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(54) **Titre : NOUVEAU MATERIAU AINSI QUE SA PREPARATION, DESTINE A ETRE UTILISE COMME MILIEU D'ACCUMULATION DANS UN SYSTEME SENSIBLE D'ACCUMULATION D'ENERGIE, DANS UNE PLAGE DE TEMPERATURE BASSE, MOYENNE ET ELEVEE**

(54) **Title: NOVEL MATERIAL AND PRODUCTION THEREOF FOR USE AS STORAGE MEDIUM IN A SENSITIVE ENERGY STORAGE SYSTEM IN THE LOW-, MEDIUM- OR HIGH-TEMPERATURE SECTOR**

(57) **Abrégé/Abstract:**

The present invention relates to a modified red sludge or a modified bauxite residue and processes for producing same, and to a storage medium comprising a modified red sludge, a heat store comprising a storage medium and numerous uses of a modified red sludge as storage medium, more particularly in a heat store system. The modified red sludge here contains the following components: - haematite (Fe_2O_3), - corundum (Al_2O_3), - rutile (TiO_2) and/or anatase (TiO_2), - quartz (SiO_2), - optionally perovskite (CaTiO_3) and - optionally pseudobrookite ($(\text{Fe}^{3+}, \text{Fe}^{2+})_2(\text{Ti}, \text{Fe}^{3+})\text{O}_5$) and/or nepheline ($(\text{Na}, \text{K})[\text{AlSiO}_4]$). A novel material is thus provided, and production thereof is described for use as storage medium in a sensitive energy storage system in the low-, medium- or high-temperature sector.

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SPEICHERMEDIUM IM SENSITIVEN ENERGIESPEICHERSYSTEM IM NIEDRIGEN, MITTLEREN UND
HOCHTEMPERATURBEREICH(57) Abstract: The present invention relates to a modified red sludge or a modified bauxite residue and processes for producing
same, and to a storage medium comprising a modified red sludge, a heat store comprising a storage medium and numerous uses of a
modified red sludge as storage medium, more particularly in a heat store system. The modified red sludge here contains the
following components: - haematite (Fe₂O₃), - corundum (Al₂O₃), - rutile (TiO₂) and/or anatase (TiO₂), - quartz (SiO₂), - optionally
perovskite (CaTiO₃) and - optionally pseudobrookite ((Fe³⁺,Fe²⁺)₂(Ti,Fe³⁺)O₅) and/or nepheline ((Na,K)[AlSiO₄]). A novel material
is thus provided, and production thereof is described for use as storage medium in a sensitive energy storage system in the low-,
medium- or high-temperature sector.(57) Zusammenfassung: Die vorliegende Erfindung betrifft einen modifizierten Rotschlamm bzw. einen modifizierten
Bauxitrückstand sowie Verfahren zu dessen Herstellung und ein Speichermedium, umfassend einen modifizierten Rotschlamm,
einen Wärmespeicher, umfassend ein Speichermedium, und zahlreiche Verwendungen eines modifizierten Rotschlammes als ein
Speichermedium, insbesondere in einem Wärmespeicher. Dabei enthält der modifizierte Rotschlamm folgende Komponenten: -
Hämatit (Fe₂O₃), - Korund (Al₂O₃), - Rutil (TiO₂) und/oder Anatas (TiO₂), - Quarz (SiO₂), - optional Perowskit (CaTiO₃) und
- optional Pseudobrookit ((Fe³⁺,Fe²⁺)₂(Ti,Fe³⁺)O₅) und/oder Nephelin ((Na,K)[AlSiO₄]). Hierdurch wird ein neuartiges Material zur
Verfügung gestellt, sowie dessen Herstellung zur Anwendung als Speichermedium im sensitiven Energiespeichersystem im
niederen, mittleren und Hochtemperaturbereich beschrieben.

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Novel material and production thereof for use as storage medium in a sensitive energy storage system in the low-, medium- or high-temperature sector

5 The present invention relates to a modified red mud or a modified bauxite residue, hereafter also referred to as ALFERROCK®, and also to methods for the production thereof and uses thereof.

10 With the Renewable Energy Act (EEG) Germany has established that nuclear power and coal-fired power generation will be phased out by 2038. As substitutes, wind farms, solar installations and biogas installations should constitute the power supply for Germany.

15 In particular, however, wind farms and solar installations have the disadvantage that in still air and in darkness no power is generated. Thus these renewable energy sources are not capable of providing base load power for customers, in particular industrial customer, who are dependent upon a consistent power supply.

20 In order to avoid this systemic disadvantage, power generation and power consumption must be separated. Only energy accumulators which are connected between the power generators and the power consumers make it possible to provide a consistent power flow in the necessary manner and thus to make the renewable energy sources capable of providing base load power.

25 Therefore there is a demand for energy accumulators and suitable storage media therefor which can remedy these disadvantages in the production of power from renewable power sources and which can themselves also be obtained in a cost-effective manner which conserves resources.

30 It is known that red mud is produced as a waste product in the Bayer process for extracting aluminium hydroxide (ATH) from bauxite. In the following description red mud (RM) is understood to be the residue from the Bayer process which is produced in the extraction of ATH from bauxite. A large proportion of the bauxite residue or red mud produced in the extraction of aluminium is not further processed and put to an economically viable use, but is disposed of at disposal sites.

35 From the prior art it is already known that bauxite residue is suitable as an adsorbent

because of its large internal surface.

Furthermore, it is known that, because of its chemical composition, modified bauxite residue can be used as an inorganic, halogen-free flame retardant in plastics of all types, which are provided as a compound or also in foamed form (WO 2012/126487 A1).

Furthermore it is known that modified bauxite residue has a high density, so that this composition can also be used as soundproofing means or for drilling mud weighting or for shielding against radiation (WO 2014/114283 A1).

Furthermore, a porous, particulate material comprising bauxite residue for fluid treatment and removal of pollutants is known from WO 2005/061408 A1. The pollutants include, for example, heavy metals, anions and gases.

In experiments with modified, chromate-deficient bauxite residue which has been heated to temperatures between 120 °C and 250 °C the present inventors found that the cooling took place unexpectedly slowly. In the light of this observation the inventors of the present invention carried out comprehensive studies on the behaviour of (optionally chromate-deficient) bauxite residue or red mud, when it is subjected to a thermal treatment, and in this case made observations by comparing the chemical, mineralogical and physical parameters, in particular the thermal parameters. Thus the inventors arrived at a new type of modified red mud which can be used as a storage medium, in particular as a heat storage means.

In one embodiment a modified red mud contains the following components:

- haematite (Fe_2O_3),
- corundum (Al_2O_3),
- rutile (TiO_2) and/or anatase (TiO_2),
- quartz (SiO_2),
- optionally perovskite (CaTiO_3) and

- optionally pseudobrookite $(\text{Fe}^{3+}, \text{Fe}^{2+})_2(\text{Ti}, \text{Fe}^{3+})\text{O}_5$ and/or nepheline $((\text{Na}, \text{K})[\text{AlSiO}_4])$.

Thus the modified red mud can contain, in particular, the components haematite (Fe_2O_3), corundum (Al_2O_3), rutile (TiO_2) and/or anatase (TiO_2) and quartz (SiO_2) or substantially
 5 consist of these components. Further components can be included, but do not have to be included. In particular, perovskite (CaTiO_3), pseudobrookite $((\text{Fe}^{3+}, \text{Fe}^{2+})_2(\text{Ti}, \text{Fe}^{3+})\text{O}_5)$ and/or nepheline $((\text{Na}, \text{K})[\text{AlSiO}_4])$ may be mentioned as further (optional) components.

