature increases the effect of controlling the double salt producing reaction. However, it is 10℃ or higher for the present invention, because decreasing the level excessively is accompanied by increased cooling system cost.

(2) Production Method (B)

Production Method (B) involves a procedure which uses an aqueous solution of carbonate of an alkali metal containing a hydroxide of an alkali metal at 5 to 40g/L as the carbonate of an alkali metal. More specifically, incorporation of a hydroxide of an alkali metal brings favorable effects, e.g., increasing OH^- ion in the reaction solution while decreasing CO_3^{2-} ion content, and triggering the reaction for producing the hydroxide simultaneously with that for producing the carbonate, the former proceeding faster than the latter, thereby accelerating production of the hydroxide and carbonate while controlling production of the double salt to decrease alkali metal content.

In Production Method (B), a hydroxide of an alkali metal is incorporated in the aqueous solution of carbonate of an alkali metal at 5 to 40g/L, preferably 20 to 40g/L, more preferably 20 to 30g/L. At below 5g/L, the effect of decreasing sodium content of the cobalt carbonate product may be insufficient. At above 40g/L, on the other hand, cobalt hydroxide may be the major component of the precipitate produced, because the reaction for producing the hydroxide will prevail over that for producing the carbonate. As a result, the precipitate is composed of finer particles, to have deteriorated filterability and washability.

Reaction temperature for the above method is not limited, and it is carried out at a reaction solution temperature of room temperature or higher. The hydroxide of an alkali metal for the above method is not limited. For example, sodium or potassium is used as the alkali metal. Sodium hydroxide is particularly preferable because of its inexpensiveness.

(3) Production Method (C)

Production Method (C) is a combination of Production Methods (A) and (B), involving a procedure which uses an aqueous solution of carbonate of an alkali metal containing a hydroxide of an alkali metal at 5 to 40g/L as the carbonate of an alkali metal, and another procedure to control reaction temperature at 25℃ or lower. Each of these procedures is carried out under the conditions similar to those for Production Method (A) or (B).

2. Cobalt carbonate

Cobalt carbonate of the present invention is the one containing an alkali metal at a low content, produced by one of Production Methods (A) to (C) described above. The cobalt carbonate precipitate produced is separated into the solid and reaction filtrate by centrifugal separation or the like, and the solid is washed with water and then dehydrated.

Cobalt carbonate of the present invention contains an alkali metal at 0.3% or less and 0.1% or less, both by weight, when produced by Production Method (A) or (C) operating at 25 and 20℃, respectively, and 0.7% or less and 0.3% or less, both by weight, when produced by Production Method (B) operating under the preferable conditions for the latter. By contrast, cobalt carbonate produced by the conventional method operating at 30 to 70℃ contains an alkali metal at 1 to 4% by weight.

As discussed above, the production method of the present invention gives cobalt carbonate of lower alkali metal content.

3. Cobalt oxide

Cobalt oxide of low alkali metal content of the present invention is produced from the cobalt carbonate as the starting compound, which is produced by the method described above and calcined. It contains an alkali metal at a lower content than the one produced by the conventional method, and is suitable as materials for electronic device and battery, for which materials containing an alkali metal, e.g., sodium, is unsuitable.

The method for producing the cobalt oxide is not limited. It is produced by calcination of the cobalt carbonate, preferably heated at 350℃ or higher in air. At a calcination temperature below 350℃, the carbonate may be decomposed insufficiently and left undecomposed in the product.

EXAMPLES

The present invention is described in more detail by EXAMPLES and COMPARATIVE EXAMPLES, which by no means limit the present invention. Cobalt carbonate was analyzed for metal and moisture contents by the following procedures in EXAMPLES and COMPARATIVE EXAMPLES.

(1) Analysis of metal contents

The dehydration-treated sample was dried at 105℃ for 24 hours, and analyzed by ICP emission spectrometry.

(2) Analysis of moisture content

The dehydration-treated sample was dried at 105℃ for 24 hours, and measured for weight difference before and after the drying treatment.

(EXAMPLE 1)

Cobalt carbonate was prepared by Production Method (A), and evaluated for its sodium content.

