Data Article

Dataset of multiple methodology characterization of an illite-kaolinite clay mineral for the purpose of using it as ceramic membrane supports

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ARTICLE INFO

Article history:
Received 5 January 2020
Received in revised form 21 January 2020
Accepted 10 February 2020
Available online 18 February 2020

Keywords:
Membrane supports
Methylene blue test
Clay minerals
Ultrafiltration
Pores structures
Desalination

ABSTRACT

This article describes the data generated from multiple approach methodology physico-chemical characterization of a clay mineral from the West-Central region of Morocco, Safi province (https://doi.org/10.1016/j.heliyon.2019.e02281) [1]. Data were generated from classical chemical analytical techniques namely; organic matter content, linear firing and shrinkage analysis, weight loss on ignition, porosity and methylene blue stain tests according to the French Association of Normalization (AFNOR) and American Society for Testing and Materials (ASTM). In addition to data generated using instrumental analytical techniques namely; Infrared spectroscopy (FTIR), thermal gravimetric analysis (TGA) and deferential thermal analysis (DTA), X-ray diffraction (XRD), scanning electron microscopy (SEM) and elemental energy disperse spectroscopy (EDX).

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DOI of original article: https://doi.org/10.1016/j.heliyon.2019.e02281.
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The data reveal new ways in which largely available clay minerals could be used to develop sustainable and cheap clay materials. These data gave a detailed and complete set of experiments that could be used in the characterization of widely available clay fractions (illite and kaolinite) using the blue stain test according to AFNOR and ASTM.

The data would allow other researchers to identify the key parameters that need to be controlled when investigating new clay material. These data gave a detailed and complete set of experiments that could be used in the characterization of widely available clay fractions (illite and kaolinite) using the blue stain test according to AFNOR and ASTM. These data are relevant to ceramic membrane support fabrication, especially fabrication of membrane supports from clay minerals.

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Value of the data

- These data are relevant to ceramic membrane support fabrication, especially fabrication of membrane supports from clay minerals.
- The data would allow other researchers to identify the key parameters that need to be controlled when investigating new clay material.
- These data gave a detailed and complete set of experiments that could be used in the characterization of widely available clay fractions (illite and kaolinite) using the blue stain test according to AFNOR and ASTM.
- The data reveal new ways in which largely available clay minerals could be used to develop sustainable and cheap clay supports.

1. Data

Successive weight loss on ignition for both clay powder samples (SA) and (CH) at final temperatures of 250 °C, 500 °C, 700 °C, 800 °C, 900 °C and 1000 °C are presented in Table 1. While, data for the weight loss of compacted clay discs at final temperatures of 500 °C, 700 °C, 800 °C, 850 °C, 900 °C, 950 °C, 1000 °C, 1050 °C and 1100 °C are presented in Table 2. The shrinkage analysis data for the compacted flat discs (SA) and (CH) at final temperature of 500 °C, 700 °C, 800 °C, 850 °C, 900 °C, 950 °C, 1000 °C, 1050 °C and 1100 °C are presented in Table 3. The data for water porosity of the fabricated clay supports calcined at final temperatures of 500 °C, 700 °C, 800 °C, 900 °C and 1000 °C are presented in Table 4. The data of chemical resistance is presented as weight loss fraction (Δm/m₀) of SA and CH compacted flat discs calcined to 850 °C and treated with HCl at pH = 5 and NaOH at pH = 10.0. The elemental chemical composition of crude and calcined clays SA and CH at 850 °C and 950 °C are given in Tables.
### Table 1
Successive loss on ignition for SA and CH clays powders calcined from 25 °C to final temperatures of 250 °C, 500 °C, 700 °C, 800 °C, 900 °C and 1000 °C at rate of 5 °C/min.