In one embodiment the modified red mud can contain

- 10 - 48 to 55 % by weight, in particular 49 to 54 % by weight, in particular 50 to 53 % by weight, of haematite (Fe_2O_3),
- 13 to 18 % by weight, in particular 14 to 17 % by weight, in particular 15 to 16 % by weight, of corundum (Al_2O_3),
- 8 to 12 % by weight, in particular 9 to 11 % by weight, of rutile (TiO_2) and/or anatase
 15 (TiO_2) and
- 2 to 5 % by weight, in particular 3 to 4 % by weight, of quartz (SiO_2).

In one embodiment a modified red mud can be obtained by heating of red mud having a mineral composition of

- 20 - 10 to 55 %, in particular 10 to 50 % by weight of iron compounds,
 - 12 to 35 % by weight of aluminium compounds,
 - 3 to 17 % by weight, in particular 5 to 17 % by weight of silicon compounds,
 - 2 to 12 % by weight, in particular 2 to 10 % by weight of titanium dioxide,
 - 0.5 to 6 % by weight of calcium compounds, and
 - 25 - optionally further unavoidable impurities,
- to a temperature of at least 800 °C, in particular at least 850 °C, in particular at least 900 °C, in particular at least 950 °C, preferably at least 1000 °C. A modified red mud which can be obtained in this way can also be designated as a tempered or sintered red mud.

30 The modified red mud can be treated at the stated temperature in particular over a time period of 5 minutes to 36 hours, in particular 5 minutes to 24 hours, in particular 5 minutes to 12 hours, in particular 5 minutes to 6 hours, in particular 5 minutes to 2 hours, in particular 5 minutes to 1 hour, in particular 5 minutes to 30 minutes.

35 In one embodiment the modified red mud can be substantially free of one, two, three or all

four of the following components:

- gibbsite ($\text{Al}(\text{OH})_3$),
- goethite (amorphous)
- boehmite ($\text{AlO}(\text{OH})$),
- 5 - cancrinite ($\text{Na}_6\text{Ca}_2[(\text{CO}_3)_2 | \text{Al}_6\text{Si}_6\text{O}_{24}]$).

In the context of the present application "substantially free" can be understood in particular as a content of less than 0.5 % by weight, in particular less than 0.2 % by weight, in particular less than 0.1 % by weight, in particular less than 0.05 % by weight.

10

In one embodiment the modified red mud can have a density in the range from 3.90 to 4.0 g/cm^3 , in particular from 3.91 to 3.95 g/cm^3 , in particular from 3.92 to 3.94 g/cm^3 , in particular approximately 3.93 g/cm^3 .

15

In one embodiment the modified red mud can have a mean particle size d_{50} in the range from 3 to 10 μm , in particular from 5 to 8 μm . The mean particle size d_{50} can be determined in particular by means of laser diffraction or (MALVERN) laser diffraction according to ISO 13320 (2009).

20

In one embodiment the modified red mud can have a particle size d_{10} in the range from 0.5 to 2.5 μm , in particular from 1.0 to 2.0 μm , and/or a particle size d_{90} in the range from 15 to 50 μm , in particular from 20 to 40 μm . The mean particle size d_{50} can be determined in particular by means of laser diffraction and the particle size d_{90} can be determined by means of laser diffraction (MALVERN) according to ISO 13320 (2009).

25

In one embodiment the modified red mud can have a specific thermal capacity at 20 °C in the range from 0.6 to 0.8 $\text{kJ}/(\text{kg}\cdot\text{K})$, in particular from 0.65 to 0.75 $\text{kJ}/(\text{kg}\cdot\text{K})$, and/or a specific thermal capacity at 726.8 °C in the range from 0.9 to 1.3 $\text{kJ}/(\text{kg}\cdot\text{K})$, in particular from 0.95 to 1.2 $\text{kJ}/(\text{kg}\cdot\text{K})$. The specific thermal capacity can be determined in particular according to DIN

30

EN ISO 11357-4.

35

In one embodiment the modified red mud can have a specific thermal conductivity in the range from 3 to 35 $\text{W}/(\text{m}\cdot\text{K})$, in particular from 5 to 20 $\text{W}/(\text{m}\cdot\text{K})$. The specific thermal conductivity can be determined in particular by means of plate-shaped test objects in a lambda meter according to DIN ISO 8302.

In one embodiment the modified red mud can be present in the form of a compressed solid material.

- 5 The method for producing a modified red mud comprises heating of red mud with a mineral composition of
- 10 to 55% by weight, in particular 10 to 50% by weight of iron compounds,
 - 12 to 35 % by weight of aluminium compounds,
 - 3 to 17 % by weight, in particular 5 to 17 % by weight of silicon compounds,
 - 10 - 2 to 12 % by weight, in particular 2 to 10 % by weight of titanium dioxide,
 - 0.5 to 6 % by weight of calcium compounds, and
 - optionally further unavoidable impurities,
- to a temperature of at least 800 °C, in particular at least 850 °C, in particular at least 900 °C, in particular at least 950 °C, preferably at least 1000 °C.

15

In one embodiment the modified red mud can be treated at the stated temperature in particular over a time period of 5 minutes to 36 hours, in particular 5 minutes to 24 hours, in particular 5 minutes to 12 hours, in particular 5 minutes to 6 hours, in particular 5 minutes to 2 hours, in particular 5 minutes to 1 hour, in particular 5 minutes to 30 minutes.

20

In one embodiment, in the method for production of a modified red mud a modified red mud according to the invention can be produced.

In one embodiment the method can further comprise a compression of the (sintered or
25 tempered) red mud after the heating.

The storage medium comprises a modified red mud according to the invention. In the context of the present application a "storage medium" can be understood in particular as the active (or actual) storing material. For example, in the case of a heat storage means the
30 storage medium can be a heat-storing material which must have a corresponding (or suitable) thermal capacity and thermal conductivity.

The storage medium can optionally contain further components, in addition to the modified red mud.

35

In one embodiment the storage medium can further comprise one or more of the following components:

- an agent for prevention of the inclusion of air (air absorption in the interior of the storage medium) and/or air adsorption (accumulation of air on the surface). (For example, 5 the matrix for the current/heat storage device can be treated by means of a double screw extruder with the addition of 5 to 10 % by weight of polydimethylsiloxane or polydiphenylsiloxane polymer and with simultaneous use of a vacuum degassing with a maximum output (for example series-connected rotary vane vacuum pumps) so that all air inclusions are eliminated by the intensive dispersion in the polysiloxane system. The 10 material obtained is kneadable).
- an agent for improvement of the thermal conductivity, in particular selected from the group consisting of metal colloids, metal powder, graphite and substances containing silicon,
- an agent for formation of a thixotropic composition. (In particular by the addition of 15 polypentaerythritol and a carboxylic acid (C18 for example) to the heat carrier matrix before the treatment in the double screw extruder, the matrix can be adjusted thixotropically for example in an extended range. In the activation process of the current/heat storage means, i.e. slow heating, in addition to the polysiloxane these agents can also be thermally decomposed to carbon, wherein the resulting carbon has no negative effects on the desired characteristics of the current/heat storage device means, but improves the thermal 20 conductivity).

The heat storage means comprises a storage medium according to the invention. In the context of the present application a "heat storage means" can be understood in particular as a device which contains the storage medium as an active heat-storing material and in 25 addition it optionally also has further apparatus elements.

In one embodiment the heat storage means can be a current/heat store. In the context of the present application a "current/heat store" can be understood in particular as a storage device which can convert electrical power into thermal power and/or can convert the thermal power 30 into electrical power, and which in particular can both convert electrical power into thermal power and also thermal power into electrical power.

In one embodiment the heat storage means can also comprise devices for charging and discharging the heat storage means. Devices for charging and discharging the heat storage 35 means can for example contain mechanical components, such as for example openings,

conveying or transport devices, etc., and/or other components, such as for example conductive contacts or connections, in particular electrically conductive contacts or connections and/or heat-conducting contacts or connections.

