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(54) Title: CATALYST FOR HYDROLYSIS OF CARBONYL SULFIDE AND HYDROGEN CYANIDE AND USE OF TITANIUM OXIDE-BASED COMPOSITION

(54) 発明の名称: 硫化カルボニルおよびシアン化水素の加水分解用触媒ならびに酸化チタン系組成物の使用

(57) Abstract: Provided are: a hydrolysis catalyst which is capable of removing COS and HCN at the same time at high decomposition rate; and a use of a titanium oxide-based composition. A catalyst for hydrolysis of carbonyl sulfide and hydrogen cyanide, which is characterized by containing at least an active component that is mainly composed of at least one metal selected from the group consisting of barium, nickel, ruthenium, cobalt and molybdenum, and a titanium oxide-based carrier that supports the active component.

(57) 要約: COSやHCNを同時に高分解率で除去することが可能な加水分解用触媒、および酸化チタン系組成物の使用について提供する。硫化カルボニルおよびシアン化水素の加水分解用触媒であって、バリウム、ニッケル、ルテニウム、コバルト、モリブデンからなる群から選択される金属の少なくとも1種を主成分とする活性成分と、前記活性成分を担持する酸化チタン系担体とを少なくとも含むことを特徴とする加水分解用触媒。

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## CATALYST FOR HYDROLYSIS OF CARBONYL SULFIDE AND HYDROGEN CYANIDE AND USE OF TITANIUM DIOXIDE-BASED COMPOSITION

[0001]

The present invention relates to a catalyst for hydrolysis of carbonyl sulfide and hydrogen cyanide and use of a titanium dioxide-based composition.

[0002]

Effective utilization of coal has attracted attention as a last resort to the recent energy issue. To convert coal to a high-value added energy medium, advanced technologies such as a technology for gasifying coal and a technology for purifying the gasified gas are employed.

10 [0003]

The gasified coal gas contains carbonyl sulfide (COS) and hydrogen cyanide (HCN), which cause air pollution. Hence, it is important to remove these compounds in a gas purification step.

A purification process of gasified coal gas may be configured, for example, as shown in Fig. 1. Specifically, gas obtained by gasifying coal in a gasification furnace 4 and subjected to a dust removal treatment in a dust removal apparatus 5 is introduced into a COS conversion apparatus 6 to convert COS in the gas to carbon dioxide (CO<sub>2</sub>) and hydrogen sulfide (H<sub>2</sub>S), which are then recovered in an H<sub>2</sub>S/CO<sub>2</sub> recovery apparatus 7. The thus purified gas is then used for synthesis 8 of chemicals, electric power generation 9, or the like.

20 [0004]

Moreover, systems are also proposed in which the purified gas obtained by gasifying

and purifying coal is applied to synthesis of chemicals such as methanol and ammonia or used directly for electric power generation. The electric power generation system includes the integrated coal gasification combined cycle (IGCC) system (for example, Patent Document 1). Specifically, in this system, coal is converted to a combustible gas in a high-temperature and high-pressure gasification furnace, and electric power is generated by a combination of a gas turbine and a steam turbine using the gasified gas as a fuel.

[0005]

The COS conversion is based on the hydrolysis reaction represented by the following Formula (1), and an example of the catalyst for the hydrolysis is a catalyst obtained by supporting a metal carbonate on titanium dioxide (Patent Document 2).

[0006]

[Math 1]



[0007]

Meanwhile, HCN can be converted to ammonia ( $\text{NH}_3$ ) by the hydrolysis reaction of the following Formula (2) or the reaction of Formula (3), and the ammonia can be recovered. An example of the catalyst for hydrolysis of HCN is a titanium dioxide-based composition containing a combination of titanium dioxide, a sulfate of an alkaline-earth metal, and a doping compound (Patent Document 3).

