DESCRIPTION

TITLE OF INVENTION: PROCESSED RICE HUSK ASH AND METHOD FOR PRODUCING SAME

TECHNICAL FIELD

[0001]

The present invention relates to a processed rice husk ash and a method for producing the same.

CROSS REFERENCE TO RELATED APPLICATIONS

This application claims priority to Japan Patent Application No. 2023-107013, filed on June 29, 2023, the entire contents of which are incorporated herein by reference.

BACKGROUND ART

[0002]

In recent years, realization of a society capable of sustainable regeneration is required for protecting the global environment. As one specific measure to achieve this goal, biomass technology, such as biomass boiler and biomass power generation, which does not use fossil fuels, has been spreading in various regions. In facilities utilizing such technology, a large amount of rice husk is used as fuel. However, the rice husk contains a large amount of non-combustible components (particularly silica), and these facilities face the problem of disposing of combustion ash (rice husk ash) that contains a large amount of uncombusted components.

[0003]

Hence, an attempt has already been made as one method to solve this problem, for example, by dissolving rice husk ash, which contains a large amount of silica components, in an alkaline aqueous solution (an aqueous solution containing alkali metal salts, such as a sodium hydroxide aqueous solution), producing a silicate aqueous solution containing alkali metal components (hereinafter referred to as an “alkaline silicate aqueous solution”), and reusing it as a raw material for wet process silica (e.g., Patent Literature 1 and Patent Literature 2).

[0004]

Alkaline silicate aqueous solutions are widely used not only as raw materials for wet process silica but also in fields relating to raw materials for synthetic zeolites, soil hardeners for civil engineering and construction, cleaning agents, textile and pulp and the like.

[0005]

Patent Literature 1: JP patent publication 2007-510613 (WO 2005/044727)
Patent Literature 2: JP patent publication 2007-522069 (WO 2005/077828)

The entire descriptions in Patent Literatures 1 and 2 are incorporated herein by reference.

SUMMARY OF INVENTION

TECHNICAL PROBLEM

[0006]

However, the inventors discovered that when producing industrial-grade alkaline silicate aqueous solutions based on conventional technology with rice husk ash discharged from biomass boilers and the like, the following new problems arise.

[0007]

(1) When the rice husk ash is fed directly into an alkaline aqueous solution, the rice husk ash absorbs the solvent during dissolution, causing an increase in viscosity and making slurry preparation difficult. On the other hand, when the rice husk ash is fed into an alkaline aqueous solution together with water for slurry preparation, only alkaline silicate aqueous solutions with low concentrations can be produced.

[0008]

(2) When the rice husk ash is fed into an alkaline aqueous solution, the rice husk ash floats on the aqueous solution, resulting in poor dissolution efficiency, particularly poor initial sinking property into the alkaline aqueous solution.

[0009]

(3) The rice husk ash has a low apparent density and is bulky relative to its weight. Therefore, the weight of rice husk ash that can be transported at one time is small, meaning poor transport efficiency. In particular, the longer the distance from a boiler facility to a dissolution facility, the higher the cost of transportation. Furthermore, since the rice husk ash is bulky, its storage efficiency is poor, and the cost of storage also increases.

[0010]

The present invention was made in view of the above problems, and an object of the present invention is to provide a rice husk ash more suitable for producing alkaline silicate aqueous solutions than the conventional rice husk ash, as well as a method for producing the same.

SOLUTION TO PROBLEM

[0011]

To solve the above problems, the present inventors intensively studied. As a result, the present inventors found that the above problems can be solved by increasing the apparent density of the rice husk ash by subjecting the rice husk ash to a processing of consolidation or pulverization. The present invention is shown in <1>, and preferably shown in <2> and thereafter.

<1>

A rice husk ash having an apparent density in the dry state of 400 to 800 g/L and a water content of 30 mass% or less.

<2>

The rice husk ash according to <1>, having a carbon content of 0.1 to 8.0 mass% and a silica content of 85 to 95 mass%.

<3>

The rice husk ash according to <1> or <2>, having a volume average particle size D50 measured by laser diffraction method of 5.0 to 200 μm.

<4>

A method for producing rice husk ash, comprising:

consolidating or pulverizing a raw rice husk ash having an apparent density in the dry state of 350 g/L or less so that the apparent density in the dry state becomes 400 to 800 g/L; and

optionally, adjusting a water content of the raw rice husk ash within the range of 30 mass% or less.

<5>

The method according to <4>, wherein the consolidation or pulverization is performed to achieve a volume average particle size D50 of 5.0 to 200 μm.

<6>

The method according to <4> or <5>, wherein the consolidation or pulverization is performed using a roller compactor at a linear pressure of 1.2×10^3 N/cm or more.

ADVANTAGEOUS EFFECT OF INVENTION

[0012]

The present invention allows to provide a rice husk ash more suitable for producing alkaline silicate aqueous solutions than the conventional rice husk ash, as well as a method for producing the same. The alkaline silicate aqueous solutions prepared from the rice husk ash of the present invention can be used as a raw material for wet process silica, as well as in the same applications as for the conventional sodium silicate aqueous solution derived from silica sand.

BRIEF DESCRIPTION OF DRAWINGS

[0013]

[Figure 1] Figure 1 shows photographs of raw rice husk ashes used in EXAMPLES and a photograph of a processed rice husk ash of one example of the present invention.

[Figure 2] Figure 2 shows a photograph of the liquid surface after feeding an unprocessed rice husk ash into a sodium hydroxide aqueous solution and passing some time after the sinking has finished.

[Figure 3] Figure 3 shows graphs showing the relationships between the duration and

SiO_2 concentrations after feeding rice husk ashes into sodium hydroxide aqueous solutions.

EMBODIMENTS OF INVENTION

[0014]

<Rice husk ash>

The rice husk ash of the present invention is a rice husk ash having an apparent density in the dry state of 400 to 800 g/L and a water content of 30 mass% or less.

[0015]

Typically, the apparent density of unprocessed rice husk ash after combustion is 350 g/L or less, and often 300 g/L or less, thus the rice husk ash of the present invention has a higher apparent density than unprocessed rice husk ash. The rice husk ash of the present invention has an adequately high apparent density and an adequate weight, thereby improving its sinking property into alkaline aqueous solution and further reducing solvent absorption. Compared to the unprocessed rice husk ash, a larger amount of rice husk ash can be fed into a vessel, allowing to obtain an increased SiO_2 concentration. Furthermore, when the water content is 30 mass% or less, the rice husk ash of the present invention maintains its weight within a range suitable for transportation and an appropriate solid state. In addition, the rice husk ash of the present invention has an adequately high apparent density, thereby reducing its bulkiness and improving transportation and storage efficiencies. As a result, the rice husk ash of the present invention improves transportation efficiency, storage efficiency, and sinking property into alkaline aqueous solution, enabling a preparation of slurry with higher concentration. Thus, the rice husk ash of the present invention is more suitable for the production of alkaline silicate aqueous solutions than the unprocessed rice husk ash after combustion.