- 5 In one embodiment the heat storage means can be a current/heat store which also comprises resistance wires. As a result, in particular, a conversion of electrical power into thermal power is possible in the current/heat store. In this case the resistance wires can be in indirectly or direct contact with the storage medium.
- 10 The invention further relates to the use of a modified red mud according to the invention as a storage medium, in particular in a heat storage means.

In one embodiment the modified red mud can be used for storage of heat at a temperature up to 1000 °C, in particular at a temperature of more than 100 °C up to 1000 °C. However,
15 use for storage of heat at a temperature of more than 80 °C, in particular more than 90 °C is possible.

In one embodiment the modified red mud can be used as a storage medium which can be repeatedly heated and cooled (or in a heat storage means which can be repeatedly heated
20 and cooled).

In one embodiment the modified red mud can be used a storage medium which can be simultaneously heated and cooled.

- 25 In one embodiment the modified red mud can be used a storage medium in a current/heat store.

In one embodiment the storage medium can be heated by means of electrical current and/or cooled while electrical current is generated.
30

In one embodiment the modified red mud can be used for storage of electrical power obtained from renewable energy sources. In the context of the present application "renewable energy sources" can be understood in particular to be wind power, hydropower, tidal power, solar power, geothermal energy and biomass, but preferably renewable energy
35 sources for which the timescale is largely removed from human influence and for which

therefore a possibility of effective storage is very important, such as in particular wind power, tidal power and solar power.

5 In one embodiment the current/heat store can comprise resistance wires, which are heated by electrical current and thus heat the storage medium.

10 In one embodiment thermal power stored in the storage medium can be transferred to another medium and so the storage medium can be cooled (discharged), wherein the other medium is selected in particular from the group consisting of water, molten salt, such as for example ionic liquids, and thermal oil.

In one embodiment the storage medium and a discharge device can be provided integrally or as separate elements.

15 In one embodiment the current/heat store can be used for transport of electrical power after cogeneration has taken place without the presence of line systems.

20 In one embodiment the current/heat store can generate electrical current again after cogeneration.

In one embodiment the current/heat store can be used for supplying power to insulated energy consumers.

25 In one embodiment insulated energy consumers can be supplied with thermal energy and electrical energy.

In one embodiment the current/heat store can be used for supplying power to machines or mobile devices such as vehicles.

30 Further objects and advantages of embodiments of the present invention are disclosed with reference to the following detailed description and the attached drawings.

Figure 1 shows a particle size distribution of a dry conventional bauxite residue.

35 Figure 2 shows the density characteristics of a test sample during the heating of red mud from 100 °C up to 1000 °C in an oxygen (O₂) or a nitrogen (N₂) atmosphere.

Figure 3 shows a particle size distribution of a red mud tempered at 1000 °C according to an exemplary embodiment of the invention.

Figure 4 is a graphical representation of the series of measurements shown in Table 3 for the specific thermal capacity of ALFERROCK® according to an exemplary embodiment of
5 the invention.

Further details of the present invention and further embodiments thereof are described below. However, the present invention is not limited to the following detailed description, but it serves merely for illustration of the teaching according to the invention.

10

It may be pointed out that features which are described in connection with an exemplary embodiment or an exemplary subject can be combined with any other exemplary embodiment or with any other exemplary subject. In particular, features which are described in connection with an exemplary embodiment of a modified red mud according to the
15 invention can be combined with any other exemplary embodiment of a modified red mud according to the invention as well as with any exemplary embodiment of a method for production of a modified red mud, of a storage medium, of a heat storage means and of uses of a modified red mud, and vice versa, unless explicitly stated otherwise.

20 If a term is designated with an indefinite or definite article, such as for example "a", "an" and "the", in the singular, this also includes the term in the plural, and vice versa, so long as the context does not specify otherwise unambiguously. The expression "comprise" or "have", such as is used here, includes not only the meaning of "contain" or "include", but can also mean "consist of" and "substantially consist of".

25

For the studies conducted within the context of the present invention, first of all the material to be studied was characterised at room temperature, and in particular the chemical as well as the mineralogical composition were determined. Furthermore, this material was heated slowly to 1000 °C heated, and in this case every 100 °C the mineralogical phases as well as
30 the density and the specific thermal capacity were determined.

The characterisation of the material to be studied:

1. Chemical composition (typical for bauxite residue)

- 10 to 50 % by weight of iron compounds
- 12 to 35 % by weight of aluminium compounds
- 5 • 5 to 17 % by weight of silicon compounds
- 2 to 10 % by weight of titanium dioxide
- 0.5 to 6 % by weight of calcium compounds
-

2. Mineralogical composition

10

In the initial state of the study the following mineral phases were determined radiographically:

- haematite
- 15 • goethite
- anatase
- rutile
- perovskite
- boehmite
- 20 • gibbsite
- cancrinite
- quartz

3. Particle sizes

25

The particle diameters (μm) are shown in Figure 1. According to this the substance is very fine and has 3 maxima. With a good distribution it was to be expected that the substance has a high density, since the very fine crystals can be inserted into cavities in the medium-fine crystals and these latter can be inserted into cavities in the coarser crystals. The
30 measured density of $3.63 \text{ (g/cm}^3\text{)}$ confirms this assessment.

4. Conduct of the test

Samples of the test substance were heated in stages under oxygen and under nitrogen up to

1,000 °C. Samples were taken in each case at 100 °C, 200 °C, 300 °C, 400 °C, 500 °C, 600 °C, 700 °C, 800 °C, 900 °C and 1000 °C and the changes to the mineralogical composition as well as the density were determined.

- 5 The specific thermal capacity was measured in the temperature range from room temperature (30.26 °C) to 584.20 °C.

5. Interpretation of the results

10 5.1 Mineral phases

The mineralogical composition of the substance changes in accordance with the temperature (see following Table 1).

- 15 At approximately 300 °C gibbsite decomposes, at approximately 400 °C goethite decomposes and at approximately 500 °C boehmite breaks down.

Above 600 °C the release of CO₂ from cancrinite Na₆Ca₂ [(AlSiO₄)₆ (CO₃)₂] takes place. A first maximum of the density occurs at 600 °C. Here the substance consists substantially of
 20 haematite (Fe₂O₃) and corundum (Al₂O₃) and, in smaller proportions, of TiO₂, cancrinite and perovskite.

At 1000 °C cancrinite and the two TiO₂ phases anatase and rutile are converted into the minerals pseudobrookite [(Fe³⁺)₂Ti]O₅ and nepheline [(Na,K)[AlSiO₄].

25

Table 1: Mineral phases bauxite residue (bulk density 0.944 g/cm³)

T[°C]	Mineral phases Bauxite residue GEA	Density <u>g</u>
100	haematite, goethite, anatase, rutile, perovskite, boehmite, gibbsite, cancrinite, quartz	3.63
200	haematite, goethite, anatase, rutile, perovskite, boehmite, gibbsite, cancrinite, quartz	3.64
300	haematite, goethite, anatase, rutile, perovskite, boehmite, cancrinite, quartz, α-Al ₂ O ₃	3.74
400	haematite, goethite, anatase, rutile, perovskite, boehmite, cancrinite, quartz, α-Al ₂ O ₃	3.81

500	haematite, anatase, rutile, perovskite, boehmite, cancrinite, quartz, α -Al ₂ O ₃	3.81
600	haematite, anatase, rutile, perovskite, cancrinite, quartz, α -Al ₂ O ₃	3.89
700	haematite, anatase, rutile, perovskite, cancrinite, quartz, α -Al ₂ O ₃	3.60
800	haematite, anatase, rutile, perovskite, cancrinite, quartz, α -Al ₂ O ₃	3.71
900	haematite, anatase, rutile, perovskite, cancrinite, quartz, α -Al ₂ O ₃	3.73
1000	haematite, anatase, rutile, perovskite, quartz, α -Al ₂ O ₃ , nepheline, pseudobrookite	3.93

5.2 Density

As can be seen from Figure 2, the density develops as a function of the temperature from
 5 3.63 (*g/cm³*) at 100 °C to 3.93 (*g/cm³*) at 1000 °C. The decomposition of mineral phases with
 elimination of water and CO₂ as well as sintering processes reduce the density between
 600 °C and 700 °C, in order then up to 1,000 °C to rise again to a value of 3.93 (*g/cm³*).

