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(54) Title: A PROCESS FOR THE PREPARATION OF SODIUM SILICATE FROM KIMBERLITE TAILINGS

(57) Abrégé/Abstract:

In the present invention a process for the preparation of sodium silicate from Kimberlite tailing generated as solid waste during diamond mining is disclosed. The process comprises, reacting Kimberlite tailing with mineral acid to remove acid soluble impurities followed by digesting acid treated Kimberlite tailing with alkali solution in a open or closed system to obtain sodium silicate useful for commercial applications.



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(54) Title: A PROCESS FOR THE PREPARATION OF SODIUM SILICATE FROM KIMBERLITE TAILINGS

(57) Abstract: In the present invention a process for the preparation of sodium silicate from Kimberlite tailing generated as solid waste during diamond mining is disclosed. The process comprises, reacting Kimberlite tailing with mineral acid to remove acid soluble impurities followed by digesting acid treated Kimberlite tailing with alkali solution in an open or closed system to obtain sodium silicate useful for commercial applications.

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A PROCESS FOR THE PREPARATION OF SODIUM SILICATE FROM KIMBERLITE TAILINGS

FIELD OF THE INVENTION

Around four million tones of soluble silicates are produced annually all over the world that
5 find major applications in the detergent, pigment, catalyst and silica gel producing industries.
These silicates are also used in adhesives, cement & building materials as well as in the pulp
and paper industry. Soluble silicates are also used for general purposes such as cleaning, e.g.
water cleaning, plant cleaning in the brewing, dairy, food processing and metal processing
industries. In foundry industry, silicates are used as a binder for production of the sand cores
10 and moulds from which castings can be produced. They also find application for binding
insulating materials such as vermiculite, perlite and many minerals and ores, sealing porous
surfaces such as asbestos and fibrous materials.

Alkali silicates are a family of chemicals with a wide range of physical and chemical
properties. These are generally prepared by fusing sodium or potassium carbonate with sand
15 at greater than 1000°C or digesting sand with sodium or potassium hydroxide solution under
pressure. The characteristics of alkali silicates permit their use in a variety of applications
such as: welding rods, soaps, detergents, hard-surface cleaners, coating materials, and
electronics.

Sodium silicate, in particular, is used for the manufacture of specialty inorganic materials
20 namely precipitated silica, silica sol / colloidal silica and silica gel, calcium and aluminium
silicates, zinc and titanium silicates, magnesium trisilicate, zeolites etc. Sodium silicate is
also used in ore flotation, canal lining and in the consolidation of earth.

Kimberlite tailings are produced as a huge solid waste during diamond mining. In a country
like India from its Panna diamond mines, typically around 100 tones of Kimberlite is
25 generated per 10 carat of diamond mined. Around 3-4 million tones of Kimberlite is already
accumulated during previous diamond mining in India. With an estimated life of 20 years for
the Panna mines, a huge quantity of Kimberlite tailings waste is produced in the country.
Considering this problem on a global scale, as countries like South Africa and Canada are

also involved in diamond mining, the quantity of Kimberlite tailings generated is of serious concern. Therefore, it is pertinent to look for technical solutions to gainfully utilize Kimberlite accumulated during diamond mining. Typical chemical composition of Kimberlite is as follows:

5 SiO_2 30 - 32%, Al_2O_3 2 - 5%, TiO_2 5 - 8%, CaO 8 - 10%, MgO 20 - 24%, Fe_2O_3 5 - 11%, LOI 13 - 15%

As Kimberlite is rich in magnesia and silica, there is an opportunity to develop silica and magnesium based products from this material. Therefore, efforts have been made to prepare value-added product like Sodium silicate after enrichment of silica content followed by
10 chemical treatment. The value-addition of Kimberlite will not only make diamond mining more economical but will make it an environmentally friendly process also.