[0016]

The apparent density of the rice husk ash is 400 to 800 g/L. The apparent density of 400 g/L or more allows to adequately reduce its bulkiness relative to weight, improving transportation efficiency, storage efficiency, and sinking property into alkaline aqueous solution, resulting in a slurry with high concentration. The lower limit of the range of the apparent density is preferably 450 g/L or more, more preferably 500 g/L or more, and even more preferably 550 g/L or more, from the viewpoint of improving the transportation efficiency, storage efficiency, and the sinking property into alkaline aqueous solution, and enabling the preparation of slurry with higher concentration more easily. The apparent density of 800 g/L or less allows for an adequate space between rice husk ash particles, improving its sinking property into alkaline aqueous solution. The upper limit of the range of the apparent density is preferably 750 g/L or less, more preferably 730 g/L or less, and even more preferably 720 g/L or less, from the viewpoint of further improving the sinking property into

alkaline aqueous solution.

[0017]

Apparent density is measured in the dry state (approximately less than 1.5 mass% of water content). The unprocessed rice husk ash after combustion is usually in the dry state and its apparent density can be measured as it is. The apparent density of the rice husk ash not in the dry state can be measured after sufficient drying treatment (e.g. 125°C, 6 hours).

[0018]

The water content of the rice husk ash of the present invention is 30 mass% or less. The water content of 30 mass% or less allows the rice husk ash of the present invention to maintain its weight within a range suitable for transportation and an appropriate solid state. The water content may be 25 mass% or less, 20 mass% or less, 15 mass% or less, or 10 mass% or less. When the rice husk ash of the present invention contains a certain water content, scattering of dust can be suppressed, and handling property during packaging, transportation, and unpacking, and further handling property when the rice husk ash is fed into aqueous solution are more improved. In addition, when the rice husk ash of the present invention has a certain water content, the sinking property of the rice husk ash into alkaline aqueous solution is further improved due to an appropriate increase in weight. The water content may be 0.1 mass% or more, 0.5 mass% or more, 0.7 mass% or more, or 1.0 mass% or more. The water content can be adjusted by any process, such as adding water to rice husk ash and drying. The water content of the rice husk ash of the present invention may not be adjusted at all, i.e. no water may be added.

[0019]

The silica and carbon contents of the rice husk ash of the present invention are not particularly limited, but since the rice husk ash is used as a silica source, the silica content is preferably high, and the content of carbon as an impurity is preferably low. The silica content of the rice husk ash may be 85 mass% or more, and is preferably 90 mass% or more. The upper limit of the silica content varies depending on the type of rice, its growing area, and the combustion method of rice husk (boiler type, combustion conditions, and the like), but typically it is approximately 95 mass% or less. The silica content of rice husk ash can be measured, for example, with a fluorescent X-ray analyzer or an ICP emission analyzer. On the other hand, the carbon content of the rice husk ash of the present invention is preferably 0.1 to 8.0 mass%. The carbon content of the rice husk ash discharged from general combustion facilities is approximately 0.1 mass% or more. The carbon content of the rice husk ash may be 0.3 mass% or more, 0.5 mass% or more, or 1.0 mass% or more. The carbon content of 8.0 mass % or less of the rice husk ash allows to reduce an incomplete combustion component of the rice husk, improving the quality of the rice husk ash (for example, the content of the silica component). The carbon content of the rice husk ash is

preferably 6.0 mass% or less, 5.0 mass% or less, or 4.0 mass% or less. The carbon content of rice husk ash can be measured, for example, with a carbon analyzer.

[0020]

The rice husk ash of the present invention preferably has a volume average particle size D50 as determined by laser diffraction method of the range of 5.0 to 200 μm. When the volume average particle size D50 is 5.0 to 200 μm, the apparent density is more easily maintained within 400 to 800 g/L. Furthermore, when the volume average particle size D50 is 5.0 μm or more, excessive dust generation is suppressed, and handling property are further improved. When the volume average particle size D50 is 5.0 to 200 μm, the volume of void space between the particles is within an appropriate range, and when the rice husk ash is fed into alkaline aqueous solution, the rice husk ash absorbs the solution appropriately. As a result, in combination with the adequate weight, the time required for rice husk ash to sink into alkaline aqueous solution is reduced, further improving its sinking property. Since a proportion of fine particles of 1.0 μm or less in the particle size distribution is preferably as small as possible, the volume average particle size D50 is preferably 10 to 150 μm, more preferably 15 to 100 μm, and particularly preferably 20 to 80 μm.

[0021]

The particle size distribution measurement can be performed by a commercially available laser diffraction type particle size distribution measurement device. Specifically, for particle size distribution measurement, the sample is dispersed in water to achieve a predetermined concentration, the dispersion is fed into the circulation system within the measurement device, and the value when the D50 value stabilizes is adopted as the measured value. When the rice husk ash is processed into a consolidated molded body as described later, it is preferable to adjust the consolidation conditions so that the volume average particle size D50 measured by the above method falls within the range of 5.0 to 200 μm.

[0022]

The form of the rice husk ash of the present invention may be either a powder or a molded body, and the shape of the molded body is not particularly limited. The rice husk ash of the present invention has been processed by consolidation or pulverization, as described below, and the distinction from the unprocessed rice husk ash after combustion is easily made based on visual appearance, microscopic appearance, apparent density, and the shape and particle size distribution of the rice husk ash particles. For example, regarding appearance, the particles of the rice husk ash of the present invention have a relatively uniform particle size compared to unprocessed rice husk ash, and there are almost no particles exceeding 1.0 mm. The apparent density of the rice husk ash of the present invention is 400 g/L or more, while the unprocessed rice husk ash is 350 g/L or less.

[0023]

The rice husk ash of the present invention can contribute to an improvement in transportation efficiency both in bulk transportation and in packaging transportation. The processed rice husk ash of the present invention has a high apparent density, enabling for example to load a larger amount of rice husk ash per single transportation compared to the conventional rice husk ash. Additionally, the rice husk ash of the present invention can shrink storage space per storage amount when the rice husk ash is stored in a packaging bag (a paper bag, a polyethylene bag, a flexible container bag, or the like). Moreover, the rice husk ash of the present invention has an advantage that the generation of dust can be suppressed when the rice husk ash is packaged in a packaging bag or when the rice husk ash is unpacked.