[0008]

[Math 2]



[Prior Art Documents]

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[Patent Documents]

[0009]

[Patent Document 1] Japanese Patent Application Publication No. 2004-331701

[Patent Document 2] Japanese Patent No. 3746609

5 [Patent Document 3] Japanese Patent No. 4556159

[0010]

Gasification and purification of coal requires many steps (Fig. 1). Hence, conducting each step in shorter period leads to shortening of the production period and reduction in costs.

Providing a catalyst having better decomposition performance than hydrolysis catalysts  
10 used so far in the conversion step of COS and HCN is an objective of embodiments of the present invention.

[0011]

The present inventors have focused on the conversion step of COS and HCN, and reinvestigated the reaction mechanisms and reaction catalysts of the hydrolysis reactions of  
15 COS and HCN.

As a result, the present inventors have found that COS and HCN can be removed simultaneously at high degradation percentages when an active component containing a predetermined metal as a main component is supported on a titanium dioxide-based support having excellent corrosion resistance in a highly dispersed manner. This finding has led to  
20 embodiments of the present invention.

[0012]

The invention provides a catalyst for hydrolysis of carbonyl sulfide and hydrogen cyanide, comprising:

an active component containing at least one metal salt selected from the group  
25 consisting of barium carbonate, nickel carbonate, ruthenium nitrate, and cobalt carbonate;  
and

- 4 -

a titanium dioxide-based support supporting the active component, and selected from the group consisting of composite oxides of titanium dioxide and silicon dioxide, composite oxides of titanium dioxide and aluminum oxide, and composite oxides of titanium dioxide and zirconium dioxide.

5 [0013]

The invention also provides the use of a titanium dioxide-based composition as a catalyst for hydrolyzing carbonyl sulfide and hydrogen cyanide, wherein the composition comprises:

an active component containing at least one metal salt selected from the group  
10 consisting of barium carbonate, nickel carbonate, ruthenium nitrate, and cobalt carbonate; and

a titanium dioxide-based support supporting the active component, and selected from the group consisting of composite oxides of titanium dioxide and silicon dioxide, composite oxides of titanium dioxide and aluminum oxide, and composite oxides of  
15 titanium dioxide and zirconium dioxide.

[0014]

The catalyst for hydrolysis of carbonyl sulfide and hydrogen cyanide and the use of a titanium dioxide-based composition of embodiments of the present invention make it possible to remove COS and HCN simultaneously at high degradation percentages.

20 [0014A]

The present invention will now be described, by way of non-limiting example only, with reference to the accompanying drawings briefly described below.

[0015]

Fig. 1 is a schematic diagram of an entire coal gasification and purification process.

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[0016]

Hereinafter, a catalyst for hydrolysis of carbonyl sulfide and hydrogen cyanide and use of a titanium dioxide-based composition of embodiments of the present invention are described in detail.

5 First, the catalyst of embodiments of the present invention is a catalyst for hydrolyzing carbonyl sulfide and hydrogen cyanide, and comprises at least an active component containing, as a main component, at least one metal selected from the group consisting of barium, nickel, ruthenium, cobalt, and molybdenum. The use of any of these metals as the main component makes it possible to achieve satisfactorily high decomposition performance  
10 of not only carbonyl sulfide but also hydrogen cyanide. The main component may be a combination of any of the above-described metals, and the amount of the main component supported is preferably 0.1 to 25% by mass, and more preferably 5 to 22% by mass.

Moreover, the catalyst of embodiments of the present invention comprises a titanium dioxide-based support supporting the active component. The titanium dioxide-based support  
15 may be titanium dioxide, a composite oxide of titanium dioxide with another oxide, or the like.

Such a support can immobilize the active component reliably. In addition, the support is chemically stable under operating conditions of the catalyst, and hence does not inhibit the activity of the catalyst.

[0017]

20 Next, the use of embodiments of the present invention is use of a titanium dioxide-based composition as a catalyst for hydrolyzing carbonyl sulfide and hydrogen cyanide. The titanium dioxide-based composition comprises at least an active component containing, as a main component, at least one metal selected from the group consisting of barium, nickel, ruthenium, cobalt, and molybdenum. Moreover, the titanium dioxide-based composition  
25 comprises a titanium dioxide-based support supporting the active component. The amount of the main component supported is preferably 0.1 to 25% by mass and more preferably 5 to 22% by mass. In addition, the titanium dioxide-based support may be titanium dioxide, a composite oxide of titanium dioxide with another oxide, or the like, for the reasons described above.