BACKGROUND OF THE INVENTION

US Patent No. 2,219,646 (1940) assigned to Beecher and Mich disclosed a process for the preparation of sodium silicate by dry fusion of silica with sodium hydroxide at temperature
15 below the fusion or melting point of sodium hydroxide and about 2 to 4 % soda ash is added to the reaction mixture. The source of silica could be silica sand, quartz, volcanic ash, silica flour etc. The drawback of the process is the high temperature fusion of silica with sodium hydroxide which is energy intensive and uneconomical.

US Patent No. 2,829,030 (1958) assigned to Habernickel discloses a process for producing
20 alkali metal silicates having $\text{SiO}_2/\text{Na}_2\text{O}$ mole ratio 2.74 to 3.16 from a mixture of sand and alkali chlorides with about 2 to 4 % other alkali metal salts, such as sodium carbonate, sodium bicarbonate, potassium carbonate and bicarbonate or a mixture of them under the action of steam. The process involves the sintering step in which mixture of silica and alkali metal chloride is ground and then sintered at 780°C to 840°C and then cooled before steam
25 treatment at 1150°C to 1250°C . This process replaces conventionally used alkali carbonates and sulfates with cheaper alkali chlorides. However, the process is highly energy intensive as it is carried out at a high reaction temperature of $1150\text{-}1250^\circ\text{C}$ for 2-6 hours and involves pretreatment of sintering of the reaction mixture.

US Patent No. 2,988,423 (1961) assigned to McDaniel describes a process for the preparation of sodium silicate having $\text{SiO}_2/\text{Na}_2\text{O}$ mole ratio in the range of 1.0 to 2.8 by reacting silica sand and aqueous solution of sodium carbonate at 155°C to 217°C under pressure of 65 to 300 pound per square inch for about 20 hours or more. The drawbacks of the process are that the mole ratio of the sodium silicate prepared is comparatively low, and high reaction pressure and temperature and very long reaction time make the process uneconomical.

US Patent No. 3,984,526 (1976) assigned to Haase, et al. discloses a process for the preparation of alkali metal polysilicate solution having an SiO_2 :alkali metal oxide mole ratio of 2.5 - 5.5 : 1 and a silica content of 16 - 23% by weight, which comprises contacting a tetraalkoxysilane of the formula $\text{Si}(\text{OR})_4$, wherein each R is independently a straight or branched- chain alkyl group of 1 to 3 carbon atoms with an aqueous alkali metal hydroxide solution at a temperature between room temperature and the boiling point of the reaction mixture and at a temperature up to 100°C , distilled over alcohol formed by the reaction of the silane with the alkali metal hydroxide. The drawback of this process is that the source of silica is tetraalkoxysilane which makes the process uneconomical.

US Patent No. 4,029,736 (1977) assigned to Melkonian, discloses a process for the preparation of water glass using perlite as a source of silica by treating the same with an alkaline solution having a concentration of 40-140 g/l, used in an amount which brings the ratio of the liquid and solid phases to (0.7-1.5): 1 and then separating by filtration the water glass, obtained in the process of heat treatment, from the residue formed. The drawback of the process is the reaction of perlite with alkali metal hydroxide at 130°C to 150°C under autogenous pressure.

US Patent 4,336,235 (1982) assigned to Deabriges Jean discloses a process for the manufacture of sodium silicate solution in a continuous manner from a silicon dioxide-containing material. This process includes the continuous addition of a stream of said silicon dioxide-containing material and sodium hydroxide-sodium carbonate solution to a reaction zone at a temperature 225 - 245°C and pressure 27-32 bar and for a time sufficient to form a sodium silicate solution, and recovering said sodium silicate solution from the reaction zone. In this process the stream of NaOH - Na_2CO_3 is pre-heated at a temperature 250 - 280°C in a nickel plated heat exchanger. This requires high cost equipment and operating condition

which means that the utility cost required is comparatively high, which increases the production cost.