[0024]

The rice husk ash of the present invention can be used as a silica source. For example, the rice husk ash of the present invention can be used as a raw material for alkaline silicate aqueous solutions, either alone or in mixture with silica sand. The alkaline silicate aqueous solution can be produced by a known method, for example, by adding the rice husk ash to a sodium hydroxide aqueous solution and dissolving it. The dissolution conditions are not particularly limited, and known dissolution methods can be used. Under normal pressure, the dissolution can be performed at a temperature of 70°C or more, and in a pressurized vessel such as an autoclave, the dissolution can be performed at a temperature of 100°C or more. Generally, the silica component of the rice husk ash that dissolves in alkaline aqueous solution is approximately 70 to 90 mass% thereof. Therefore, the amount of the rice husk ash to be fed into alkaline aqueous solution must be larger than the silica amount required to achieve a target concentration. The rice husk ash of the present invention has a high apparent density, allowing a larger amount of rice husk ash to be loaded into a vessel compared to the conventional rice husk ash. Furthermore, the rice husk ash of the present invention has an adequately high apparent density, resulting in low solvent absorption and eliminating the need to add water for making slurry. Therefore, there are advantages of producing a larger amount of alkaline silicate aqueous solution at once or producing a high-concentration alkaline silicate aqueous solution. For example, once a pressurized vessel, such as an autoclave, is sealed, it is difficult to add more raw materials. In this way, the rice husk ash of the present invention can be charged in a larger amount compared to the conventional rice husk ash, and is more effective when a large amount of silica source needs to be charged at once.

[0025]

In addition, the rice husk ash of the present invention has a higher apparent density and an adequate weight compared to the conventional rice husk ash, thereby improving the sinking property into aqueous alkaline solution. The use of the rice husk ash of the present invention allows to reduce industrial process time, to cut down on energy consumption, and to improve manufacturing efficiency.

[0026]

The alkaline silicate aqueous solution produced with a raw material of the rice husk ash of the present invention can be used as a raw material for wet process silica, either alone or in a mixture with the conventional alkaline silicate aqueous solution (e.g., one produced with a raw material of silica sand). Wet process silica has a high BET specific surface area and contains micropores to mesopores. Therefore, it is widely used in various applications, including a reinforcing filler for industrial rubbers, tires, silicone rubbers and the like; a filler for paper; a matting agent for paints; a polishing agent for toothpastes and the like; and an anti-blocking agent for films.

[0027]

<Method for producing rice husk ash>

The method for producing rice husk ash of the present invention comprises consolidating or pulverizing a raw rice husk ash having an apparent density in the dry state of 350 g/L or less so that the apparent density in the dry state becomes 400 to 800 g/L, and optionally adjusting a water content of the raw rice husk ash to 30 mass% or less.

[0028]

The raw rice husk ash is not particularly limited as long as it is an ash discharged by the combustion of rice husk. However, the raw rice husk ash is preferably a combustion ash of rice husk discharged from combustion facilities using biomass fuel, such as biomass boilers or biomass power generation facilities designed for the purpose of heat recovery or power generation, from the perspective of suppressing environmental impact such as CO_2 emissions. The raw rice husk ash has an apparent density in the dry state of 350 g/L or less. In the producing method of the present invention, the raw rice husk ash is processed by consolidating or pulverizing to adjust the apparent density to an appropriate range. The lower limit of the apparent density of the raw rice husk ash is not particularly limited, but is preferably 150 g/L or more, 200 g/L or more, 250 g/L or more, or 300 g/L or more from the viewpoint of processing efficiency. The equipment for combusting rice husk is not particularly limited and may be, for example, a boiler or combustion furnace commonly used.

[0029]

The silica and carbon contents of the raw rice husk ash are not particularly limited; however, since these contents are reflected in the silica and carbon contents of the rice husk ash of the present invention, the silica content is preferably high, and the carbon content is preferably low. The low carbon content also contributes to improved energy utilization efficiency of biofuels in combustion facilities. The silica content of the raw rice husk ash may be 85 mass% or more, and is preferably 90 mass% or more. The upper limit of the silica content varies depending on the type of rice, its growing area, and the combustion method of rice husk (boiler type, combustion conditions, and the like), but typically it is approximately 95 mass% or less. On the

other hand, the carbon content of the raw rice husk ash is preferably 0.1 to 8.0 mass%. The carbon content of the rice husk ash discharged from general combustion facilities is approximately 0.1 mass% or more. It is possible to reduce the carbon content of the rice husk ash to less than 0.1 mass% by high temperature combustion, but in such a case, crystalline components of silica, such as cristobalite, tends to be by-produced in the rice husk ash, which may be undesirable when the rice husk ash is used as a silica source. For example, when the rice husk ash is used as a silica source to produce an alkaline silicate aqueous solution, the crystalline components of silica cannot dissolve in the solvent and remain as foreign substances. The carbon content of the raw rice husk ash may be 0.3 mass% or more, 0.5 mass% or more, or 1.0 mass% or more. The carbon content of 8.0 mass % or less in the raw rice husk ash allows to reduce the incomplete combustion component of the raw rice husk, improving the quality of the raw rice husk ash (for example, the content of the silica component). The carbon content of the raw rice husk ash is preferably 6.0 mass% or less, 5.0 mass% or less, or 4.0 mass% or less.

[0030]

In the producing method of the present invention, the raw rice husk ash is subjected to a processing of consolidation or pulverization to increase the apparent density in the dry state to a desired range. Consolidation refers to a process of compacting the raw rice husk ash, and pulverization refers to a process of crushing the raw rice husk ash. The processing may be either one of consolidation and pulverization, and both processes may be performed simultaneously or sequentially. Depending on the type and conditions of the processing, it may be indistinguishable between consolidation and pulverization, and there is no need to distinguish between the two.

[0031]

The type of consolidation and pulverization is not particularly limited. The pulverizing process may be dry or wet. In the case of the dry processing, although dust is easily generated, there is no need for pre-processing steps, resulting in efficient processing. In the case of the wet processing, although the pre-processing, such as a slurry preparation, is required, the dust generation can be suppressed. The dry pulverization can be performed using commercially available dry pulverizers such as a roller mill, high-speed rotary mill (pin mill), jet mill, ball mill, or bead mill. The wet pulverization can be performed using commercially available wet pulverizers such as an inline mixer, jet mill, ball mill, or bead mill. The pulverization can break down plant-derived structures in the rice husk ash and reduce the void space between particles, thereby increasing the apparent density of the rice husk ash. The consolidation can be performed using commercially available granulator, such as a roller compactor with compression rollers or briquetting rollers, or a tablet press. The consolidation can compress the rice husk ash and reduce the void space between particles, thereby increasing the apparent density of the rice husk ash while

suppressing the dust generation. When both consolidation and pulverization are performed, the rice husk ash is compacted while the plant-derived structural components in the rice husk ash are broken down, thereby suppressing the dust generation and readily achieving a higher apparent density.

[0032]

The consolidation is preferably performed using a roller compactor, and more preferably at a linear pressure of 1.2×10^3 N/cm or more. The linear pressure is a value obtained by dividing the total pressure (N) applied to the roll of the roller compactor by the rotational axial width (cm) of the roll. The linear pressure of 1.2×10^3 N/cm or more allows to readily achieve an adequate apparent density. The linear pressure during consolidation is preferably 2.9×10^3 N/cm or more, or 4.7×10^3 N/cm or more. Furthermore, the linear pressure during consolidation is preferably 12.8×10^3 N/cm or less, and more preferably 11.7×10^3 N/cm or less.