[0018]

In the catalyst and the use of embodiments of the present invention, the titanium dioxide-based composition is preferably obtained by adding, to the titanium dioxide-based support, at least one metal salt selected from the group consisting of barium carbonate, nickel carbonate, ruthenium nitrate, cobalt carbonate, and ammonium molybdate. This is because these materials are stable as raw materials.

[0019]

In the titanium dioxide-based composition of the catalyst or the use of embodiments of the present invention, titanium dioxide can be used as the titanium dioxide-based support. The use of titanium dioxide having an anatase-type crystal structure with a large specific surface area as the titanium dioxide is more preferable, because the amount of the active component supported increases, so that the catalytic activity improves.

[0020]

Besides the above-described titanium dioxide, a titanium dioxide-based composite oxide can be used as the titanium dioxide-based support in the titanium dioxide-based composition of the catalyst or the use of embodiments of the present invention.

When titanium dioxide is converted to a composite metal oxide, the specific surface area increases, and the heat resistance also improves.

The titanium dioxide-based composite oxide may be at least one selected from the group consisting of composite oxides of titanium dioxide and silicon dioxide, composite oxides of titanium dioxide and aluminum oxide, and composite oxides of titanium dioxide and zirconium dioxide. The composite ratio of titanium dioxide to the metal oxide composited with the titanium dioxide is preferably 1:99 to 99:1, and particularly preferably in a range from 50:50 to 95:5. This is because the specific surface area on which the active component is supported can be large in this range.

[0021]

In the catalyst and the use of embodiments of the present invention, the titanium dioxide-based composition preferably has a honeycomb shape. This is because clogging and



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pressure drop of the catalyst and the titanium dioxide-based composition can be prevented even in a situation in which dust or the like is coexistent, so that the catalyst can be maintained in a highly active state.

[0022]

5           In the catalyst and the use of embodiments of the present invention, the titanium dioxide-based composition can be produced, for example, as follows. Specifically, any of the above-described metal salts is added to a powder of the titanium dioxide-based support, and further a binder and a plasticizer are added. Then, the mixture is kneaded, molded into a spherical shape, a pellet shape, or a honeycomb shape, as appropriate, and then dried and  
10   calcined.

          In addition, when the support is made of a titanium dioxide-based composite oxide, it is necessary to prepare the composite oxide in advance. The composite oxide can be prepared, for example, as follows. Specifically, coprecipitation is conducted by adding dropwise an alkali solution of ammonia or the like to an aqueous solution of metal salts such as nitrates,  
15   chlorides, and sulfates of metals such as titanium, silicon, aluminum, and zirconium. Thus, a composite hydroxide is formed. Then, the composite hydroxide is, for example, rinsed, dried, and calcined. The composite oxide can also be prepared by methods other than this method.

[Examples]

[0023]

20           Hereinafter, embodiments of the present invention are described more specifically based on Examples and Comparative Example. However, the present invention is not at all limited to the following Examples.

[0024]

[Production of Catalysts for Hydrolysis]

## [Example 1]

To 100 parts by mass of an anatase-type titanium dioxide powder (CSP-003 manufactured by JGC Catalysts and Chemicals Ltd.), 4 parts by mass, in terms of barium oxide, of barium carbonate (manufactured by HAYASHI PURE CHEMICAL IND., LTD., special grade reagent) was added, and 5 parts by mass of 10% ammonia water was added, followed by kneading with a kneader for 60 minutes. Next, to the kneaded material, 3 parts by mass of glass fiber and 5 parts by mass of kaolin as binders, 5 parts by mass of cellulose acetate as an organic plasticizer, and 5 parts by mass of 10% ammonia water were added, followed by kneading.