First, 20L of a 140g/L aqueous solution of sodium bicarbonate as the carbonate of an alkali metal was put in a reaction tank, to which 20L of a 50g/L aqueous solution of cobalt chloride was added in 30 minutes for the reaction, while reaction temperature was kept at 18℃ with stirring. The carbonate of the alkali metal was added to the aqueous cobalt salt solution at 1.0 chemical equivalent required for transforming cobalt into $CoCO_3$.

The reaction was allowed to continue for 20 minutes, and the resultant slurry was centrifugally separated into cobalt carbonate (solid) and reaction filtrate (liquid). The separated cobalt carbonate was repulp-washed with water and centrifugally dehydrated again. It was analyzed for sodium content. The result is given in Table 1.

(EXAMPLE 2)

Cobalt carbonate was prepared by Production Method (A), and evaluated for its sodium content.

First, 1L of a 50g/L aqueous solution of cobalt chloride was put in a reaction tank, to which a 100g/L aqueous solution of sodium carbonate (reagent grade) as the carbonate of an alkali metal was added for the reaction, which was allowed to proceed for 4 hours while reaction temperature was kept at 14℃ with stirring, where the reaction solution was adjusted at a final pH level of 7.7.

The resultant slurry was separated into cobalt carbonate (solid) and reaction filtrate (liquid) by filtration under a vacuum. The separated cobalt carbonate was repulp-washed with water and dehydrated again by filtration under a vacuum. It was analyzed for sodium content. The result is given in Table 1.

(EXAMPLE 3)

Cobalt carbonate was prepared by Production Method (A), and evaluated for its sodium content.

It was prepared in the same manner as in EXAMPLE 2, except that a 73g/L aqueous solution of sodium bicarbonate (reagent grade) was used as the carbonate of an alkali metal and the reaction solution was adjusted at a final pH level of 6.9. The resultant cobalt carbonate was analyzed for sodium content. The result is given in Table 1.

(EXAMPLE 4)

Cobalt carbonate was prepared by Production Method (A), and evaluated for its sodium content.

It was prepared in the same manner as in EXAMPLE 1, except that reaction temperature was controlled at 25℃. The resultant cobalt carbonate was analyzed for sodium content. The result is given in Table 1.

(EXAMPLE 5)

Cobalt carbonate was prepared by Production Method (C), and evaluated for its sodium and moisture content.

First, a 100g/L aqueous solution of sodium carbonate (reagent grade) as the carbonate of an alkali metal was incorporated with sodium hydroxide to 10g/L. Next, the above aqueous solution containing the alkali metal was pumped into a 1L reactor together with a 50g/L aqueous solution of cobalt chloride (reagent grade), and the reaction was allowed to proceed while reaction temperature was kept at 18℃ with stirring. These aqueous solutions were pumped in such a way to keep the residence time at 1 hour and reaction solution pH level at 7.5.

The resultant slurry was separated into cobalt carbonate (solid) and reaction filtrate (liquid) by filtration under a vacuum. The separated cobalt carbonate was washed with sprayed water and dehydrated again by filtration under a vacuum. It was analyzed for sodium content. The result is given in Table 1.

(EXAMPLES 6 to 9)

Cobalt carbonate was prepared by Production Method (B) in each of EXAMPLES 6 to 9, and evaluated for its sodium and moisture contents.

First, a 100g/L aqueous solution of sodium carbonate (reagent grade) as the carbonate of an alkali metal was incorporated with sodium oxide to 5, 10, 20 or 40g/L. Next, the above aqueous solution containing the alkali metal was pumped into a 1L reactor together with a 50g/L aqueous solution of cobalt chloride (reagent grade), and the reaction was allowed to proceed while reaction temperature was kept at 70℃ with stirring. These aqueous solutions were pumped in such a way to keep the residence time at 1 hour and reaction solution pH level at 7.5.

The resultant slurry was separated into cobalt carbonate (solid) and reaction filtrate (liquid) by filtration under a vacuum. The separated cobalt carbonate was washed with sprayed water and dehydrated again by filtration under a vacuum. It was analyzed for sodium, cobalt and moisture contents. The result is given in Table 2.