[0033]

In the producing method of the present invention, a water content of the raw rice husk ash may be adjusted to within the range of 30 mass% or less, as necessary. The water content adjusted within the range allows the rice husk ash of the present invention, as the final product, to maintain its weight within a range suitable for transportation and an appropriate solid state. Usually, the water content of raw rice husk ash after combustion is about 1.0 mass%. The water content may be 5.0 mass% or more, 10 mass% or more, and 25 mass% or less, or 20 mass% or less. The water content can be adjusted by drying or by adding water. The timing of adjusting the water content may be before or after the consolidation or pulverization. When the addition of water is performed prior to the consolidation or pulverization, it also contributes to suppressing the dust generation during these processing procedures. When the addition of water is performed prior to the consolidation or pulverization, it is preferable to adjust the water content to a level where the raw rice husk ash does not solidify in equipments due to the influence of the water. When the addition of water is performed after the consolidation or pulverization, it also contributes to suppressing the dust generation during the transportation and packaging of the processed rice husk ash. When the addition of water is performed after the consolidation or pulverization, it is preferable to mix the water and rice husk ash so as to be uniform. If the water content of the rice husk ash of the present invention as the final product is 30 mass% or less, the water content may be adjusted multiple times.

EXAMPLES

[0034]

Hereinafter, the present invention will be described in more detail with reference to examples. The materials, usage amounts, ratios, processing details, processing steps, etc., shown in the examples can be changed as appropriate without departing from the spirit of the present invention. Therefore, the scope of the present invention is not limited to the specific examples shown below.

[0035]

<Raw rice husk ash>

In the examples, the following raw rice husk ashes A to C were used. The basic physical properties of the rice husk ashes are shown in Table 1.

- Raw rice husk ash A: Ash discharged from a fluidized-bed type biomass boiler.
- Raw rice husk ash B: Ash discharged from a fixed-bed type biomass boiler.
- Raw rice husk ash C: Ash produced by combusting rice husk using a small muffle furnace (Model: KDF S100G, manufactured by DENKEN Corporation). Specifically, 500 g of rice husk was fed into each of two stainless steel boxes (300 × 300 × 115 mm) with lids and air holes. These were stacked in two layers and fired at 500°C for 2 hours, followed by 700°C for 2 hours.

[0036]

[Table 1]

		Raw rice husk ash		
		A	B	C
Water content	[mass%]	0.7	1.2	1.0
Apparent density	[g/L]	300	180	151
Bulk density	[g/mL]	0.380	0.210	0.180
Average particle size (D50)	[μm]	220	277	338
Mercury pore volume	[cm^3/g]	1.76	3.09	2.91
Ash composition [mass%]	Carbon content	3.3	3.5	0.2
	SiO_2	90.1	92.1	93.4
	K_2O	1.2	2.5	3.5
	CaO	0.4	0.5	0.7
	Al_2O_3	0.2	0.0	0.0
	P_2O_5	0.7	0.6	0.6
	Other	4.1	0.8	1.6

[0037]

<Analytical method of rice husk ash>

• Water content (loss on heating) (mass%)

The water content was determined based on the mass loss of the sample after drying at 105°C for 2 hours, according to JIS K 5101-15-1 (Test methods for pigments - Loss on Heating).

[0038]

• Apparent density (g/L)

A dedicated measuring device (0.5 mm mesh sieve, funnel, 30 mL cylinder receiver, receiver stand, and funnel stand) was used based on JIS K 5101-12-1:2004 (Test methods for pigments—Apparent density or apparent specific volume—Loose packing method). The sample (rice husk ash) was dropped through the sieve into the funnel using a brush. Then, the top portion of the sample piled up in the receiver was leveled off using a spatula, followed by measuring the sample's mass. An apparent density of rice husk ash in a unit of g/L was calculated based on its mass by the following equation.

[0039]

[Numerical Formula 1]

$$Apparent\ density\ (g/L) = \frac{Mass\ of\ sample\ in\ cylinder\ (g)}{30\ (mL)} \times 1000$$

[0040]

• Bulk Density (g/mL)

A dedicated measuring device (consisting of a cylinder made of general steel with an inner diameter of 22.00 ± 0.05 mm and internal depth of 100 mm, and a piston with an outer diameter of 21.80 ± 0.05 mm, length of 115 mm, mass of 190 g and an internal cavity) based on the section "7.8 Bulk Density" of JIS K 6220-1:2015 (Rubber compounding ingredients-Organic chemicals-Test methods-Part 1: General). The piston was naturally dropped into the cylinder before adding the sample (rice husk ash), and the height of the piston protruding from the top of the cylinder was measured. Next, the weighed sample of about 1 g was fed into the cylinder, the piston was slowly lowered for 5 seconds, the cylinder sidewall was lightly tapped with a wooden piece to ensure the piston was well seated, and the height of the piston protruding from the top of the cylinder was measured. A bulk density in a unit of g/mL was calculated by the following formula with the change in piston height protruding from the top of the cylinder and the bottom area within the cylinder.

[0041]

[Numerical Formula 2]

$$Bulk\ density\ (g/mL) = \frac{Mass\ of\ sample\ in\ cylinder\ (g)}{Change\ in\ piston\ height\ (cm) \times Bottom\ area\ within\ cylinder\ (cm^2)}$$

[0042]

• Volume average particle size (D50)

A laser diffraction particle size measuring apparatus (Model: SYNC30, manufactured by MicrotracBEL Corporation) was used to measure the particle size

distribution, and the value of the particle size at which the cumulative volume percentage reaches 50% in the particle size distribution was determined as D50.

[0043]

• Pore volume (cm^3/g) measured by mercury intrusion technique (hereinafter referred to as “mercury pore volume”)

A mercury porosimeter (Model: PASCAL440, manufactured by ThermoQuest Corporation) was used to measure the mercury pore volume in the range of pore radial 3.6 to 6,400 nm with an increased pressure from 100 kPa to 200 MPa.

[0044]

• Carbon content of rice husk ash (mass%)

The rice husk ash was dried at 105°C for 2 hours as a pre-processing, and then the carbon content was quantified using the following method.

A carbon analyzer (Model: CS744, manufactured by LECO Japan LLC) according to the method of combustion in oxygen stream coupled with non-dispersive infrared absorption was used to measure the corbon content by subjecting the sample to a heat treatment under the conditions of 1,350°C, an oxygen inlet pressure of 0.24 MPa, and a measurement time of 50 seconds, and quantifying the CO gas and CO_2 gas using the infrared detector (NDIR) within the analyzer.

[0045]

• Composition of inorganic components other than carbon in rice husk ash (mass%)

A wavelength-dispersive X-ray fluorescence analyzer (Model: ZSX PrimusII, manufactured by Rigaku Corporation) was used to analyze qualitatively for elements other than carbon and confirm the types of detected inorganic impurities. Subsequently, the detected inorganic impurities were analyzed quantitatively. The measurement sample was prepared by placing rice husk ash in a ring-shaped mold and performing pressure molding. The concentration (mass%) of the quantified inorganic component composition (excluding carbon) was calculated by converting it to oxide equivalents using the analysis software provided with the analyzer.