The kneaded material was extrusion molded to obtain a monolithic honeycomb molded article having a pitch of 5.0 mm and a wall thickness of 1.0 mm. The molded article was dried at room temperature until the water content reached 10%, and then calcined at 500°C for 5 hours to remove the organic plasticizer. Thus, a honeycomb catalyst was obtained.

[0025]

## [Example 2]

A honeycomb catalyst was obtained in the same method as in Example 1, except that 4 parts by mass, in terms of nickel oxide, of nickel carbonate (manufactured by HAYASHI PURE CHEMICAL IND., LTD., special grade reagent) was added instead of barium carbonate.

[0026]

## [Example 3]

A honeycomb catalyst was obtained in the same method as in Example 1, except that 0.1 parts by mass, in terms of ruthenium, of ruthenium nitrate (manufactured by Tanaka Kikinzoku Kogyo K. K., Ru content: 50 g/L) was added instead of barium carbonate.

[0027]

[Example 4]

A honeycomb catalyst was obtained in the same method as in Example 1, except that 4 parts by mass, in terms of nickel oxide, of nickel carbonate and 10 parts by mass, in terms of molybdenum oxide, of ammonium molybdate (manufactured by HAYASHI PURE CHEMICAL IND., LTD., special grade) were added instead of barium carbonate.

[0028]

[Example 5]

A honeycomb catalyst was obtained in the same method as in Example 1, except that 5 parts by mass, in terms of cobalt oxide, of cobalt carbonate (manufactured by HAYASHI PURE CHEMICAL IND., LTD., special grade) and 10 parts by mass, in terms of molybdenum oxide, of ammonium molybdate were added instead of barium carbonate.

[0029]

[Example 6]

A honeycomb catalyst was obtained in the same method as in Example 1, except that a composite oxide of titanium dioxide and silicon dioxide obtained as follows was used as the support. Specifically, 1125.8 g of  $\text{Ti}(\text{O}i\text{-C}_3\text{H}_7)_4$  as a Ti source and 57.6 g of  $\text{Si}(\text{OC}_2\text{H}_5)_4$  were mixed with each other. The mixture was hydrolyzed by being added to 15000 g of water at 80°C, and further aged by being stirred in water at the same temperature for 2 hours. After the aging, the obtained product was filtered and washed sufficiently. Then, the product was dried and calcined (at 500°C for 5 hours). Thus, the composite oxide of titanium dioxide and silicon dioxide was obtained.

[0030]

[Example 7]

A honeycomb catalyst was obtained in the same method as in Example 6, except that

a composite oxide of titanium dioxide and aluminum oxide obtained by using 316.8 g of  $\text{Al}(\text{O}i\text{-C}_3\text{H}_7)_3$  instead of  $\text{Si}(\text{OC}_2\text{H}_5)_4$  used in Example 6 was used as the support.

[0031]

[Example 8]

5           A honeycomb catalyst was obtained in the same method as in Example 6, except that a composite oxide of titanium dioxide and zirconium dioxide obtained by using 985.5 g of  $\text{Zr}(\text{O}-i\text{C}_4\text{H}_9)_4$  instead of  $\text{Si}(\text{OC}_2\text{H}_5)_4$  used in Example 6 was used as the support.

[0032]

[Comparative Example 1]

10           To 100 parts by mass of an anatase-type titanium dioxide powder (CSP-003 manufactured by JGC Catalysts and Chemicals Ltd.), 5 parts by mass of 10% ammonia water was added, followed by kneading with a kneader for 60 minutes. Next, to the kneaded material, 3 parts by mass of glass fiber and 5 parts by mass of kaolin as binders, 5 parts by mass of cellulose acetate as an organic plasticizer, and 5 parts by mass of 10%  
15 ammonia water were added, followed by kneading.

The kneaded material was extrusion molded to obtain a monolithic honeycomb molded article having a pitch of 5.0 mm and a wall thickness of 1.0 mm. This molded article was dried at room temperature until the water content reached 10%, and it was then calcined at 500°C for 5 hours to remove the organic plasticizer. Thus, a honeycomb catalyst was  
20 obtained.