For applications in the thermal range it is only possible to use substances which are stable
 10 as bodies and which in the respective arbitrary temperature ranges do not eliminate any
 further gases such as H₂O or CO₂ and also do not undergo any further sintering processes.
 Oxides such as Fe₂O₃, Al₂O₃, TiO₂ or SiO₂ hardly change significantly in the event of
 temperature increases. A significant characteristic is revealed by the fact that the density of
 the substance heated to 1,000 °C remained constant during cooling constant remained, for
 15 example no rehydrations occurred.

5.3 Particle size

The tempering of the substance according to the invention up to temperatures of 1,000 °C
 20 shifts the particle diameters to significantly higher values for example by decomposition of
 hydroxides, oxide hydrates or carbonates and by sintering processes (cf. Figure 3). Thus for
 example

- the d₁₀ values grow from 0.74 μm to 1.341 μm
- the d₅₀ values grow from 0.261 μm to 6.743 μm
- 25 • the d₉₀ values grow from 1.692 μm to 28.17 μm

5.4 Specific thermal capacity

The specific thermal capacity of substances is a function of the temperature. As the temperature increases, the specific thermal capacity also increases. The following Table 2 shows corresponding examples.

5

Table 2: Specific thermal capacity at different temperatures:

		20 °C	726.8 °C
Fe₂O₃	(kJ/(kg * K))	0.650	0.944
Al₂O₃	(kJ/(kg * K))	0.775	1.223
SiO₂	(kJ/(kg * K))	0.732	1.148
TiO₂ rutile	(kJ/(kg * K))	0.689	0.937
TiO₂ anatase	(kJ/(kg * K))	0.691	0.943

In mixtures the specific thermal capacity is the sum of the specific thermal capacities of the respective components of the mixtures.

10

The characterisation of the substance provided shows a mixture of different mineral substances. During tempering, parts of the substances decompose, and for example eliminate water or CO₂ with formation of oxides or other chemically stable mineral phases. Furthermore, sintering processes take place.

15

The measurement results of these substances show a value of 0.791 (kJ/(kg K)) at 30 °C and a value of 1.037 at 584 °C. At 1,000 °C a value of 1.14 - 1.18 can be assumed by extrapolation (cf. Figure 4).

20 Table 3: Series of measurements of specific thermal capacity ALFERROCK®

Temperature of the sample [°C]	thermal capacity c _p ALFERROCK® [(kJ)/(kg * K)]
30.3 °C	0.791
50.1 °C	0.832
69.8 °C	0.858
89.6 °C	0.877
109.4 °C	0.89
129.2 °C	0.898
149.0 °C	0.907
168.8 °C	0.915

188.5 °C	0.922
208.3 °C	0.928
228.1 °C	0.935
247.9 °C	0.94
267.7 °C	0.948
287.5 °C	0.95
307.3 °C	0.96
327.0 °C	0.963
346.8 °C	0.969
366.6 °C	0.977
386.4 °C	0.985
406.2 °C	0.989
426.0 °C	0.999
445.7 °C	1.002
465.5 °C	1.01
485.3 °C	1.017
505.1 °C	1.02
524.9 °C	1.026
544.6 °C	1.031
564.2 °C	1.035
584.2 °C	1.037

During cooling of the substance heated to 1,000 °C the c_p values revert to values which correspond to the respective temperatures. However, since the starting substance has changed due to decompositions of mineral phases and the formation of other substances and also sintering processes have taken place, after the cooling the substance will have different values of the specific thermal capacity from the starting substance. It is important to establish that after the tempering a stable substance is available, which can be heated and cooled with any frequency and which in this case does not undergo any further change to the individual substances in the mixture. As already mentioned, this applies for the density.

5.5 Specific thermal conductivity

The thermal conductivity of a system is *inter alia* a function of parameters such as pressure, temperature, mineralogical composition, porosity, density, etc.

As described, all thermally unstable components have been decomposed by heating of the substance provided. After the tempering a substance occurs which consists of corundum (Al_2O_3), haematite (Fe_2O_3), rutile and anatase (TiO_2) as well as fireproof substances such as pseudobrookite [$(Fe^{3+})_2Ti$]O₅ or nepheline [(Na,K)[AlSiO₄].

The following Table 4 shows the values of the thermal conductivity and density of those

substances which constitute the most important components of the tempered substance.

Table 4: Thermal conductivity and density of the individual components

	Thermal conductivity	Density
Anatase TiO₂	4.8 -11.8 (W/(m · k))	3.89 (g/cm ³)
Rutile TiO₂	4.8 -11.8 (W/(m · k))	4.25 (g/cm ³)
Haematite (Fe₂O₃),	6 (W/(m · k))	5.26 (g/cm ³)
Corundum (Al₂O₃),	3.0-35 (W/(m · k))	3.99 (g/cm ³)
Quartz (SiO₂),	18,37 (W/(m · k))	2.65 (g/cm ³)

- 5 During the tempering process the particle diameter of the substances produced has increased significantly and in this case the surface area is decreased. Thus within the primary crystals the conductivity also increased to values which are set out in Table 4.

Air is still contained in the substance mixture and as a poor conductor of heat it lowers the measured thermal conductivity. In order to avoid this effect, different methods are possible, including for example application of pressure, i.e. pressing the substance to form solid bodies.

15 Furthermore, substances can be added, which prevent air inclusions between the crystallites or on the surface of the crystallites and thus enable the production of solid substance blocks.

These include for example:

- metal colloids
- 20 • metal powder
- graphite
- sinterable pyrolysing substances based on Si

As well as the addition of the said substances, pressure and thermal energy can additionally be used.

5 It is crucial to be able to produce good heat conducting substance blocks. For the use of the substance obtained after the tempering as a heat storage means, a good thermal conductivity, in particular the prevention of air inclusions, is significant for the charging operation (heating of the substance) and for the discharging operation (transfer of the stored heat to systems which generate steam).

10 Example:

A mixture of the provided substance consisting of untempered substance and substance tempered up to 1,000 °C in the ratio of 1:1 is surface-modified with 5 % PDMS (polydimethylsiloxane prepolymer) and is introduced into a BUSS kneader or a co-rotating double screw extruder. The compounding machine has a housing temperature of 135 °C and maximum vacuum degassing. The torque is set to 65 - 85 % of the maximum. The material is removed by means of a cooling conveyor.

20 The resulting water-free and air-free product is introduced into the insulated container and is mechanically compressed. Then the temperature is slow increased to 1,000 °C and thus the heat storage means is made ready for operation. Instead of PDMS, other substances can be used, such as metal dusts, graphite or salt solutions.

6. Summary

25

During tempering up to temperatures of 1,000 °C all components within the substance mixture which are unstable in this temperature range decomposed. These include gibbsite, goethite, boehmite as well as cancrinite and the TiO₂ phases which, where applicable, form pseudobrookite [(Fe³⁺)₂Ti]O₅ and nepheline [(Na,K) (AlSiO₄)] at 1,000 °C.

30

After the cooling, a substance mixture was formed, consisting of oxides such as Al₂O₃, Fe₂O₃, TiO₂, SiO₂ and optionally substances which are resistant to high temperatures, such as pseudobrookite and nepheline, which did not show any further change after renewed tempering to 1,000 °C.

35

With the aforementioned change to the material composition the density also changed from 3.63 (g/cm^3) at room temperature to 3.93 (g/cm^3) at 1,000 °C. This expected operation was additionally accompanied by sintering effects. During cooling of the substance mixture tempered to 1,000 °C, the density reached at 1,000 °C remains unchanged, since the
 5 density of oxides such as Al_2O_3 , Fe_2O_3 as well as TiO_2 and SiO_2 does not change in the temperature ranges between 25 °C and 1,000 °C.