[0046]

<Comparative Example 1 (unprocessed)

Raw rice husk ash A was not subjected to the processing of consolidation and pulverization, and subjected as it is to dissolution test (1) described later, and then evaluated for a suppression of the dust generation when the rice husk ash is fed into an alkaline aqueous solution, a sinking of rice husk ash, and a viscosity after the feeding. For the physical property of the resulting alkaline silicate aqueous solution, the SiO_2 concentration and the molar ratio of $SiO_2/(Na_2O+K_2O)$ were determined.

[0047]

Example 1 (consolidation)

Raw rice husk ash A was consolidated with a roller compactor (Model: FR125×40, manufactured by Freund-Turbo Corporation) under the conditions of a

roller rotation speed of 9 rpm, linear pressure of 5.8×10^3 N/cm, and screw rotation speed of 90 rpm, thereby obtaining a processed rice husk ash.

[0048]

After the processing, the measurement of physical property of the rice husk ash and Dissolution Test (1) were performed in the same manner as in Comparative Example 1.

[0049]

Example 2 (Dry pulverization)

Raw rice husk ash A was pulverized with a pin-type high-speed pulverizer (Model: Free Pulverizer M-2, manufactured by Nara Machinery Corporation) under the conditions of a feed rate of 30 kg/h and a rotational speed of 7,600 rpm, thereby obtaining a processed rice husk ash.

After the processing, the measurement of physical property of the rice husk ash and Dissolution Test (1) were performed in the same manner as in Comparative Example 1.

[0050]

Example 3 (Wet pulverization)

500 g of the raw rice husk ash A was mixed with 1,500 g of water to prepare a rice husk ash slurry with a concentration of 25 mass%. The whole amount of this slurry was fed into a ceramic pot (capacity 3.6 L) containing 4,200 g of alumina balls (diameter 10 mm, media density 4.09 g/cm^3, about 1 L) and sealed. The ceramic pot was rotated at 700 rpm for 3 hours using a pot mill rotary table (Model: ANZ-100S, manufactured by Nitto Kagaku Corporation) to perform the wet pulverization of the rice husk ash slurry.

[0051]

After the wet pulverization, the slurry was separated from the alumina balls using a sieve with a mesh size of 1 mm. A suction filter was constructed by attaching a magnetic Büchner funnel (Nutsche) with a diameter of 150 mm to a 3-liter suction filtration bottle and placing a polyester plain weave filtration cloth (mesh size: approximately 50 μm × 400 μm) in the funnel. This suction filter was then used to perform an additional solid-liquid separation processing on the slurry. The solid fraction after the solid-liquid separation was subjected to a static drying at 125°C for 6 hours with a constant-temperature drying oven (Model: NDO-420, manufactured by Tokyo Rikakikai Corporation), thereby obtaining a processed rice husk ash.

After the processing, the measurement of physical property of the rice husk ash and Dissolution Test (1) were performed in the same manner as in Comparative Example 1.

[0052]

Comparative Example 2 (Excessive pulverization)

Raw rice husk ash A was pulverized with a supersonic jet pulverizer (Model:

PJM-100NP, manufactured by Nippon Pneumatic Kogyo Corporation) under the conditions of a feed quantity of 2.7kg/h, pulverization pressure of 0.55 MPa, four nozzles, and nozzle diameter of 3 mm, thereby obtaining a processed rice husk ash.

After the processing, the measurement of physical property of the rice husk ash and Dissolution Test (1) were performed in the same manner as in Comparative Example 1.

[0053]

Example 4 (pulverization and consolidation)

The rice husk ash pulverized in the same manner as in Comparative Example 2 was consolidated with a roller compactor (Model: FR125×40, manufactured by Freund-Turbo Corporation) under the condition of a roller rotation speed 9 rpm, linear pressure of 5.8×10^3 N/cm, and screw rotation speed 90 rpm, thereby obtaining a processed rice husk ash.

After the processing, the measurement of physical property of the rice husk ash and Dissolution Test (1) were performed in the same manner as in Comparative Example 1.

[0054]

Comparative Example 3 (unprocessed)

Raw rice husk ash B was not subjected to the processing of consolidation and pulverization, and subjected as it is to the measurement of the physical property of the rice husk ash and Dissolution Test (1) in the same manner as in Comparative Example 1.

[0055]

Comparative Example 4 (unprocessed)

Raw rice husk ash C was not subjected to the processing of consolidation and pulverization, and subjected as it is to the measurement of the physical property of the rice husk ash and Dissolution Test (1) in the same manner as in Comparative Example 1.

[0056]

Comparative Example 5 (wet pulverization)

Raw rice husk ash B was processed in the same manner as in Example 3 (wet pulverization and solid-liquid separation) to obtain a processed rice husk ash.

After the processing, the measurement of physical property of the rice husk ash and Dissolution Test (1) were performed in the same manner as in Comparative Example 1.

[0057]

Comparative Example 6 (wet pulverization)

Raw rice husk ash C was processed in the same manner as in Example 3 (wet pulverization and solid-liquid separation) to obtain a processed rice husk ash.

After the processing, the measurement of physical property of the rice husk ash and Dissolution Test (1) were performed in the same manner as in Comparative Example 1.

[0058]

<Dissolution Test (1)>

The rice husk ash was dissolved in an alkaline aqueous solution according to the procedure in Dissolution Method (1) below, to obtain an alkaline silicate aqueous solution. The alkaline silicate aqueous solution was evaluated for a dust suppression when the rice husk ash was fed into the alkaline aqueous solution, a sinking of the rice husk ash, and a viscosity after the feeding. The SiO_2 concentration and the $SiO_2/(Na_2O+K_2O)$ molar ratio were determined as the physical property of the resulting alkaline silicate aqueous solution.

[0059]

• Dissolution Method (1)

Dissolution Method (1) aimed to prepare an alkaline silicate aqueous solution having a SiO_2 concentration of 17 to 19 mass% and the above molar ratio of 3.00 to 3.40. These values are standard for commercially available alkaline silicate aqueous solutions. 1,310 g of sodium hydroxide aqueous solution that was adjusted to 8.9 mass% was added to a 3L stainless steel vessel equipped with a stirring device and heated to 90°C. Then, 410 g of rice husk ash was added to the vessel while continuously stirring, the vessel was closed with a lid, and the mixing was continued for 6 hours while maintaining 90°C. The aqueous solution was then cooled to room temperature. A suction filter was prepared by attaching a magnetic Buchner funnel (Nutsche) with a diameter of 150 mm to a suction filtration bottle with a volume of 3 L, and placing a filter paper (mesh size 1 μm, Standard 5C, manufactured by Advantec Corporation) in the funnel. The suction filter was used to remove undissolved residues from the aqueous solution, thereby obtaining an alkaline silicate aqueous solution.