US Patent No. 4,520,001 (1985) assigned to Metzger, et al. discloses a process for the preparation of a clear solution of alkali metal silicate having an SiO_2 /alkali metal oxide weight ratio of 2.5 or less which comprises passing an aqueous solution of an alkali metal hydroxide through a bed of crystallized silica having an average particle size of between about 0.1 mm and 2 mm formed in the bottom of a vertical tubular reactor without mechanical agitation, said silica and alkali metal hydroxide being fed from the top of the reactor, and recovering the resulting clear solution of alkali metal silicate from the bottom of the reactor. The reaction temperature was kept between 150°C and 240°C . The drawbacks of the process includes that the SiO_2 /alkali metal oxide weight ratio is 2.5 or less; high SiO_2 /alkali ratio silicate cannot be obtained; and a minimum 150°C reaction temperature is required.

US Patent No. 4,676,953 (1987) assigned to Jeromin, et al. describes a process for the continuous production of sodium silicate having a mole ratio of $\text{SiO}_2/\text{Na}_2\text{O}$ 1-2.8:1 by fusing sand in aqueous sodium hydroxide solution at 150 - 300°C and 5-40 bar pressure. The drawback of the process is the high reaction temperature under high pressure which makes the process uneconomical.

US Patent No. 4,770,866 (1988) assigned to Christophliemk, et al. discloses a the process of producing sodium silicate by melting quartz sand and soda together at $1,400^\circ\text{C}$ to $1,500^\circ\text{C}$ in a suitable furnace with evolution of CO_2 . The melt that solidifies upon cooling in the form of a glass lump is then dissolved in water in another process step carried out under pressure at elevated temperature. The solution thus obtained is optionally filtered, depending upon the quality requirements. However, this process consumes very high amounts of energy because of the very high process temperature.

US Patent No. 5,000,933 (1991) assigned to Novotny, et al. discloses a process for the preparation of sodium silicate having a high $\text{SiO}_2/\text{Na}_2\text{O}$ molar ratio, by reaction of a silicon dioxide source with an aqueous sodium hydroxide solution, or with an aqueous sodium silicate solution using a silicon dioxide source that contains a sufficient fraction of

cristobalite phase or by conditioning other crystalline forms or by conditioning other crystalline forms of silicon dioxide by heating at or above 1100° C, but below the melting point of silica, before the hydrothermal treatment. The reaction is carried out in a closed pressure reactor at temperatures of 150° C to 300° C and under saturated steam pressures
 5 corresponding to those temperatures. The drawback of this process is the requirement of a sufficient amount of cristobalite phase of SiO₂ in the silica source, and if not, the silica source is required to be treated at very high temperature before the hydrothermal reaction with sodium hydroxide solution. Another drawback of the process is that the hydrothermal reaction is carried out in the range of 150° C to 300° C under autogenous pressure.

10 According to the above prior art, sodium silicate is prepared using quartz sand, cristobalite, perlite etc. and sodium carbonate or sodium hydroxide. Processes known in the prior art for the preparation of sodium silicates are either energy intensive or the operating conditions are such that the utility cost is very high. Nothing is reported in published or patented literature wherein Kimberlite tailings have been used as a source of silica for the preparation of sodium
 15 silicate and other value added silica based products.

DESCRIPTION OF THE INVENTION

The main object of the present invention is to use Kimberlite tailings as a source of silica for the preparation of sodium silicate.

Another object of the present invention is to prepare sodium silicate employing an energy
 20 efficient process which obviates the drawbacks as detailed above.

Yet another object of the present invention is to provide a process for enabling maximum recovery of silica from Kimberlite tailings generated during diamond mining.

Yet another object of the present invention is to provide a process for preparing sodium silicate with different SiO₂/ Na₂O mole ratios suitable for various commercial applications.

25 Yet another object of the present invention is to provide a process wherein Kimberlite tailings are substantially enriched in silica content for its use as a silica source.