These sintering effects and the decomposition of mineral phases have led to an increase in the particle diameter in the substance mixture. Whereas before the tempering for example
 10 $d_{50} = 0.261 \mu m$ and $d_{90} = 1.692 \mu m$ applied, after the tempering the following values could be measured: $d_{50} = 6.743 \mu m$ and $d_{90} = 28.17 \mu m$. The enlargement of the particles means a reduction of the surface and a better thermal conductivity. The air content (poor conductor of heat) between the very small crystallites was reduced.

15 The study of the specific thermal capacity of the characterised substance showed an increase in the specific thermal capacity of 0.79 ($kJ/(kg K)$) at 25 °C to 1.037 ($kJ/(kg K)$) at 600 °C. At 1,000 °C a value of 1.14-1.18 ($kJ/(kg K)$) is to be expected by extrapolation.

Since, as already stated, the density has also increased, the product of the density and the
 20 specific thermal capacity as a crucial criterion for applications as heat storage means reaches values higher than that of water. Water has a density at 20 °C of 998.2 (kg/m^3) and an outstanding specific thermal capacity of 4.182 ($kJ/(kg K)$). This results in a volumetric thermal capacity of 4175 ($kJ/m^3 K$). On the other hand, the provided substance has a density of 3890 (kg/m^3) and a specific thermal capacity of 1.037 ($kJ/kg K$) and thus a
 25 volumetric thermal capacity of 4.034 ($kJ/(m^3 K)$) at approximately 600 °C. At 1000 °C values for the density of 3,930 (kg/m^3) and a c_p von 1.16 ($kJ/(kg K)$) are produced. Thus the volumetric thermal capacity reaches a value of 4.559 ($kJ/(m^3 K)$). This value significantly exceeds the value of water.

30 A substantial difference between water and the specified substance is the temperature at which the storage media can operate. Whilst water ideally operates in temperature ranges between 40 °C and 90 °C, that is to say it has a ΔT of 50 °C, the provided substance can operate in the temperature range up to 1,000 °C, i.e. the substance can evaporate water above a temperature of 100 °C and thus can operate with a ΔT of 900 °C. For this reason
 35 the provided substance can store 15 - 20 times as much heat by comparison with water.

In storage media the coefficient of thermal conductivity is more important for the charging operation (heating up of the storage device) than for the discharging operation. The thermal conductivity of the oxides significantly contained in the substance is between 3 and 35 ($W/(m$
 5 $K)$). What is crucial for heat storage means is the necessity of being able to compact the substance used as storage medium to form solid blocks in which the thermal power can flow optimally, i.e. from the heating element into the storage substance, within the storage substance and from the storage substance into the systems consuming thermal energy. In
 10 this respect it is advantageous if poorly heat-conducting gases within the substance or on the surface of the substance are eliminated. In addition to applications of pressure substances can be added by which the primary crystals are "stuck together". These include, for example, metal colloids, metal powder, graphite, sinterable pyrolysing substances containing Si. Above all, it is also crucial that in the tempering process of the provided
 15 substance up to 1,000 °C all unstable substances are decomposed and so a predominantly oxidic, thermally stable storage substance is made available which can be heated and cooled with any frequency without generating gases such as H₂O or CO₂ which can destroy the storage block.

Use of the provided substance as storage material for high-temperature-heat storage
 20 **means**

The storage system

Both water and also solid substances - for example the previously described substance -
 25 belong to the sensitive heat storage systems (sensitive, because the heat of the storage device is perceptible).

The heat storage means can be heated by means of force/heat coupling by electrical power from wind farms or solar installations. In still air or in darkness to be able these heat storage
 30 means generate steam which drives turbines which in turn generate electrical power (heat/force coupling). Thus the heat storage means takes on the role of emergency generator or, on a large scale, of "replacement power plants". If this process is successful, the power line systems can also be simply and effectively designed.

35 The requirements for energy accumulators are set out below:

- High energy density
- High power density
- Low cumulative energy consumption
- 5 • Low losses
- Low self-discharge
- Long cycle life
- Long service life
- Low investment costs
- 10 • Low operating costs

The provided substance meets the set requirements to a large extent.

The substance is

- 15 • inorganic
- safe
- with a long service life
- recyclable
- available in very large quantities
- 20 • highly economical
- operates in the temperature range up to 1,000 °C
- can be simultaneously charged and discharged
- can be simply manufactured.

25 In particular the fact that, as a sensitive high-temperature storage means, the provided substance can be simultaneously charged and discharged makes it possible to operate a controllable, permanently running storage power plant. In this way power generation deficiencies can be compensated for or higher demands can be met.

30 Furthermore heat storage means can be used in particular for wind farms or solar parks and thus render the power generated there capable of providing base load power as a "package solution".

Furthermore, small heat storage units can be used for example for a complete power supply

for example for residential buildings. These small units are heated for example by renewable energy sources and are then used as a routine replacement for the complete power supply, i.e. supply of thermal power and electricity, for residential buildings.

- 5 Furthermore, small heat storage units in machines of all types can be used for the purpose of power supply.

Furthermore, after cogeneration, electrical power can be made "transportable" in the form of heat storage means without line systems.

10

Vehicles can also be powered in this way. After cogeneration has taken place, heat storage means which are regularly replaced like batteries can operate electric motors, comparably to lithium batteries.

- 15 The equipment for the conversion of heat into electrical power can be provided as an integral component of the storage device take place or in units which are independent thereof.

Example

- 20 The provided substance is a filter cake which first of all must be subjected to a thermal treatment, i.e. it must be heated slowly up to 1,000 °C. In this case first of all the water content of the filter cake is evaporated, then up to 1,000 °C all minerals which are unstable in the high-temperature range are calcined. Then the substance consists only of oxides as well as stable inorganic phases such as nepheline or others. This substance is cooled and
25 forms the storage mass.

- The charging (i.e. heating) of the storage mass takes place directly by means of embedded resistance wires or heating elements, i.e. resistance wires in ceramic sleeves or other systems. By means of corresponding control devices the storage mass can be constantly
30 adjusted to arbitrary temperatures.

- The discharging takes place by means of a water circulation which passes through the storage mass at a suitable and optimal point of the temperature range/steam pressure. Water is evaporated, steam drives turbines, current is generated. The excess steam is
35 guided back again into the water circulation by means of cooling equipment ("cooling

tower"). Optimal conditions can be set by means of the specific thermal conduction of the heat storage medium between the delivery of heat (hottest point) and the heat consumption.

5 The heat storage material is consolidated with heating means for the supply of heat and the pipe system (water) for the heat dissipation to form a block. This block is thermally insulated against the exterior.

10 The statement that the heat storage system characterised in this way can be simultaneously charged and discharged is crucial. As a rule storage facilities are designed so that either charging or discharging takes place; cf. in this connection pumped storage facilities. On the other hand, with the possibility of the simultaneous charging by renewable energy sources and the discharging it is possible to construct stable storage power plants which are capable of providing base load power.

15 Currently the most important heat storage system for sensitive heat storage means is water. This system is characterised in that it operates with water ideally in a temperature range from 40 ° - 90 ° C, since above 100 °C water is present as steam. Thus water has a ΔT von 50 °C.

20 In contrast to this, the heat storage system which operates with a storage mass which has been produced from the provided substance can operate at temperatures up to 1,000 °C, i.e. the substance can evaporate water above a temperature of 100 °C and thus can operate with a ΔT of 900 °C. Thus this system is a high-temperature storage system.