|          | SA (g)          | CH (g)          | Loss difference (SA) | Loss difference (CH) |
|----------|-----------------|-----------------|----------------------|----------------------|
| Calcined to 250 °C | 4.12 ± 0.19  | 3.74 ± 0.27    | 4.12 ± 0.19          | 3.74 ± 0.27          |
| Calcined to 500 °C | 5.23 ± 0.09  | 5.05 ± 0.13    | 1.11 ± 0.21          | 1.31 ± 0.30          |
| Calcined to 700 °C | 11.26 ± 0.09  | 11.17 ± 0.13   | 0.60 ± 0.13          | 6.12 ± 0.18          |
| Calcined to 800 °C | 11.52 ± 0.06  | 11.33 ± 0.07   | 0.26 ± 0.11          | 0.16 ± 0.14          |
| Calcined to 900 °C | 12.38 ± 0.05  | 12.28 ± 0.07   | 0.87 ± 0.08          | 0.95 ± 0.10          |
| Calcined to 1000 °C| 12.69 ± 0.07  | 12.60 ± 0.07   | 0.31 ± 0.09          | 0.31 ± 0.10          |

### Table 2
Loss on ignition for SA and CH clays compacted flat discs calcined from 25 °C to final temperatures of 500 °C, 700 °C, 800 °C, 850 °C, 900 °C, 950 °C, 1000 °C, 1050 °C and 1100 °C at rate of 5 °C/min.

|          | SA (g)          | CH (g)          | Δm/m₀ (CH) |
|----------|-----------------|-----------------|------------|
| Before Calcination | 3.0900 ± 0.0001 | 3.0000 ± 0.0001 | –          |
| Calcined to 500 °C | 2.8800 ± 0.0001 | 2.7400 ± 0.0001 | 8.83 ± 0.10 |
| Calcined to 700 °C | 2.6600 ± 0.0001 | 2.5300 ± 0.0001 | 15.65 ± 0.09 |
| Calcined to 800 °C | 2.6700 ± 0.0001 | 2.5400 ± 0.0001 | 15.59 ± 0.09 |
| Calcined to 850 °C | 2.6400 ± 0.0001 | 2.5100 ± 0.0001 | 16.38 ± 0.08 |
| Calcined to 900 °C | 2.6400 ± 0.0001 | 2.5100 ± 0.0001 | 16.41 ± 0.08 |
| Calcined to 950 °C | 2.6400 ± 0.0001 | 2.5100 ± 0.0001 | 16.53 ± 0.08 |
| Calcined to 1000 °C| 2.6300 ± 0.0001 | 2.5000 ± 0.0001 | 16.61 ± 0.08 |
| Calcined to 1050 °C| 2.6300 ± 0.0001 | 2.5000 ± 0.0001 | 16.65 ± 0.08 |
| Calcined to 1100 °C| 2.6300 ± 0.0001 | 2.5000 ± 0.0001 | 16.81 ± 0.08 |

### Table 3
Shrinkage on ignition for SA and CH clays compacted flat discs calcined from 25 °C to final temperatures of 500 °C, 700 °C, 800 °C, 850 °C, 900 °C, 950 °C, 1000 °C, 1050 °C and 1100 °C at rate of 5 °C/min.

|          | SA (mm)          | CH (mm)          | ΔL/Lo (SA) | ΔL/Lo (CH) |
|----------|-----------------|-----------------|------------|------------|
| Before Calcination | 30.42 ± 0.02  | 30.42 ± 0.02    | –          | –          |
| Calcined to 500 °C | 30.41 ± 0.02  | 30.39 ± 0.02    | 0.03 ± 0.01 | 0.10 ± 0.01 |
| Calcined to 700 °C | 30.40 ± 0.02  | 30.37 ± 0.02    | 0.07 ± 0.02 | 0.16 ± 0.01 |
| Calcined to 800 °C | 30.38 ± 0.02  | 30.35 ± 0.02    | 0.13 ± 0.02 | 0.23 ± 0.01 |
| Calcined to 850 °C | 30.16 ± 0.02  | 30.14 ± 0.02    | 0.85 ± 0.05 | 0.92 ± 0.08 |
| Calcined to 900 °C | 29.92 ± 0.02  | 30.00 ± 0.02    | 1.64 ± 0.06 | 1.38 ± 0.08 |
| Calcined to 950 °C | 29.76 ± 0.02  | 29.86 ± 0.02    | 2.17 ± 0.06 | 1.84 ± 0.08 |
| Calcined to 1000 °C| 29.72 ± 0.02  | 29.80 ± 0.02    | 2.30 ± 0.08 | 2.04 ± 0.07 |
| Calcined to 1050 °C| 29.70 ± 0.02  | 29.78 ± 0.02    | 2.37 ± 0.02 | 2.10 ± 0.06 |
| Calcined to 1100 °C| 29.42 ± 0.02  | 29.49 ± 0.02    | 3.29 ± 0.01 | 3.06 ± 0.08 |