A known weight of Kimberlite tailings is added to 18% wt. /Vol. of hydrochloric acid previously taken in a glass round bottom flask, keeping solid to liquid ratio 1:4 with continuous stirring at boiling temperature under refluxing conditions. After refluxing for 3 to 5 hours, the slurry is filtered and wet cake is washed with tap water until free from acid. For the preparation of sodium silicate, acid treated Kimberlite tailings thus obtained is digested with 8 to 10 weight percent NaOH solution keeping solid to liquid ratio as 1:4. The required quantity of sodium hydroxide is dissolved in a requisite volume of water in a stainless steel jacketed reactor. To this alkali solution, a required weight of acid treated Kimberlite tailings is added slowly under stirring in order to prepare a homogenous slurry. The reaction temperature is achieved by means of passing steam through the jacket of the reactor. After attaining the temperature, the digestion is continued for a specific time under constant stirring. After completion of the reaction, the slurry is filtered and residue is washed with tap water to recover silica and alkali. Filtrate and wash water are analyzed for its SiO_2 and Na_2O content.

15 SUMMARY OF THE INVENTION

Accordingly, a process for a process for the recovery of silica from Kimberlite tailings which comprises: (i) treating Kimberlite tailings with 2 to 15% wt./vol. mineral acid for a period ranging between 3-5 hours at a temperature ranging between 95-100°C to obtain a silica enriched material as a solid; (ii) digesting the silica enriched solid obtained in step (i) with an alkali solution of strength ranging between 8-10% wt./vol. in a closed system at 95-190°C or in an open system at boiling temperature for a period ranging between 3-4 hours to obtain sodium silicate having a ratio of SiO_2 to Na_2O ranging between 2.3 to 3.3.

Accordingly, the present invention provides a process for maximum recovery of silica from Kimberlite tailings wherein initially Kimberlite tailings is treated with 1 to 5 N mineral acid such as hydrochloric acid for a period of 3 to 5 hours at 95 - 100°C to remove acid soluble impurities as filtrate and to obtain silica rich material as solids, following which silica rich solid is digested with 8 to 10 % alkali solution, namely sodium hydroxide, in a closed system at 95 - 190° C or in an open system at boiling temperature for 3 to 4 hours to prepare sodium silicate of the required properties useful for commercial applications.

The Raw Kimberlite typically contains about 32-35% silica. In order to recover silica and to convert this silica into value added product, it is essential to increase the silica content of the Kimberlite tailings by means of an upgrading process. Enrichment of silica in the Kimberlite tailings is necessary for its effective use as a source of silica to produce sodium silicate.

5 Kimberlite tailings was treated with hydrochloric acid at boiling temperature under refluxing conditions and continuous stirring to remove the acid soluble impurities. The silica content in the acid treated Kimberlite tailings increases up to 72-78%. Kimberlite tailings enriched in silica is used for the preparation of storage stable sodium silicate.

10 In an embodiment of the present invention, Kimberlite tailings is ground using a Pin mill and passed through a 60 mesh sieve.

In another embodiment of the present invention, the 60-mesh fraction of Kimberlite tailings is treated with mineral acid such as hydrochloric acid at boiling temperature under refluxing conditions with continuous stirring to remove the acid soluble impurities present in the Kimberlite tailing as a filtrate.

15 In yet another embodiment of the present invention, the acid treated Kimberlite tailing which is rich in silica content is digested with an alkali such as sodium hydroxide solution at 105°C under stirring for a fixed period of 3 to 5 hours.

In yet another embodiment of the present invention the alkali digested Kimberlite tailings is filtered using vacuum filtration system.

20 In yet another embodiment, 25% loss of solid was observed during the acid treatment of the Kimberlite tailings as soluble metallic salts in the filtrate which can be further used for obtaining value added products like $\text{Mg}(\text{OH})_2$, MgO and Fe_2O_3 .

In yet another embodiment, 75-85 % silica recovery was obtained from the acid treated Kimberlite tailings at the boiling temperature.

25 In yet another embodiment, the total reduction of solids was found to be 82-87%, which could remarkably reduce the quantity of solid waste generated during diamond mining. Moreover, Kimberlite tailings - the solid waste generated during diamond mining - is converted to silica rich raw material for producing sodium silicate.

The digestion of acid treated-silica rich Kimberlite with a sodium hydroxide solution at relatively moderate temperature and atmospheric pressure gives sodium silicate essentially because the silica present in Kimberlite is an active silica and not in a highly thermodynamically stable form such as Quartz. The sodium silicate obtained is analyzed as
5 per the following methods.