25 Example:

Comparison of the sensitive water/ ALFERROCK® heat storage means

Calculation of the amount of heat which can be stored

30

The amount of heat Q which a storage material can store is calculated according to the following equation:

$$Q = m * c_p * \Delta T = \rho * c_p * V * \Delta T [J]$$

35

m = mass [kg]

c_p = specific thermal capacity [$\frac{kJ}{m^3 K}$]

5 ρ = Dichte [$\frac{kg}{m^3}$]

V = volume [m^3]

$\rho * c_p$ = volumetric thermal capacitance [$\frac{kJ}{m^3 K}$]

ΔT = temperature range [K]

10

$Q_{(1m^3)}$ = volumetric thermal capacitance * ΔT [J]

1. Water (for 1 m³)

15 $\rho = 998.2 \frac{kg}{m^3}$

$C_p = 4.182 \frac{kJ}{m^3 K}$

$\rho * C_p = 4.175 \frac{kJ}{m^3 K}$

$\Delta T = 50 K$

20 $Q = 4.175 \frac{kJ}{m^3 K} * 50 K * 1 m^3$

$Q = 208.7 * 10^3 kJ$

Converted into Wh:

25

$1 J = \frac{1 Wh}{3600}$

$Q_{water} = 57.88 kWh$

30 2. ALFERROCK® (for 1 m³)

$\rho = 3.930 \frac{kg}{m^3}$

$$C_p = 1.16$$

$$\rho * C_p = 4,558.8 \frac{\text{kJ}}{\text{m}^3 \text{K}}$$

$$\Delta T = 900 \text{ K}$$

$$5 \quad Q = 4,558.8 \frac{\text{kJ}}{\text{m}^3 \text{K}} * 900 \text{ K} * 1 \text{ m}^3$$

$$Q = 4,102.9 * 10^3 \text{ kJ/m}^3$$

10

Converted into Wh:

$$1 \text{ J} = \frac{1 \text{ Wh}}{3600}$$

$$Q_{\text{ALFERROCK}} = 1.1397 \text{ MWh}$$

15

3. Comparison ALFERROCK®/water.

$$\frac{Q_{\text{ALFERROCK}}}{q_{\text{water}}} = \frac{1.1397 \text{ MWh}}{57.88 \text{ kWh}} = 19.7$$

20 ALFERROCK® can store 19.7 times the amount of heat at an operating temperature up to 1,000 °C.

The ALFERROCK® high-temperature-heat storage medium can also be used in an outstanding manner at lower temperatures as a heat storage means, heat exchanger and thermostat. It is worthy of note that during tempering of the provided substance the increase in the density from 3.63 $\frac{\text{g}}{\text{cm}^3}$ at 100 °C to 3.93 $\frac{\text{g}}{\text{cm}^3}$ at 1,000 °C does not decline, but remains constant at 3.93 $\frac{\text{g}}{\text{cm}^3}$. Thus the value $\rho * c_p$ is increased by 9%.

30 In the following Table 5 the storable quantities of heat in the region of approximately 200 °C, 300 °C, 400 °C, 500 °C and 600 °C are set out and present very attractive values.

Table 5: Requirements for energy accumulators

Temperature [° C]	c_p <u>kJ</u>	Density ρ [kg/m ³]	$\rho * c_p * V$ [10 ⁶ $\frac{J}{K}$]	ΔT [K]	Q (for 1 m ³) [J]
208.32	0.928	3930	3.65	100	365 * 10 ⁶
307.26	0.960	3930	3.77	200	754 * 10 ⁶
406.17	0.989	3930	3.88	300	1.16 * 10 ⁹
505.07	1.020	3930	4.01	400	1.60 * 10 ⁹
584.20	1.037	3930	4.08	500	2.04 * 10 ⁹

I Claim:

1. Modified red mud, which contains the following components:
 - 5 - 48 to 55 % by weight of haematite (Fe_2O_3),
 - 13 to 18 % by weight of corundum (Al_2O_3),
 - 8 to 12 % by weight of rutile (TiO_2) and/or anatase (TiO_2),
 - 2 to 5 % by weight of quartz (SiO_2).

- 10 2. Modified red mud according to claim 1, wherein the modified red mud has a density in the range from 3.90 to 4.0 g/cm³.

3. Modified red mud according to claim 2, wherein the modified red mud has a density of 3.93 g/cm³.

- 15 4. Modified red mud obtained by heating of red mud having a mineral composition of
 - 10 to 55 % by weight of iron compounds,
 - 12 to 35 % by weight of aluminium compounds,
 - 3 to 17 % by weight of silicon compounds,
 - 20 - 2 to 12 % by weight of titanium dioxide,
 - 0.5 to 6 % by weight of calcium compounds, and
 - further unavoidable impurities,to a temperature of at least 1000°C,
characterised in that the modified red mud has a density in the range from 3.90 to 4.0 g/cm³.

- 25 5. Modified red mud according to any one of claims 1 to 4, wherein the modified red mud has a mean particle size d₅₀ in the range from 3 to 10 µm.

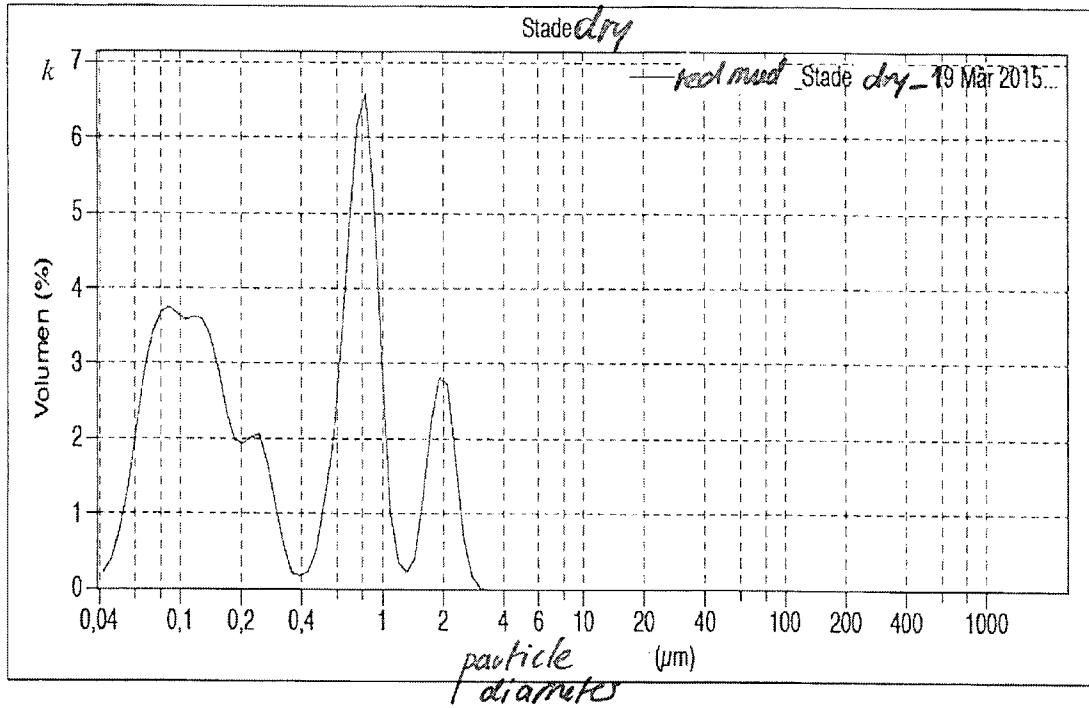
6. Modified red mud according to any one of claims 1 to 5, wherein the modified red
30 mud has a particle size d₁₀ in the range from 0.5 to 2.5 µm and/or a particle size d₉₀ in the range from 15 to 50 µm.

7. Modified red mud according to any one of claims 1 to 6, wherein the modified red
mud has a specific thermal capacity at 20 °C in the range from 0.6 to 0.8 kJ/(kg*K) and/or a
35 specific thermal capacity at 726.8 °C in the range from 0.9 to 1.3 kJ/(kg*K).