[0033]

[Hydrolysis Reaction of COS]

Hydrolysis reaction of COS was conducted by using the catalysts of Examples and Comparative Example described above under the test conditions shown in Table 1. The  
25 COS conversion was calculated by the following Formula (4). Table 2 shows the results.

[0034]

[Math 3]

$$\text{COS conversion (\%)} = (1 - \text{Outlet COS concentration} / \text{Inlet COS concentration}) \times 100 \quad (4)$$

[0035]

5 [Table 1]

Catalyst amount: 10cc	(Gas composition)
Gas flow rate: 60literN/h	H <sub>2</sub> : 10%
GHSV: 6000h <sup>-1</sup>	CO: 25%
Pressure: 9kgf/cm <sup>2</sup>	H <sub>2</sub> O: 3.2%
Temperature: 300 °C	CO <sub>2</sub> : 3.5%
	COS: 210ppm
	H <sub>2</sub> S: 880ppm
	N <sub>2</sub> : the balance

[0036]

[Table 2]

	Catalyst composition	COS conversion(%)
Example 1	BaCO <sub>3</sub> /TiO <sub>2</sub>	79
Example 2	NiCO <sub>3</sub> /TiO <sub>2</sub>	78
Example 3	Ru(NO <sub>3</sub> ) <sub>3</sub> /TiO <sub>2</sub>	76
Example 4	NiCO <sub>3</sub> +(NH <sub>4</sub> ) <sub>6</sub> Mo <sub>7</sub> O <sub>24</sub> ·4H <sub>2</sub> O/TiO <sub>2</sub>	80
Example 5	CoCO <sub>3</sub> +(NH <sub>4</sub> ) <sub>6</sub> Mo <sub>7</sub> O <sub>24</sub> ·4H <sub>2</sub> O/TiO <sub>2</sub>	78
Example 6	BaCO <sub>3</sub> /TiO <sub>2</sub> SiO <sub>2</sub>	83
Example 7	BaCO <sub>3</sub> /TiO <sub>2</sub> Al <sub>2</sub> O <sub>3</sub>	80

Example 8	BaCO <sub>3</sub> /TiO <sub>2</sub> ZrO <sub>2</sub>	79
Comparative Example 1	TiO <sub>2</sub>	47

[0037]

The results in Table 2 showed that each of the catalysts of the Examples was better in COS conversion performance than the catalyst of Comparative Example 1.

[0038]

#### 5 [Hydrolysis Reaction of HCN]

Hydrolysis reaction of HCN was conducted by using the catalysts of Examples and Comparative Example described above and actual gasified coal gas shown in Table 3. The HCN conversion was calculated by the following Formula (5). Table 4 shows the results.

[0039]

#### 10 [Math 4]

$$\text{HCN conversion (\%)} = (1 - \text{Outlet HCN concentration} / \text{Inlet HCN concentration}) \times 100 \quad (5)$$

[0040]

[Table 3]

	(Gas composition)
Catalyst amount: 10 liter	Gasified gas composition
Gas flow rate: 60m <sup>3</sup> N/h	H <sub>2</sub> : 10%
GHSV: 6000h <sup>-1</sup>	CO: 25%
Pressure: 9kgf/cm <sup>2</sup>	H <sub>2</sub> O: 3.2%
Temperature: 300-350 °C	CO <sub>2</sub> : 3.5%
	COS: 210ppm
	H <sub>2</sub> S: 880ppm

	HCN : 1-70ppm N <sub>2</sub> : balance
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[0041]

[Table 4]