Analysis of Sodium Silicate

1. Preparation of Sodium silicate sample:

Weigh accurately sodium silicate (~10.0 g) in a pre-weighed sample bottle, transfer the sample quantitatively into a 250ml volumetric flask, wash the sample bottle thoroughly with
10 distilled water and transfer the contents to the volumetric flask, wash the bottle repeatedly and transfer the wash water to the volumetric flask quantitatively. Shake the solution well and make up the volume to a mark on the flask with distilled water, mix it thoroughly by shaking the flask and label it as stock solution.

2. Analysis of SiO₂ in Sodium Silicate:

Pipette out 50ml sodium silicate solution from the stock solution by using a 50 ml pipette and transfer the solution into a clean beaker containing 50.0 ml 1:1 HCl solution, mix the solution well with a glass rod. Evaporate the contents of the beaker to complete dryness on the water bath, crush all lumps with a glass rod. Add 50.0 ml 1:1 HCl, mix the solid lumps and acid with the help of the glass rod, and again evaporate the acid solution to dryness. Repeat the
20 procedure thrice. After complete evaporation, when the powder appears to be dry, place the beaker in an air oven at 100 - 110°C for 1 hour in order to dehydrate the silica. Moisten the residue with 5 ml of 1:1 HCl, and bring the acid into contact with the solid with the aid of a stirring rod. Add 75 ml of distilled water to rinse down the sides of the beaker and heat the beaker on a water bath for 10-20 minutes. Filter off the separated silica on a Whatman - 42
25 filter paper, wash the precipitate first with warm dilute HCl and then with hot distilled water until free from chloride, pour the filtrate and washings into the original beaker, evaporate to dryness on the water bath, and heat in an air oven at 100-110°C for 1 hour. Moisten the residue with 5 ml 1:1 HCl, add 75 ml distilled water, warm the beaker to extract soluble salts, and filter through a fresh but smaller filter paper wash with warm dilute HCl and finally with
30 a little hot distilled water. [The mother liquor + washing are to be used for the analysis of

other elements.] Fold the moist filter papers and place them in a pre-weighed platinum crucible, dry the paper, char the paper, and burn off the carbon over a low flame and take care that none of the fine powder is blown away. After burning the carbon completely, cover the crucible, and heat it for an hour at the full temperature ($1000 \pm 25^\circ\text{C}$). Allow the crucible to cool in a desiccator, and weigh it. Repeat the ignition until the weight is constant, record the weight (W_2). Moisten the residue with 1.0 ml distilled water; add 2 to 3 drops of concentrated H_2SO_4 (AR grade) and about 5 ml of the purest available (AR grade) HF. Place the crucible in water bath and evaporate the HF in a fume chamber with small flame or on a hot plate until the acid is completely expelled. The liquid should not be boiled. Increase the heat to volatilize the H_2SO_4 and finally in a muffle furnace at 1000°C for 1 hour. Allow the crucible to cool in a desiccator and weigh it. Re-heat the crucible to constant weight (W_3). The loss in weight represents the weight of the pure silica (SiO_2). Find % of pure silica (SiO_2) using the following equation:

$$\% \text{SiO}_2 = [(W_2 - W_3)/W_1] \times 1000$$

where, W_1 = weight of sodium silicate taken for the preparation of stock solution.

3. Analysis of Na_2O in Sodium silicate sample:

Pipette out 25 ml of solution from the stock solution with a 25 ml pipette and dilute it to ~ 100 ml with distilled water in 250 ml conical flask, add 2-3 drops of methyl orange indicator, titrate it against standard HCl (0.1 N), until a color change from yellow to orange is observed, note the burette reading and find % Na_2O using the following equation:

$$\% \text{Na}_2\text{O} = N \times \text{BR} \times 31/W_1$$

where N = Normality of acid; BR == Burette Reading; W_1 = Weight of sodium silicate taken for the preparation of stock solution.