8. Modified red mud according to any one of claims 1 to 7, wherein the modified red mud has a specific thermal conductivity in the range from 3 to 35 W/(m*K).
- 5 9. Modified red mud according to any one of claims 1 to 8, wherein the modified red mud is present in the form of a compressed solid material.
10. Method for production of a modified red mud, comprising heating of red mud with a mineral composition of
- 10 - 10 to 55 % by weight of iron compounds,
- 12 to 35 % by weight of aluminium compounds,
- 3 to 17 % by weight of silicon compounds,
- 2 to 12 % by weight of titanium dioxide,
- 0.5 to 6 % by weight of calcium compounds, and
- 15 - further unavoidable impurities,
to a temperature of at least 800 °C in an oxygen (O₂) atmosphere, and compressing the red mud after the heating.
11. Method for production of a modified red mud according to claim 10, wherein the
20 modified red mud is modified red mud according to any one of claims 1 to 9.
12. Storage medium comprising a modified red mud according to one of claims 1 to 9.
13. Storage medium according to claim 12, further comprising one or more of the
25 following components:
- an agent for preventing inclusion of air and air adsorption,
- an agent for improvement of the thermal conductivity, in particular selected from the group consisting of metal colloids, metal powder, graphite and substances containing silicon,
- an agent for formation of a thixotropic composition.
- 30
14. Heat storage means, comprising a storage medium according to claim 12 or 13.
15. Heat storage means according to claim 14, further comprising devices for charging
and discharging the heat storage means.
- 35

16. Heat storage means according to claim 14 or 15, further comprising resistance wires.
17. Use of a modified red mud according to one of claims 1 to 9 as a storage medium.
- 5 18. Use according to claim 17 for storage of heat at a temperature up to 1000 °C.
19. Use according to claim 17 or 18 as a storage medium which can be repeatedly heated and cooled.
- 10 20. Use according to claim 17 or 18 as a storage medium which can be simultaneously heated and cooled.
21. Use according to one of claims 17 to 20 as a storage medium in a current/heat store.
- 15 22. Use according to claim 21, wherein the storage medium is heated by means of electrical current and/or cooled while electrical current is generated.
23. Use according to claim 21 or 22 for storage of electrical power obtained from renewable energy sources.
- 20 24. Use according to one of claims 21 to 23, wherein the current/heat store comprises resistance wires which are heated by electrical current and thus heat the storage medium.
- 25 25. Use according to one of claims 18 to 24, wherein thermal power stored in the storage medium is transferred to another medium and so the storage medium is cooled.
26. Use according to one of claims 17 to 25, wherein the storage medium and a discharge device are provided integrally or as separate elements.
- 30 27. Use according to one of claims 21 to 26, wherein the current/heat store is used for transport of electrical power after cogeneration has taken place without the presence of line systems.
- 35 28. Use according to one of claims 21 to 27, wherein the current/heat store generates electrical current again after cogeneration.

29. Use according to one of claims 21 to 28, wherein the current/heat store is used for supplying power to insulated energy consumers.
- 5 30. Use according to claim 29, wherein insulated energy consumers are supplied with thermal energy and electrical energy.
31. Use according to one of claims 21 to 28, wherein the current/heat store is used for supplying power to machines or mobile devices such as vehicles.



Volume statistics (Arithmetic) Rotschlamm_Stade trocken_19 Mär 2015_11.\$ts

calculation from 0,040 µm bis 2000 µm

Volume.: 100%

average value: 0,580 µm S.D.: 0,616 µm

medium value: 0,261 µm Var.: 0,379 µm²

average/medium value: 2,220 C.V.: 106%

max.: 0,829 µm Tilt: 1,429 right tilt

Kurtosis: 1,337 Leptokurtic

d ₁₀ : 0,074 µm	d ₅₀ : 0,261 µm	d ₉₀ : 1,692 µm		
<10%	<25%	<50%	<75%	<90%
0,074 µm	0,109 µm	0,261 µm	0,831 µm	1,692 µm
<1 µm	<10 µm	<100 µm	<200 µm	<500 µm
84,9%	100%	100%	100%	100%

Fig. 1

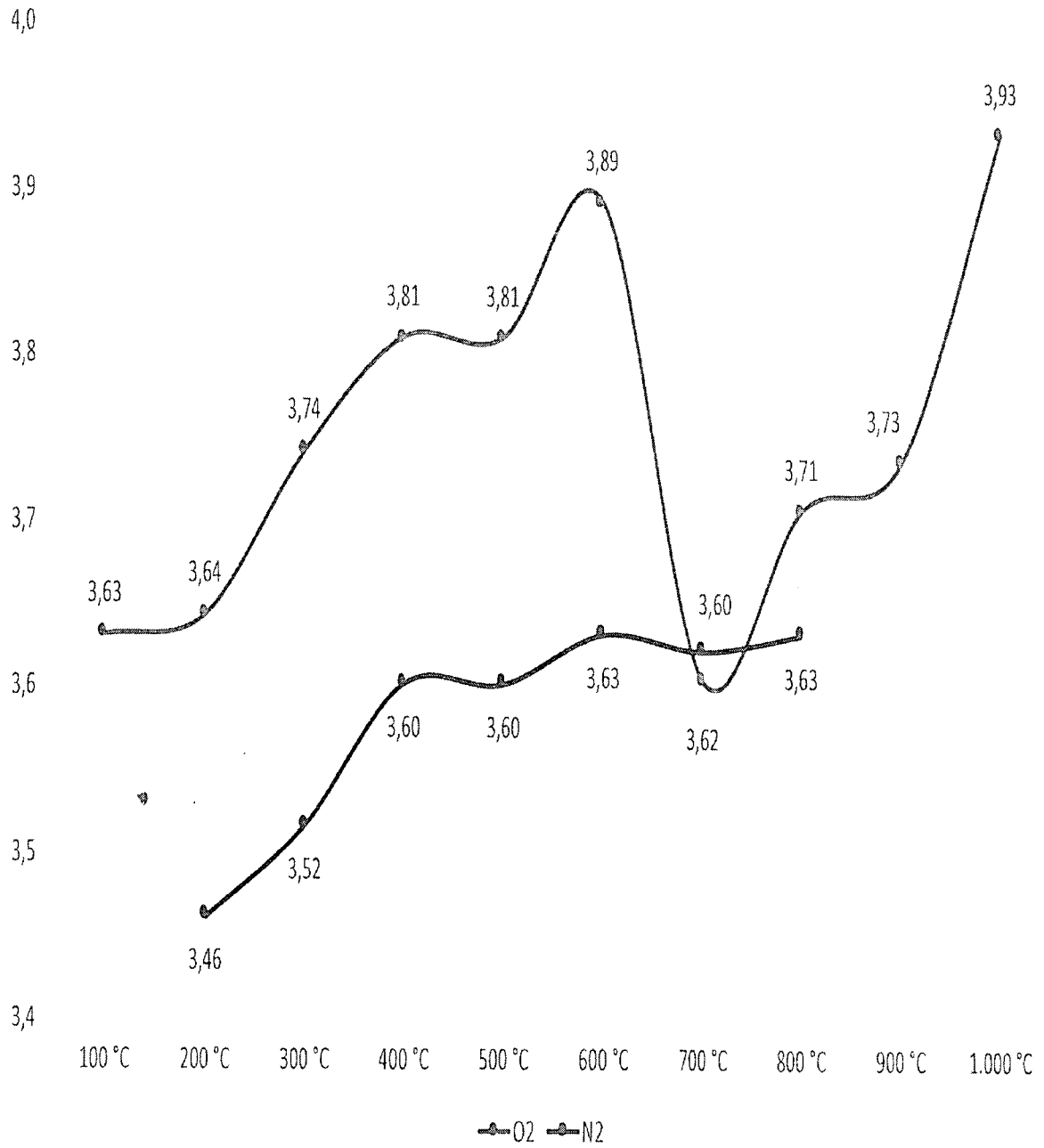
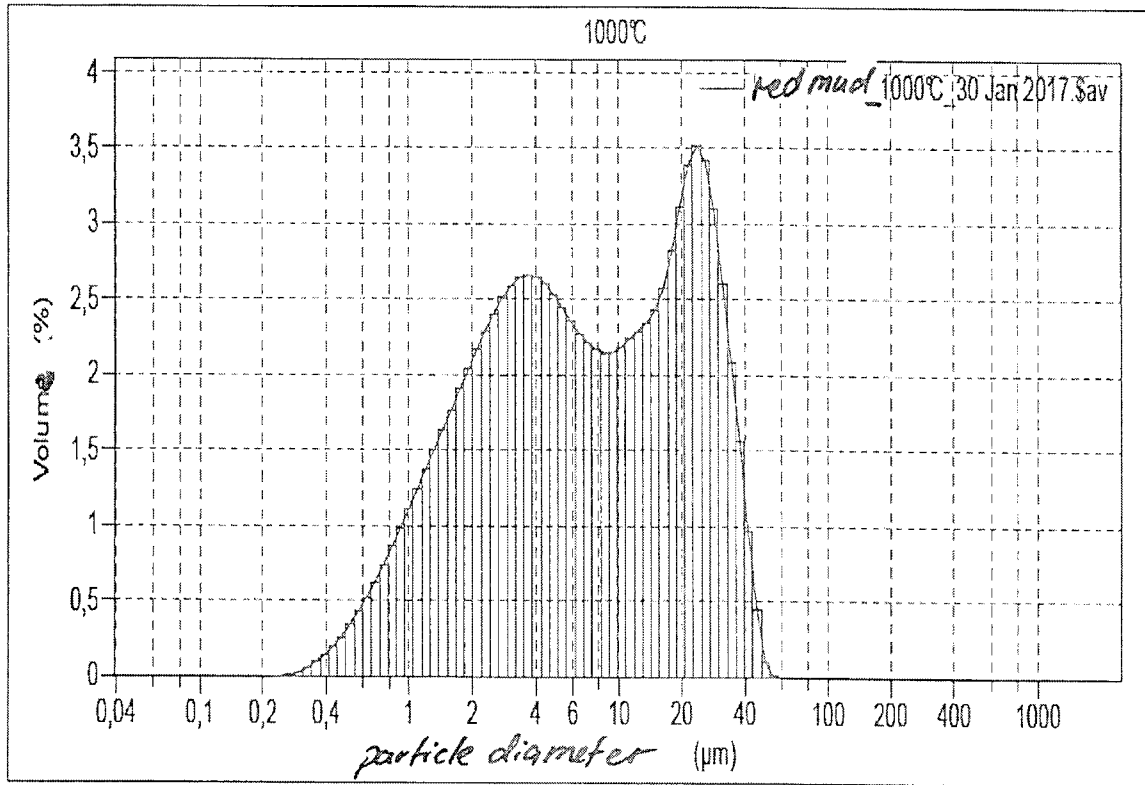