### Table 4
Water porosity for SA and CH clays compacted flat discs calcined from at final temperatures of 500 °C, 700 °C, 800 °C, 900 °C, and 1000 °C at rate of 5 °C/min.

|          | SA (g) before immersing in water | CH (g) before immersing in water | SA (g) after drying | CH (g) after drying | SA-Porosity (%) | CH-Porosity (%) |
|----------|---------------------------------|---------------------------------|---------------------|---------------------|-----------------|-----------------|
| Before Calcination | 3.0033 ± 0.0001 | 3.0894 ± 0.0001    | –                   | –                   | –               | –               |
| Calcined to 500 °C | 2.7382 ± 0.0001 | 2.8804 ± 0.0001    | 3.2985 ± 0.0001    | 2.7820 ± 0.0001    | 20.12 ± 0.48    | 18.23 ± 0.47    |
| Calcined to 700 °C | 2.5333 ± 0.0001 | 2.6649 ± 0.0001    | 3.0620 ± 0.0001    | 2.5717 ± 0.0001    | 23.46 ± 0.66    | 21.61 ± 0.60    |
| Calcined to 800 °C | 2.5351 ± 0.0001 | 2.6655 ± 0.0001    | 3.0156 ± 0.0001    | 2.5714 ± 0.0001    | 18.39 ± 0.80    | 16.72 ± 0.79    |
| Calcined to 900 °C | 2.5109 ± 0.0001 | 2.6428 ± 0.0001    | 2.9462 ± 0.0001    | 2.5433 ± 0.0001    | 17.58 ± 0.32    | 16.06 ± 0.31    |
| Calcined to 1000 °C| 2.5045 ± 0.0001 | 2.6336 ± 0.0001    | 2.8581 ± 0.0001    | 2.5346 ± 0.0001    | 16.00 ± 0.65    | 14.62 ± 0.62    |
6–11. Figs. 1 and 2 represent the X-ray diffraction of the crude samples SA and CH respectively. The interreticular measured distances, the Miller indices and the 2θ position of the diffractometric reflects X-ray diffraction for all phases are given in Table 12. Fig. 3 represent SA clay FTIR spectra at final temperatures of 250 °C, 500 °C and 850 °C, CH sample was not shown because of similarities with SA sample while FTIR peak attributions are given in Table 13. Fig. 4 represents the blue stains test spotted on a Whatman Filter Paper, 55mm Diameter. Thermal phenomenon observed during the linear calcination of the SA clay powder are presented in Table 14.

2. Experimental design, materials, and methods

2.1. Equations used in classical method analysis

2.1.1. Weight loss on ignition

\[
\text{Mass loss \%} = \left( \frac{m_{110} - m_T}{m_{110}} \right) \times 100
\]  \hspace{1cm} (1)

In Eq. (1), \(m_{110}\) is the specimen’s weight at 110 °C, \(m_T\) is the specimen’s weight fired at final temperature of 250, 500, 700, 800, 850, 900, 950, 1000, 1050 and 1100 °C.

2.1.2. Shrinkage analysis

\[
\frac{\Delta L}{L_0} = \left( \frac{L_T - L_0}{L_0} \right) \times 100
\]  \hspace{1cm} (2)

In Eq. (2), \(L_0\) is the diameter of the flat disc before firing and \(L_T\) is the diameter flat disc after calcination to final temperature \(T\).

2.1.3. Water absorption and porosity

\[
\text{P \%} = \left( \frac{m_f - m_0}{m_0} \right) \times 100
\]  \hspace{1cm} (3)

In Eq. (3), \(m_0\) is the initial weight and \(m_f\) is the finale weight of the flat disc after firing.