The following examples are given by way of illustration only and therefore should not be constructed to limit the scope of the present invention.

Example 1

15 g of Kimberlite tailings was placed in a Teflon™ container with 5 g of sodium hydroxide. To this 20 g of water is added. Then the container was closed tightly and kept in an air oven for 5 hours at 150°C . After completion of the reaction, the slurry was centrifuged and the filtrate was analyzed for SiO_2 and Na_2O . The $\text{SiO}_2/\text{Na}_2\text{O}$ mole ratio in sodium silicate was

found to be 1.81. The stability of the sodium silicate produced was found to be poor. Hydrolysis of sodium silicate to silica particles was observed during storing of the product at room temperature within 10-15 days for the above product due to the presence of impurity in the sodium silicate.

5 **Examples 2**

100 g of alkali was dissolved in 2 liters of tap water and placed in to a glass round bottom flask. To this, 500 g of Kimberlite tailings was added under continuous stirring to prepare a homogenous slurry and heated for 5 hours at 95-100°C. After completion of the reaction, the slurry was filtered with a vacuum filtration system and the filtrate was analyzed for SiO₂ and Na₂O. The SiO₂/Na₂O mole ratio in sodium silicate was 2.72. Hydrolysis of sodium silicate to silica particles was observed during storing of the product at room temperature within 10-15 days for this product due to the presence of some impurity in the sodium silicate.

Example 3

70 g of Kimberlite tailings was treated with 15% hydrochloric acid at 95-100°C under continuous stirring and keeping solid to liquid ratio 1:10 for 5 hours. Then the slurry was filtered and washed with tap water until the wet cake became acid free. After completion of the washing, it was dried in an oven at 110°C and the dry solid was used for sodium silicate preparation. For the preparation of sodium silicate, 16 g of alkali was dissolved in 200 ml of water and placed in a stainless steel autoclave. To this, 50 g of acid treated Kimberlite tailings was added under continuous stirring. Then the reactor was closed and heated for 5 hours at 150°C. After completion of the reaction, the slurry was filtered under vacuum and the filtrate was analyzed for SiO₂ and Na₂O. The SiO₂/Na₂O mole ratio in sodium silicate was found to be 2.99. Recovery of silica in sodium silicate was 78%. The sodium silicate prepared from the acid treated Kimberlite tailings was found to be stable and no hydrolysis of silica was observed on prolong storage. Composition in weight percent of this sodium silicate was found as follows:

SiO₂ = 13.40%, and Na₂O = 4.25%.

Example 4

140 g of Kimberlite tailings was treated with 15% hydrochloric acid at 95-100°C under continuous stirring keeping the solid to liquid ratio 1:10 for 5 hours, then the slurry was

filtered and washed with tap water until the formed wet solid cake became acid free. After completion of washing the cake was dried in an oven at 110°C and the dry solid was used for the preparation of sodium silicate. 32 g of alkali was dissolved in 300 ml of water and placed in a stainless steel autoclave. To this, 100 g of acid treated Kimberlite tailings was added under continuous stirring. The reactor was closed and heated for 5 hours at 150°C. After completion of the reaction, the reaction slurry was filtered using a Buckner funnel under vacuum and the filtrate was analyzed for SiO₂ and Na₂O. The SiO₂/Na₂O mole ratio in sodium silicate was found to be 3.36. Recovery of the silica in sodium silicate was found to be 90 %. Sodium silicate composition in weight percent was SiO₂ = 21.42 %, and Na₂O = 7.18 %.