[0060]

• Suppression of dust during feeding

In Dissolution Method (1) described above, the amount of dust generated when the rice husk ash was fed into the alkaline aqueous solution was visually evaluated. The smaller the generation of dust, the lower the deterioration of the work environment can be suppressed. The evaluation was conducted on a five-graded scale, from 5 for the state observed in Example 1 where the dust generation was minimal, to 1 for the state observed in Comparative Example 3 where the dust generation was most significant. An evaluation of 4 or higher indicates the dust generation within acceptable levels, and the results are labeled “GOOD”; results of 3 or lower are labeled “BAD”.

[0061]

• Sinking speed

In Dissolution Method (1) described above, the time (seconds) from the immediate moment of feeding the rice husk ash to when all the rice husk ash at the liquid surface (excluding rice husk ash that scattered as dust) became completely wet and sank, was measured. The shorter the sinking time, the more the manufacturing

process time including a step for dissolving rice husk ash can be reduced, contributing to improved productivity. In the results, cases where the sinking time was 60 seconds or less were labeled “GOOD”, while cases exceeding 60 seconds were labeled “BAD”.
[0062]
• Viscosity

In Dissolution Method (1) described above, immediately after all the rice husk ash on the liquid surface (excluding rice husk ash that scattered as dust) became wet and sank, a portion (200 mL) of the aqueous solution was transferred to a beaker. The viscosity of samples was measured with a commercially available Type B viscometer (Model: TVB-10M, manufactured by Toki Sangyo Corporation). For samples with a viscosity of 50 mPa·s or less, the No. 20 rotor was used to measure the viscosity after rotating at 60 rpm for 1 minute, while for other samples, the No. 21 rotor was used to measure the viscosity after rotating at 3 rpm for 1 minute. In the results, cases where the viscosity was 50 mPa·s or less are labeled “GOOD”, while cases exceeding 50 mPa·s are labeled “BAD”.
[0063]
• SiO_2 concentration and molar ratio in alkaline silicate aqueous solution

The alkaline silicate aqueous solution sample obtained by Dissolution Method (1) was collected (about 0.2 mL) with a syringe equipped with a membrane filter having a mesh size of 1 μm. The collected sample was diluted 10,000-fold with pure water (standard solution for calibration curve: 20 ppm by mass). Quantitative analysis of SiO_2, Na_2O, and K_2O was performed on the diluted samples with a high-resolution ICP emission spectrometer (Model: PS3520DDII, manufactured by Hitachi High-Tech Science Corporation). The measured mass concentrations of SiO_2, Na_2O, and K_2O were converted to molar quantities, and the molar ratio of silicon dioxide relative to the total of sodium oxide and potassium oxide was determined. Specifically, the molar masses of each component were set to 60.08 (SiO_2), 61.98 (Na_2O), and 94.19 (K_2O), and the molar ratio was calculated by the following equation.
[0064]
[Numerical Formula 3]

$$SiO_2/(Na_2O + K_2O)\ molar\ ratio = \frac{SiO_2\ (mass\%)/60.08}{Na_2O\ (mass\%)/61.98 + K_2O\ (mass\%)/94.19}$$

[0065]

Table 2 shows the characteristics of rice husk ash of Examples 1 to 4 and Comparative Examples 1 to 2, along with the results of Dissolution Test (1).
[0066]

[Table 2]

		Comp. Example 1	Example 1	Example 2	Example 3	Comp. Example 2
Raw rice husk ash		A	A	A	A	A
Processing method		Non	Consolidation	pulverization	pulverization	excess pulverization
Physical property of Rice husk ash	Water content [mass%]	0.7	0.7	0.8	0.3	0.5
	Apparent density [g/L]	300	590	450	600	380
	Bulk density [g/mL]	0.38	0.65	0.60	0.75	0.45
	Particle size (D50) [μm]	220	40	46	13	34
	Mercury pore volume [cm^3/g]	1.76	1.05	1.24	0.96	1.32
Evaluation results in Dissolution Test (1)	Dissolution method	(1)	(1)	(1)	(1)	(1)
	Dust suppression	3 BAD	5 GOOD	4 GOOD	4 GOOD	2 BAD
	Sinking property [Seconds]	180 BAD	10 GOOD	15 GOOD	40 GOOD	unmeasurable BAD
	Viscosity [mPa·s]	10,000< BAD	3.0 GOOD	10.8 GOOD	11.8 GOOD	13.4 GOOD
	SiO_2 concentration [mass%]	17.2	18.0	18.2	18.3	18.1
	Molar ratio	3.04	3.22	3.28	3.27	3.24

(Continuation of Table 2)

	Example 4	Comp. Example 3	Com. Example 4	Com. Example 5	Com. Example 6

Raw rice husk ash		A	B	C	B	C
Processing method		Pulverization and Consolidation	Non	Non	pulverization	pulverization
Physical property of Rice husk ash	Water content [mass%]	0.6	1.2	1.0	0.8	1.2
	Apparent density [g/L]	710	180	151	340	283
	Bulk density [g/mL]	0.82	0.21	0.18	0.43	0.35
	Particle size (D50) [μm]	36	277	338	21	62
	Mercury pore volume [cm^3/g]	0.94	3.09	2.91	2.15	1.82
Evaluation results in Dissolution Test (1)	Dissolution method	(1)	(1)	(1)	(1)	(1)
	Dust suppression	4 GOOD	1 BAD	1 BAD	3 BAD	2 BAD
	Sinking property [Seconds]	44 GOOD	unmeasurable BAD	unmeasurable BAD	79 BAD	135 BAD
	Viscosity [mPa·s]	11.5 GOOD	unmeasurable BAD	unmeasurable BAD	63.2 BAD	10,000< BAD
	SiO_2 concentration [mass%]	17.9	17.1	17.5	17.5	18.1
	Molar ratio	3.22	3.03	3.05	3.06	3.22

[0067]

<Explanation of results: Examples 1 to 4, Comparative Examples 1 to 6>

・Characteristics of rice husk ash

Figure 1 shows photographs of raw rice husk ash A, raw rice husk ash C, and the rice husk ash obtained in Example 1. As shown in Figure 1, the presence or absence of the processing of rice husk ash can be easily discriminated based on information

such as the presence of particles having plant-derived structures, the size of the largest particles, and the uniformity of particle size distribution.

[0068]

The apparent density of the rice husk ash in Comparative Example 1 is 300 g/L, whereas the apparent densities of the rice husk ash in Examples 1 to 4, which were subjected to the consolidation or pulverization processing, all increased significantly. Therefore, as the amount of rice husk ash filled into packaging bags increases, significant improvements in transportation and storage efficiencies can be expected. It was found that when the rice husk ash was excessively pulverized, the apparent density did not increase so much (Comparative Example 2). That is, even when using the same raw rice husk ash, the characteristics of the resulting rice husk ash differ depending on the processing conditions. Therefore, it was found that the processing of rice husk ash must be performed under appropriate conditions. In particular, it was found that the apparent density of rice husk ash increases significantly by pulverizing it followed by consolidating it (Example 4). Additionally, it was found that even when particle sizes were similar to each other, evaluations differed due to differences in the apparent densities (comparison of Example 4 and Comparative Example 2). This is considered because the apparent density is a parameter that also reflects the state of the void space between particles. From the perspective of providing rice husk ash suitable for manufacturing alkaline silicate aqueous solutions, the state of the void space is a crucial characteristic. Therefore, it can be considered that the solution to the problem of the present invention cannot be determined solely by particle size.