	Catalyst composition	HCN conversion (%)
Example 1	BaCO <sub>3</sub> /TiO <sub>2</sub>	100
Example 2	NiCO <sub>3</sub> /TiO <sub>2</sub>	99
Example 3	Ru(NO <sub>3</sub> ) <sub>3</sub> /TiO <sub>2</sub>	99
Example 4	NiCO <sub>3</sub> +(NH <sub>4</sub> ) <sub>6</sub> Mo <sub>7</sub> O <sub>24</sub> ·4H <sub>2</sub> O/TiO <sub>2</sub>	100
Example 5	CoCO <sub>3</sub> +(NH <sub>4</sub> ) <sub>6</sub> Mo <sub>7</sub> O <sub>24</sub> ·4H <sub>2</sub> O/TiO <sub>2</sub>	99
Example 6	BaCO <sub>3</sub> /TiO <sub>2</sub> SiO <sub>2</sub>	100
Example 7	BaCO <sub>3</sub> /TiO <sub>2</sub> Al <sub>2</sub> O <sub>3</sub>	99
Example 8	BaCO <sub>3</sub> /TiO <sub>2</sub> ZrO <sub>2</sub>	100
Comparative Example 1	TiO <sub>2</sub>	50

[0042]

The results in Table 4 showed that each of the catalysts of Examples was better in HCN decomposition performance than the catalyst of Comparative Example 1.

From these results, it is apparent that the catalyst of embodiments of the present invention is excellent in COS conversion performance and HCN decomposition performance.

[Industrial Applicability]

[0043]

The catalyst for hydrolysis of carbonyl sulfide and hydrogen cyanide and the use of a titanium dioxide-based composition of embodiments of the present invention are industrially useful, because COS and HCN can be removed simultaneously at high degradation percentages.

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[Reference Signs List]

[0043A]

Throughout this specification and the claims which follow, unless the context requires otherwise, the word "comprise", and variations such as "comprises" and "comprising", will be understood to imply the inclusion of a stated integer or step or group of integers or steps but not the exclusion of any other integer or step or group of integers or steps.

[0043B]

The reference in this specification to any prior publication (or information derived from it), or to any matter which is known, is not, and should not be taken as an acknowledgment or admission or any form of suggestion that that prior publication (or information derived from it) or known matter forms part of the common general knowledge in the field of endeavour to which this specification relates.

[0043C]

While various embodiments of the present invention have been described above, it should be understood that they have been presented by way of example only, and not by way of limitation. It will be apparent to a person skilled in the relevant art that various changes in form and detail can be made therein without departing from the spirit and scope of the invention. Thus, the present invention should not be limited by any of the above described exemplary embodiments.

20 [0044]

1 coal gasification and purification process

2 coal

3 oxygen

4 gasification furnace

25 5 dust removal apparatus

6 COS conversion apparatus



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- 7 H<sub>2</sub>S/CO<sub>2</sub> recovery apparatus
- 8 synthesis of chemicals
- 9 electric power generation

**THE CLAIMS DEFINING THE INVENTION ARE AS FOLLOWS:**

1. A catalyst for hydrolysis of carbonyl sulfide and hydrogen cyanide, comprising:  
an active component containing at least one metal salt selected from the group  
consisting of barium carbonate, nickel carbonate, ruthenium nitrate, and cobalt carbonate;  
5 and  
a titanium dioxide-based support supporting the active component, and selected  
from the group consisting of composite oxides of titanium dioxide and silicon dioxide,  
composite oxides of titanium dioxide and aluminum oxide, and composite oxides of  
titanium dioxide and zirconium dioxide.
- 10 2. The catalyst for hydrolysis according to claim 1, wherein the catalyst for hydrolysis  
has a honeycomb structure.
3. Use of a titanium dioxide-based composition as a catalyst for hydrolyzing carbonyl  
sulfide and hydrogen cyanide, wherein  
the composition comprises:  
15 an active component containing at least one metal salt selected from the group  
consisting of barium carbonate, nickel carbonate, ruthenium nitrate, and cobalt carbonate;  
and  
a titanium dioxide-based support supporting the active component, and selected  
from the group consisting of composite oxides of titanium dioxide and silicon dioxide,  
20 composite oxides of titanium dioxide and aluminum oxide, and composite oxides of  
titanium dioxide and zirconium dioxide.
4. The use of a titanium dioxide-based composition according to claim 3, wherein the  
composition has a honeycomb structure.

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FIG.1