Example 5

200 g of Kimberlite tailings was treated with 15% hydrochloric acid at 95-100°C under continuous stirring, keeping the solid to liquid ratio 1:4 for 3.5 hours, then the slurry was filtered and washed with tap water until a wet solid cake became iron free. Then the slurry was dried in an oven at 110°C and the dry solid was used for the preparation of sodium silicate. 40 g of alkali dissolved in 280 ml of water was placed in a stainless steel autoclave. To this, 150 g of acid treated Kimberlite tailings was added under continuous stirring to prepare a homogenous slurry. Then the reactor was closed and heated for 5 hours at 170°C. After completion of the reaction, the slurry was filtered with vacuum filter and the filtrate was analyzed for SiO₂ and Na₂O. The SiO₂/Na₂O mole ratio in sodium silicate was found to be 3.28. The recovery of silica in sodium silicate was found to be 30%. Reduction in silica recovery may be due to a lower solid to liquid ratio and an increase in the viscosity of the reaction mixture. Sodium silicate composition in weight percent is given below:

	Component	% by Wt.
25	SiO ₂	16.55
	Na ₂ O	5.22
	R ₂ O ₃	0.082
	CaO+MgO	0.044

Example 6

30 135 g of Kimberlite tailings was treated with hydrochloric acid having a concentration of 15% by wt. at 95-100°C under continuous stirring, keeping the solid to liquid ratio 1:4 for 3.5

hours, then the slurry was filtered and washed with tap water until the wet solid cake became acid free. It was then dried in an oven at 110°C and the dry solid was used for the preparation of sodium silicate. For the preparation of sodium silicate 31.2 g of alkali dissolved in 400 g of water was placed in a stainless steel autoclave. To this 100 g of acid treated Kimberlite
 5 tailings was added under continuous stirring to prepare a homogenous slurry. Then the reactor was heated for 2 hours at boiling temperature. After completion of the reaction, the slurry was filtered with a vacuum filter and the filtrate was analyzed for SiO₂ and Na₂O. The SiO₂/Na₂O mole ratio in sodium silicate was found to be 3.30. Recovery of silica in sodium silicate was found to be 81 %. Analysis of the product is given below.

10	Component	% by Wt.
	SiO ₂	15.18
	Na ₂ O	4.20
	R ₂ O ₃	0.087
	CaO+MgO	0.017

15 **Example 7**

2.7 kg of Kimberlite tailings was treated with 15% hydrochloric acid at 95-100°C under continuous stirring, keeping the solid to liquid ratio 1:4 for 3 hours, then the slurry was filtered and washed with tap water until the wet solid cake became acid free. The cake was dried in an oven at 110°C and was used for preparation of sodium silicate. For the preparation
 20 of sodium silicate 0.64 kg of an alkali was dissolved in 8 liters of water and placed in a stainless steel autoclave. To this 2 kg of acid treated Kimberlite tailings was added under continuous stirring to prepare a homogenous slurry. Then the reactor was heated for 3.5 hours at boiling temperature. After completion of the reaction, the slurry was filtered with a vacuum filter and the filtrate was analyzed for SiO₂ and Na₂O. The SiO₂/Na₂O mole ratio was 3.25.
 25 Recovery of silica as sodium silicate was found to be 75 %. Analysis of sodium silicate obtained is given below:

	Component	% by Wt.
	SiO ₂	13.73
	Na ₂ O	4.36
30	R ₂ O ₃	0.047
	CaO+MgO	0.017

Example 8

23 kg of Kimberlite tailings was treated with 15% hydrochloric acid at 95-100°C, under continuous stirring, keeping the solid to liquid ratio 1:4 for 3 hours, then the slurry was filtered and washed with tap water until the wet solid cake became acid free. Then it was
 5 dried in an oven at 110°C and the dry solid was used for the preparation of sodium silicate. 5.44 kg of alkali dissolved in 68 liters of water was placed in a stainless steel reactor. To this, 17 kg of acid treated Kimberlite tailings was added under continuous stirring to prepare a homogenous slurry. Then the reactor was heated for 3.5 hours at boiling temperature. After completion of the reaction, the slurry was filtered with a vacuum filter, and filtrate was
 10 analyzed for SiO₂ and Na₂O. The SiO₂/Na₂O mole ratio in sodium silicate was found to be 3.16. Recovery of silica in sodium silicate was found to be 73%. Analysis of sodium silicate obtained is given below:

	Component	% by Wt.
	SiO ₂	14.63
15	Na ₂ O	4.77
	R ₂ O ₃	0.052
	CaO+MgO	0.088

Example 9

Sodium silicate was prepared following the procedure as described in Example 8 having a
 20 dark brownish color probably due to the presence of some organic compounds. This was used as such for the preparation of precipitated silica and Zeolite-A. However, to improve the quality and color of sodium silicate, decolourization of sodium silicate was done using activated carbon as a decolorizing agent. 1 kg of sodium silicate was placed in a stainless steel reactor and heated to 75-80°C for 2 hours after adding 3-4 % by wt. of commercially
 25 available activated charcoal under continuous stirring and then filtered. After the carbon treatment the sodium silicate obtained was colorless.