[0069]

Even in rice husk ashes obtained by different combustion methods (Comparative Examples 3 and 4), the same results as Comparative Example 1 were obtained due to their low apparent density. Furthermore, it was found that even when the same processing is performed, the characteristics of the resulting rice husk ash differ depending on the raw rice husk ash used. Therefore, the processing of rice husk ash must be performed under conditions appropriate for the raw rice husk ash (comparison of Example 3 and Comparative Examples 5 to 6).

[0070]

・Evaluation of dissolution test

In both the examples and comparative examples, alkaline silicate aqueous solutions with the target SiO_2 concentration (17 to 19 mass%) and molar ratio (3.00 to 3.40) were successfully prepared.

[0071]

Figure 2 is a photograph showing the appearance of the solution surface after the raw rice husk ash A had completely sunk into the sodium hydroxide aqueous solution in Dissolution Test (1) of Comparative Example 1, followed by stirring well for several tens of seconds. The rice husk ash in Comparative Example 1 had a long

sinking time and is a high-viscosity state. In Comparative Example 1, the solubility was insufficient because the high viscosity state continued for a while.

[0072]

In contrast, the rice husk ashes in Examples 1 to 4 all exhibited low dust generation, short sinking time of rice husk ash, low viscosity, and excellent solubility. In the case of Comparative Example 2, where excessive pulverization was performed, dust was generated and the sinking time increased. However, it was found that by additionally consolidating the rice husk ash, dust generation can be suppressed and an appropriate rice husk ash with a short sinking time can be obtained.

[0073]

As described above, it was found that a rice husk ash suitable for producing alkaline silicate aqueous solutions can be provided by appropriately processing the rice husk ash so that its apparent density becomes 400 to 800 g/L.

[0074]

< Example 5>

An attempt was made to produce a high-concentration alkaline silicate aqueous solution from the rice husk ash obtained in Example 1 (apparent density: 590 g/L) by the following Dissolution Method (2). If a high-concentration alkaline silicate aqueous solution can be produced, it would improve the production efficiency of standard-concentration alkaline silicate aqueous solutions and potentially broaden the applications of rice husk ash as a silica source.

[0075]

• Dissolution Method (2)

Dissolution Method (2) aimed to prepare an alkaline silicate aqueous solution having a SiO_2 concentration of 28 to 29 mass% and the above molar ratio of 3.00 to 3.40. 1,080 g of sodium hydroxide aqueous solution that was adjusted to 17.0 mass% was added to a 3L stainless steel vessel equipped with a stirring device and heated to 90°C. Then, 649 g of rice husk ash was added to the vessel while continuously stirring, the vessel was closed with a lid, and the mixing was continued for 6 hours while maintaining 90°C. The aqueous solution was then cooled to room temperature. A suction filter was prepared by attaching a magnetic Buchner funnel (Nutsche) with a diameter of 150 mm to a suction filtration bottle with a volume of 3 L, and placing a filter paper (mesh size 1 μm, Standard 5C, manufactured by Advantec Corporation) in the funnel. The suction filter was used to remove undissolved residues from the aqueous solution, thereby obtaining an alkaline silicate aqueous solution.

[0076]

< Comparative Example 7>

As a comparison to Example 5, an attempt was made to prepare a high-concentration alkaline silicate aqueous solution from the rice husk ash obtained in Comparative Example 1 (apparent density: 300 g/L) by Dissolution Method (2) in the same manner as in Example 5. However, all of the rice husk ash did not sink into the

sodium hydroxide aqueous solution, and the load on the stirring device increased, so the dissolution operation was stopped. As a result, dissolution according to Dissolution Method (2) could not be realized with the rice husk ash from Comparison Example 1.
[0077]
<Explanation of Results and Discussion I>

Regarding the dissolution of the rice husk ash obtained in Example 1 into sodium hydroxide aqueous solution by Dissolution Methods (1) and (2), Table 3 and Figure 3(a) show the relationship between the time elapsed after the feeding and the SiO_2 concentration. When the rice husk ash obtained in Example 1 (apparent density: 590 g/L) was used, it was possible to produce a high-concentration alkaline silicate aqueous solution with a SiO_2 concentration of 28 to 29 mass%. The thermal energy and time required for dissolving rice husk ash are nearly identical between Dissolution Methods (1) and (2). This result shows that use of the rice husk ash of the present invention allows for a dissolution of a larger amount of rice husk ash into alkaline aqueous solution using the same amount of thermal energy.
[0078]
[Table 3]

Time (H)		0.5	1.0	2.0	4.0	6.0
SiO_2 concentration [mass%]	Dissolution Method (1)	10.9	14.0	15.5	16.6	17.7
	Dissolution Method (2)	17.4	22.4	24.8	26.6	28.4

[0079]
< Example 6>

An attempt was made to produce a high-concentration alkaline silicate aqueous solution from the rice husk ash obtained in Example 1 (apparent density: 590 g/L) by a pressurized dissolution according to the following Dissolution Method (3). If the pressurized dissolution can be applied, the production time for the high-concentration alkaline silicate aqueous solution can be further reduced compared to Example 5. In the case of unprocessed rice husk ash (raw rice husk ashes A to C), the necessary amount could not even be loaded into the pressure vessel due to their low apparent densities.
[0080]
• Dissolution Method (3)

Dissolution Method (3) is a method for obtaining an alkaline silicate aqueous solution of the same quality as that obtained by Dissolution Method (2) in a shorter time than in Dissolution Method (2). 450 g of sodium hydroxide aqueous solution adjusted to 17.0 mass% and 270 g of the rice husk ash were fed into a 1L pressure vessel (autoclave) equipped with a stirring device while stirring, and the vessel was

closed with a lid. Dissolution was performed under the pressure conditions of 150°C and 0.47 MPa. When the dissolution was performed under pressurized conditions, the vessel was sealed, and thereby samplings during the process were prevented. Therefore, multiple experiments were conducted with pressurization times set to 0.5, 1, 2, and 3 hours, respectively. Once the dissolution was complete, the pressure was released and the aqueous solution was cooled to room temperature. After the cooling, a suction filter was prepared by attaching a magnetic Buchner funnel (Nutsche) with a diameter of 150 mm to a suction filtration bottle with a volume of 3 L, and placing a filter paper (mesh size 1 μm, Standard 5C, manufactured by Advantec Corporation) in the funnel. The suction filter was used to remove undissolved residues from the aqueous solution, thereby obtaining an alkaline silicate aqueous solution.