(COMPARATIVE EXAMPLE 1)

Cobalt carbonate was prepared in the same manner as in EXAMPLE 6, except that sodium hydroxide was incorporated in the aqueous sodium carbonate solution to 50g/L as the aqueous carbonate solution containing the alkali metal. The resulting cobalt carbonate showed deteriorated filterability. It was analyzed for sodium, cobalt and moisture contents. The result is given in Table 2.

(COMPARATIVE EXAMPLE 2)

Cobalt carbonate was prepared in the same manner as in EXAMPLE 6, except that sodium hydroxide was not incorporated in the aqueous sodium carbonate solution as the aqueous carbonate solution containing the alkali metal. It was analyzed for sodium, cobalt and moisture contents. The result is given in Table 2

Table 1

	Production Method	Reaction temperature (℃)	Slurry pH during the reaction process	Na content of cobalt carbonate (% by weight)
EXAMPLE 1	(A)	18	-	0.01
EXAMPLE 2	(A)	14	7.7	0.02
EXAMPLE 3	(A)	14	6.9	0.01
EXAMPLE 4	(A)	25	-	0.30
EXAMPLE 5	(C)	18	-	0.02

Table 2

	Production Method	NaOH content of the aqueous Na_2CO_3 solution	Reaction temperature (℃)	Na, Co and moisture contents of cobalt carbonate (% by weight)		
				Na	Co	moisture contents*
EXAMPLE 6	(B)	5	70	0.68	49.0	26.0
EXAMPLE 7	(B)	10	70	0.52	51.2	25.9
EXAMPLE 8	(B)	20	70	0.22	51.8	27.7
EXAMPLE 9	(B)	40	70	0.13	52.5	50.4
COMPARATIVE EXAMPLE 1	(B)	50	70	0.13	52.8	62.5*
COMPARATIVE EXAMPLE 2	-	0	70	0.92	50.3	22.4

* Cobalt carbonate showed deteriorated filterability when it contained moisture at 60% or more.

As shown in Table 1, cobalt carbonate prepared in each of EXAMPLES 1 to 4 by Production Method (A) and in EXAMPLE 5 by Production Method (C) at a reaction temperature for the present invention contained sodium at a low level.

As shown in Table 2, cobalt carbonate prepared in each of EXAMPLES 6 to 9 by Production Method (B) using the aqueous solution of carbonate of an alkali metal incorporated with sodium hydroxide at a given content for the present invention contained sodium at a low level. By contrast, cobalt carbonate prepared in each of COMPARATIVE EXAMPLES 1 and 2 using the aqueous solution of carbonate of an alkali metal incorporated with sodium hydroxide at a content out of the range for the present invention showed unsatisfactory result with respect to sodium content or moisture content (which relates to filterability).

As discussed above, cobalt oxide produced from cobalt carbonate of low alkali metal content of the present invention, produced by the method of the present invention, contains an alkali metal at a sufficiently low content to be suitable as materials for electronic device and battery, in particular for purposes for which materials containing an alkali metal, e.g., sodium, is unsuitable.

CLAIMS:

1. A method for producing cobalt carbonate of low alkali metal content, which comprises:

reacting an aqueous cobalt salt solution with a carbonate of an alkali metal to produce the cobalt carbonate, under at least one of the following conditions:

(1) a reaction temperature is controlled at 25°C or lower, and

(2) an aqueous solution of an alkali metal carbonate containing an alkali metal hydroxide in an amount of 5 to 40g/L is used as the carbonate of an alkali metal.

2. The method according to claim 1, wherein the carbonate of an alkali metal is a sodium salt.

3. The method according to claim 2, wherein the carbonate of an alkali metal is sodium bicarbonate.

4. The method according to claim 1, 2 or 3, wherein the reaction temperature is controlled at 10 to 20°C.

5. The method according to claim 1, 2, 3 or 4, wherein the alkali metal hydroxide is sodium hydroxide.

6. Cobalt carbonate of low alkali metal content produced by the method according to any one of claims 1 to 5.

7. Cobalt oxide of low alkali metal content produced from the cobalt carbonate according to claim 6.

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