The process of preparing sodium silicate by the present process is novel because it makes use of raw material considered to be solid waste and has not been reported to be used for the said process previously. From a technical view point, the process is novel as it involves relatively
 30 low temperatures (95-105°C) and atmospheric pressure conditions for the preparation of sodium silicate. Quartz or silica sand used in conventional processes for the production of

sodium silicate requires high temperature ($\sim 1100^{\circ}\text{C}$) reaction with sodium carbonate as the quartz crystalline material and a thermodynamically stable form of SiO_2 . However, Kimberlite tailings is a serpentine mineral, which after acid treatment is converted to silica in amorphous form and is comparatively active and a better starting material for producing sodium silicate. The process developed is useful for the value addition of the waste material produced during diamond mining. This will make diamond mining both economically attractive as well as eco-friendly. Furthermore, this will lessen the requirement for naturally occurring good quality sand used for producing sodium silicate and thus contribute towards preserving the ecological balance.

10

We claim:

1. A process for the recovery of silica from Kimberlite tailings which comprises:
 - (i) treating Kimberlite tailings with 2 to 15% wt./vol. mineral acid for a period ranging between 3-5 hours at a temperature ranging between 95-100°C to obtain a silica enriched material as a solid;
 - (ii) digesting the silica enriched solid obtained in step (i) with an alkali solution of strength ranging between 8-10% wt./vol. in a closed system at 95-190°C or in an open system at boiling temperature for a period ranging between 3-4 hours to obtain sodium silicate having a ratio of SiO₂ to Na₂O ranging between 2.3 to 3.3.
2. A process as claimed in claim 1, wherein the sodium silicate obtained comprises: 13 to 16% by weight of SiO₂; 4 to 6% by weight of Na₂O; 0.04 to 0.09 % by weight of R₂O₃; and 0.044 to 0.088% by weight of CaO + MgO.
3. A process as claimed in claim 1, wherein the Kimberlite tailings has a chemical composition comprising:

SiO₂ 30-32% by weight, Al₂O₃ 2-5% by weight, TiO₂ 5-8% by weight, CaO 8-10% by weight, MgO 20-24% by weight, Fe₂O₃ 5-11% by weight, and a Loss on Ignition (LOI) of 13-15% by weight.
4. A process as claimed in any one of claims 1-3, where the Kimberlite tailings used for acid treatment and further alkali treatment for preparing sodium silicate are generated during diamond mining.
5. A process as claimed in any one of claims 1 to 4, wherein 15% by wt./vol. of hydrochloric acid is used for treating the Kimberlite tailings at a temperature ranging between 95-100°C with the Kimberlite to acid ratio being 1:4 for the time duration of 3 to 5 hours.

6. A process as claimed in any one of claims 1-5, wherein the silica content in the silica enriched material ranges 72 to 78% by weight.
7. A process as claimed in any one of claims 1 to 6, wherein the acid treated silica rich solid is digested with sodium hydroxide at a temperature ranging between 95-105°C with a ratio of solid to alkali of 3 to 5 for the time duration of 2 to 6 hours.
8. A process as claimed in any one of claims 1 to 7, wherein the sodium silicate produced is further purified by treatment with activated carbon.
9. A process as claimed in any one of claims 1 to 8, wherein 3-4% by wt. of activated carbon is used for the purification of sodium silicate.
10. A process as claimed in any of claims 1 to 9, wherein the amount of silica recovered from Kimberlite tailings is 73 to 90% by weight.