2.1.4. Chemical resistance

\[
R = \frac{m_0 - m_{\text{pH},10}}{m_0}
\]  \hspace{1cm} (4)

| Table 5 | Weight loss after treatment with HCl and NaOH for SA and CH clays compacted flat discs calcined at 850 °C. |
|---------|-----------------------------------------------------------------------------------------------------|
| pH  =  5.0  | 3.0033 ± 0.0001 | 3.0894 ± 0.0001 | 2.3767 ± 0.0001 | 2.4400 ± 0.0001 | 20.86 ± 0.03 | 21.02 ± 0.04 |
| pH  =  10.0 | 3.0109 ± 0.0001 | 3.0100 ± 0.0001 | 2.300 ± 0.01 | 2.1021 ± 0.0001 | 23.61 ± 0.01 | 30.16 ± 0.06 |
In Eq. (4) $m_0$ is the mass of the flat disc before pH attack and $m_{pH}$ is its mass after removing the disc from the acidic (HCl, pH = 5.0) or basic (NaOH, pH = 10.0) solutions for 24 hours.

### 2.1.5. Blue value (AFNOR)

The blue value of the clay following AFNOR procedure was calculated using Eq. (5).

$$V_B = \frac{(V \times 0.01 \times 100)}{M}$$  \hspace{1cm} (5)

In Eq. (5), $V$ defines the methylene blue volume flowed in mL, 0.01 is the concentration in g/mL of the methylene blue solution, and $M$ is the mass in grams of the dry sample.

---

**Table 6**

Percentages of the oxides composing the SA crude clay powder.

| Elem      | Wt % | Mol % | K-Ratio | Z     | A    | F     |
|-----------|------|-------|---------|-------|------|-------|
| Na$_2$O   | 1.42 | 1.6   | 0.0028  | 0.967 | 0.2776 | 1.0046 |
| MgO       | 4.48 | 7.76  | 0.0107  | 0.992 | 0.3954 | 1.0084 |
| Al$_2$O$_3$ | 25.24 | 17.3  | 0.0662  | 0.9634 | 0.5101 | 1.009  |
| SiO$_2$   | 51.88 | 60.33 | 0.1209  | 0.992 | 0.5021 | 1.0013 |
| SO$_3$    | 1.37 | 1.19  | 0.0029  | 0.9851 | 0.5436 | 1.0031 |
| Cl$_2$O   | 0.49 | 0.39  | 0.0024  | 0.9364 | 0.6477 | 1.0048 |
| K$_2$O    | 4.54 | 3.36  | 0.029   | 0.9404 | 0.8145 | 1.0057 |
| CaO       | 3.7  | 4.61  | 0.0216  | 0.964 | 0.8458 | 1.0029 |
| TiO$_2$   | 0.99 | 0.87  | 0.0048  | 0.8839 | 0.9158 | 1.0053 |
| Fe$_2$O$_3$ | 5.9  | 2.58  | 0.0361  | 0.8841 | 0.99  | 1.0    |
| Total     | 100  | 100   |         |       |      |       |

**Table 7**

Percentages of the oxides composing the CH crude clay powder.

| Elem      | Wt % | Mol % | K-Ratio | Z     | A    | F     |
|-----------|------|-------|---------|-------|------|-------|
| Na$_2$O   | 0.96 | 0.85  | 0.0017  | 0.9514 | 0.2552 | 1.0024 |
| MgO       | 6.34 | 2.97  | 0.0048  | 0.976 | 0.3699 | 1.0043 |
| Al$_2$O$_3$ | 12.92 | 6.94  | 0.0326  | 0.9479 | 0.5   | 1.0049 |
| SiO$_2$   | 25.44 | 23.18 | 0.0653  | 0.9762 | 0.5619 | 1.0008 |
| SO$_3$    | 0.46 | 0.31  | 0.0012  | 0.9695 | 0.6787 | 1.0023 |
| Cl$_2$O   | 2.66 | 1.55  | 0.0187  | 0.9229 | 0.9102 | 1.0052 |
| K$_2$O    | 2.42 | 2.36  | 0.0153  | 0.9466 | 0.9324 | 1.0026 |
| CaO       | 0.5  | 0.33  | 0.0024  | 0.8685 | 0.9752 | 1.0053 |
| TiO$_2$   | 4.15 | 1.42  | 0.0255  | 0.8684 | 1.014  | 1       |
| Total     | 100  | 100   |         |       |      |       |