[0081]

Regarding the dissolution of the rice husk ash obtained in Example 1 into sodium hydroxide aqueous solution by Dissolution Methods (2) and (3), Table 4 and Figure 3(b) show the relationship between the time elapsed after the feeding and the SiO_2 concentration. When the rice husk ash obtained in Example 1 (apparent density: 590 g/L) was dissolved in an alkaline aqueous solution under the pressurized conditions, a high-concentration alkaline silicate aqueous solution could be produced in a shorter time than by Dissolution Method (2). Specifically, the time required for producing the high concentration alkaline silicate aqueous solution with a SiO_2 concentration of 28 to 29 mass% was 6 hours for Dissolution Method (2), whereas it was 2 hours for Dissolution Method (3).

[0082]

[Table 4]

Time (H)		0.5	1.0	2.0	3.0	4.0	6.0
SiO_2 concentration [mass%]	Dissolution Method (2)	17.4	22.4	24.8	-	26.6	28.4
	Dissolution Method (3)	20.3	26.1	28.9	29.6	-	-

[0083]

<Reference Example: Preparation Example of Wet Process Silica>

A confirmation was made as to whether or not the alkaline silicate aqueous solution produced with the rice husk ash of the present invention exhibits equivalent performance to the conventional sodium silicate aqueous solution derived from silica sand. Specifically, wet process silica (“hydrous silica”) for rubber tires was produced, and both properties of silica and rubber performance were evaluated. The production of wet process silica, evaluation of silica properties, and evaluation of rubber performance were performed using the same method as Example 1 described in

International Publication WO2020/031523. When there is a difference in evaluation devices in the descriptions of said international publication and the present specification, it means that an evaluation was performed using the evaluation device described in the present specification, according to the conditions described in said international publication.

[0084]

The alkaline silicate aqueous solution in the reference example was produced by producing a high-concentration alkaline silicate aqueous solution in the same manner as Example 5, except that the amount of materials was increased 60-fold, using a 240L vessel equipped with stirring, and then diluting it with water to adjust the SiO_2 concentration to 12.44 mass%, the Na_2O concentration to 3.74 mass%, the K_2O concentration to 0.41 mass%, and the $SiO_2/(Na_2O+K_2O)$ molar ratio to 3.20.

[0085]

The sodium silicate aqueous solution for comparison was produced by dissolving commercially available sodium silicate caret (medium molar grade, Na_2O: 24.51 mass% SiO_2: 75.44 mass%, purity 99.5 mass% or more, manufactured by Tokuyama Corporation) in hot water, and further diluting it with water to adjust the SiO_2 concentration to 12.44 mass%, the Na_2O concentration to 4.01 mass%, and the SiO_2/Na_2O molar ratio to 3.20.

[0086]

Subsequently, a wet process silica was produced with each of these alkaline silicate aqueous solutions. The physical property of the wet process silicas are shown in Table 5. In addition, a rubber formulation was produced with each of these wet process silicas. Evaluation of rubber performance is shown in Table 6.

[0087]

[Table 5]

Physical property of wet process silica		From silica sand	From rice husk ash of present invention
water content	%	4.0	3.6
pH (4%Susp.)		6.0	6.2
Electrical conductivity	μS/cm	41	45
BET specific surface area	m^2/g	316	314
Mercury pore volume at the side of high pressure Vol $[V_{HP-Hg}]$ r=3.6 to 100 nm	cm^3/g	1.69	1.62

Mercury pore volume at the side of medium pressure Vol r=100 to 1,000 nm	cm^3/g	0.48	0.52
Ultrasonic homogenizer Particle size after dispersion for 10 minutes	D50 μm	0.19	0.19
	D90 μm	2.97	3.08
Mercury pore volume at the side of low pressure Vol r=1.6 to 62 μm (r=1,600 to 62,000 nm)	cm^3/g	0.34	0.30
	Hysteresis cm^3/g	0.11	0.12
CTAB specific surface area	m^2/g	274	270
Particle hardness	cN	18	19
Residue on sieve	%	80	82

[0088]

[Table 6]

				From silica sand	From rice husk ash of present invention
Formulation preparation method 1	Dust Concentration	mg/m^3	A: less than 1.5	1.1 A	1.3 A
			B: 1.5-3.0		
			C: 3.0-4.5		
			D: 4.5 or more		
	Dispersibility Evaluation	X-value	A: 8.5 or more	8.6 A	8.8 A
			B: 8.0-8.5		
			C: 7.5-8.0		
			D: less than 7.5		
	Tear Strength	Index	A: 115 or more	120 A	117 A
			B: 105-115		
			C: 95-105		
			D: less than 95		

	Abrasion resistance	Index	A: 120 or more	128 A	129 A
			B: 110-120		
			C: 90-110		
			D: less than 90		
Formulation preparation method 2	Dispersibility Evaluation	X-value	A: 8.0 or more	8 A	8.2 A
			B: 7.5-8.0		
			C: 7.0-7.5		
			D: less than 7.0		
	Tear Strength	Index	A: 110 or more	116 A	111 A
			B: 105-110		
			C: 95-105		
			D: less than 95		
	Abrasion resistance	Index	A: 130 or more	157 A	152 A
			B: 110-130		
			C: 90-110		
			D: less than 90		

[0089]

The results in Table 5 indicate that it is possible to produce a wet process silica with physical property equivalent to those of sodium silicate solution derived from silica sand, even from the alkaline silicate aqueous solution produced with the rice husk ash of the present invention. Furthermore, the results in Table 6 indicate that the wet process silica produced with a raw material of the rice husk ash of the present invention imparts to rubber formulations with rubber property equivalent to those of wet process silica produced with a raw material of silica sand.

[0090]

As described above, since the rice husk ash of the present invention has an adequately high apparent density, the following effects can be expected.

(1) Compared to unprocessed rice husk ash, a large amount of rice husk ash can be fed into a vessel, allowing to obtain a slurry having a high concentration.

(2) Compared to unprocessed rice husk ash, the sinking property into alkaline aqueous solutions is improved, the solvent absorption is reduced, and the time and energy required for slurry preparation are reduced, thereby improving the productivity of alkaline silicate aqueous solutions.

(3) Compared to unprocessed rice husk ash, it is less bulky, it can be transported in a larger amount per single transportation, and it can be stored in a larger amount within a limited space, thereby improving the transportation and storage efficiencies.
(4) Compared to unprocessed rice husk ash, the dust generation is suppressed, and the handling property during packaging and unpacking is superior, thereby improving the working environment for handling rice husk ash.
(5) The rice husk ash of the present invention is a recycled waste material, but the performance of the alkaline silicate solution produced using it as a raw material is equivalent to that of sodium silicate aqueous solution derived from silica sand. It can be concluded that the rice husk ash of the present invention can be an alternative to silica sand and is an environmental-friendly material.

INDUSTRIAL APPLICABILITY

[0091]

The processed rice husk ash of the present invention can be used as a silica source not only for the production of alkaline silicate aqueous solutions but also for other applications.