Fig. 2



Volume Statistics (Arithmetic) *red mud_1000°C_30 Jan 2017.sav*

calculated from 0,040 µm bis 2000 µm

Volume :	100%	S.D.:	10,82 µm
average value:	11,35 µm	Var.:	117,1 µm ²
medium value:	6,743 µm	C.V.:	95,3%
average/median value:	1,683	Tilt	1,083 <i>right tilt</i>
Max:	23,81 µm	Kurtosis:	0,248 <i>Leptokurtic</i>

d ₁₀ :	1,341 µm	d ₅₀ :	6,743 µm	d ₉₀ :	28,17 µm
<10%	1,341 µm	<25%	2,685 µm	<50%	6,743 µm
		<75%	18,47 µm	<90%	28,17 µm
<1 µm	5,86%	<10 µm	59,2%	<100 µm	100%
		<200 µm	100%	<500 µm	100%

Fig. 3

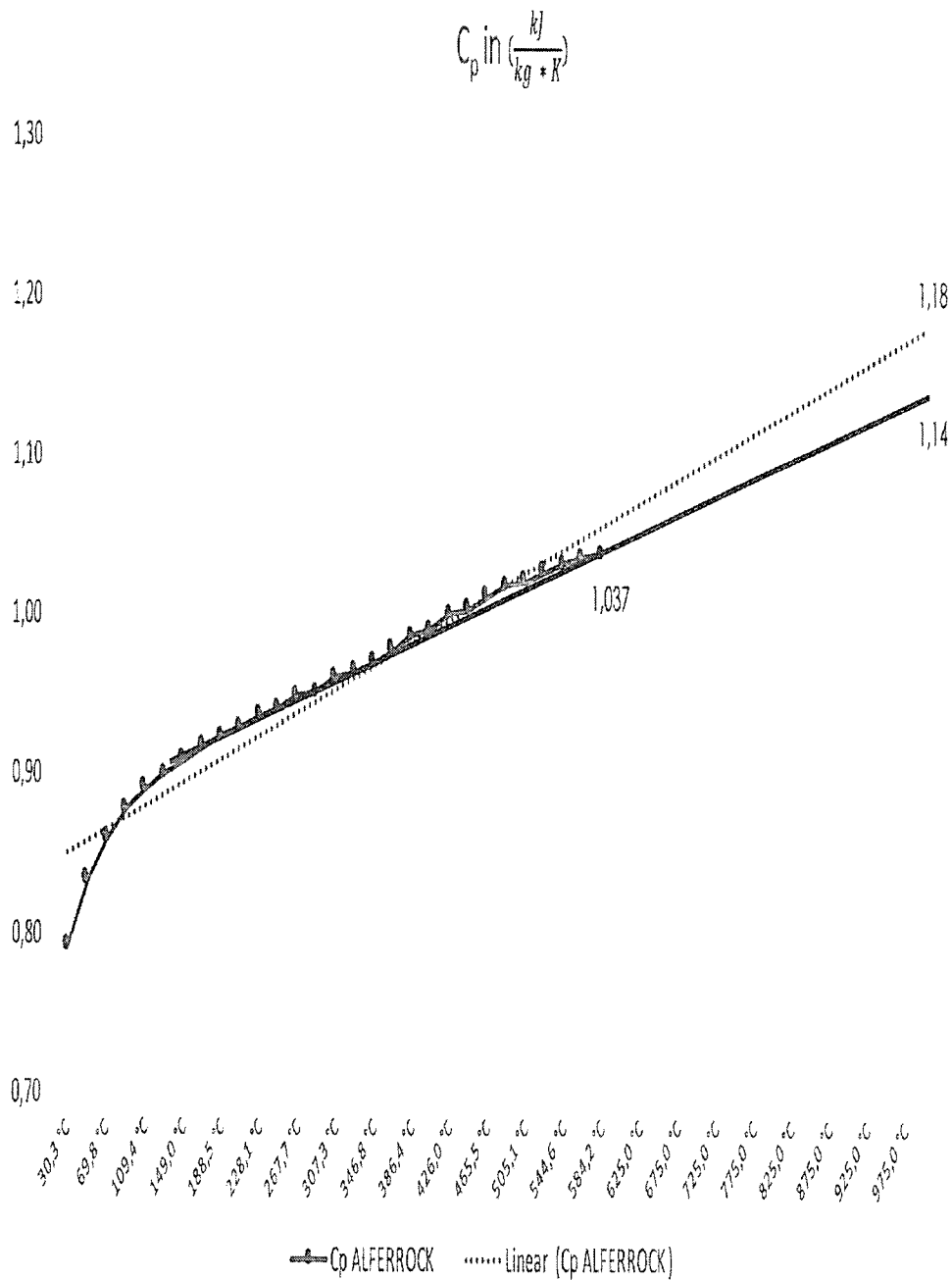


Fig. 4