**Table 8**

Percentages of the oxides composing the SA clay powder calcined to 850 °C.

| Elem      | Wt % | Mol % | K-Ratio | Z     | A    | F     |
|-----------|------|-------|---------|-------|------|-------|
| Na$_2$O   | 0.83 | 2.04  | 0.0034  | 0.9703 | 0.2589 | 1.0042 |
| MgO       | 4    | 12.34 | 0.0157  | 0.9953 | 0.3674 | 1.0071 |
| Al$_2$O$_3$ | 22.37 | 15.46 | 0.0534  | 0.9666 | 0.4632 | 1.0079 |
| SiO$_2$   | 48.61 | 57.02 | 0.1092  | 0.9953 | 0.4823 | 1.001  |
| SO$_3$    | 1.37 | 1.19  | 0.0029  | 0.9851 | 0.5436 | 1.0031 |
| Cl$_2$O   | 0.49 | 0.39  | 0.0024  | 0.9364 | 0.6477 | 1.0048 |
| K$_2$O    | 4.32 | 3.23  | 0.0279  | 0.9441 | 0.82  | 1.0066 |
| CaO       | 3.29 | 4.14  | 0.0195  | 0.9676 | 0.8516 | 1.0049 |
| TiO$_2$   | 0.5  | 0.44  | 0.0025  | 0.8871 | 0.9212 | 1.0112 |
| Fe$_2$O$_3$ | 4.12 | 5.32  | 0.0742  | 0.8874 | 0.993  | 1.0    |
| Total     | 100  | 100   |         |       |      |       |
2.1.6. Blue index (ASTM)

The blue index according to ASTM (MIB, in equivalence/100 g) was calculated following Eq. (6) [3].

\[ MIB = \left( \frac{E \times V}{C_2} \times 0.1 \right) / W \] (6)

In Eq. (6), E stands for the MB number of equivalents per mL of water, V represents the volume of the MB solution in mL (unit of titration was 1.0 mL), and W represents the weight of the dry clay sample in g.

2.2. Loss on ignition

The loss on ignition was carried out on clay powders, for this 3.0 g of crude clays (SA or CH) where introduced into porcelain crucibles and successively heated to final temperatures of 250 °C, 500 °C, 750 °C, and 950 °C.

Table 9

Percentages of the oxides composing the CH clay powder calcined to 850 °C.

| Elem  | Wt % | Mol % | K-Ratio | Z    | A    | F    |
|-------|------|-------|---------|------|------|------|
| Na₂O  | 0.85 | 0.73  | 0.0016  | 0.9514 | 0.2672 | 1.003 |
| MgO   | 7.09 | 9.41  | 0.0162  | 0.9759 | 0.3871 | 1.0046|
| Al₂O₃ | 10.29 | 5.4   | 0.0252  | 0.9478 | 0.4857 | 1.0059|
| SiO₂  | 31.11 | 27.71 | 0.0802  | 0.9761 | 0.5647 | 1.0009|
| SO₃   | 0.47 | 0.31  | 0.0012  | 0.9694 | 0.6566 | 1.0023|
| K₂O   | 2.76 | 1.57  | 0.019   | 0.9229 | 0.8966 | 1.0051|
| CaO   | 3.01 | 2.87  | 0.0188  | 0.9466 | 0.9212 | 1.0015|
| TiO₂  | 0.47 | 0.31  | 0.0024  | 0.8685 | 0.966  | 1.0025|
| Fe₂O₃ | 2.03 | 0.68  | 0.0125  | 0.8683 | 1.0105 | 1     |
| Total | 100 | 100   |         |       |      |      |

Table 10

Percentages of the oxides composing the SA clay powder calcined to 950 °C.

| Elem  | Wt % | Mol % | K-Ratio | Z    | A    | F    |
|-------|------|-------|---------|------|------|------|
| Na₂O  | 0.82 | 0.73  | 0.0015  | 0.9521 | 0.2607 | 1.0027|
| MgO   | 2.7  | 3.71  | 0.006   | 0.9766 | 0.3783 | 1.0049|
| Al₂O₃ | 18.6 | 7.87  | 0.037   | 0.9485 | 0.506  | 1.0055|
| SiO₂  | 49.96 | 26.74 | 0.0739  | 0.9768 | 0.5575 | 1.0007|
| SO₃   | 1.37 | 1.19  | 0.0029  | 0.9851 | 0.5436 | 1.0031|
| Cl₂O  | 0.49 | 0.39  | 0.0024  | 0.9364 | 0.6477 | 1.0048|
| K₂O   | 2.63 | 1.55  | 0.0182  | 0.9236 | 0.9004 | 1.0048|
| CaO   | 2.44 | 2.41  | 0.0153  | 0.9473 | 0.925  | 1.0022|
| TiO₂  | 0.5  | 0.35  | 0.0025  | 0.8691 | 0.9706 | 1.0042|
| Fe₂O₃ | 3.37 | 1.17  | 0.0207  | 0.869  | 1.0123 | 1     |
| Total | 100 | 100   |         |       |      |      |

Table 11

Percentages of the oxides composing the CH clay powder calcined to 950 °C.

| Elem  | Wt % | Mol % | K-Ratio | Z    | A    | F    |
|-------|------|-------|---------|------|------|------|
| Na₂O  | 0.91 | 0.82  | 0.0017  | 0.9562 | 0.2646 | 1.0033|
| MgO   | 9    | 12.54 | 0.0205  | 0.9809 | 0.3824 | 1.0049|
| Al₂O₃ | 11.6 | 6.39  | 0.0276  | 0.9526 | 0.4684 | 1.0064|
| SiO₂  | 34.19 | 31.96 | 0.085   | 0.981  | 0.5413 | 1.0012|
| SO₃   | 0.55 | 0.38  | 0.0013  | 0.9742 | 0.6294 | 1.0032|
| K₂O   | 3.61 | 2.15  | 0.0246  | 0.9284 | 0.879  | 1.0077|
| CaO   | 5.09 | 5.1   | 0.0313  | 0.952  | 0.902  | 1.0019|
| TiO₂  | 0.47 | 0.31  | 0.0024  | 0.8685 | 0.966  | 1.0025|
| Fe₂O₃ | 4.43 | 1.56  | 0.0271  | 0.8733 | 1.0032 | 1     |
| Total | 100 | 100   |         |       |      |      |
700 °C, 800 °C, 900 °C and 1000 °C at heating rate of 5 °C/min following the heating program derived from TGA/TDA analysis represented below.

\[
\begin{align*}
25^\circ C & \overset{5^\circ C/\text{min}}{\rightarrow} 250^\circ C (\text{hold 2h}) & & \overset{5^\circ C/\text{min}}{\rightarrow} 500^\circ C (\text{hold 2h}) & & \overset{5^\circ C/\text{min}}{\rightarrow} 700^\circ C (\text{hold 2h}) & \overset{5^\circ C/\text{min}}{\rightarrow} T_f \\
\end{align*}
\]

(7)

While the loss on ignition on specimen was carried out on flat disc made by compacting 3.0 g of the SA or CH clay powders with the granulometry of 250–500 μm made by ASTM standardized sieves and under a uniaxial pressure $4.6 \times 10^7 Pa$. The obtained pellets with diameter of 25.0 mm and thickness of 2.0 mm were calcined to various final temperatures between 250 °C and 1100 °C following the same heating program described above.

Fig. 1. X-ray diffraction for SA crude clay sample. Q: Quartz, C: Calcite, I: Illite and K: Kaolinite.

Fig. 2. X-ray diffraction for CH crude clay sample. Q: Quartz, C: Calcite, I: Illite and K: Kaolinite.
2.3. Shrinkage analysis

For shrinkage analysis, the weight before calcination and after calcination to a final temperature of previously compacted flat discs were recorded and calculations were made according to Eq. (2).

| d_{hkl} | (hkl) | 2 Theta | (hkl) | phase        |
|--------|-------|---------|-------|--------------|
| 4.27   | (100) | 20.9    | (100) | Quartz       |
| 3.35   | (101) | 26.7    | (101) | Quartz       |
| 2.45   | (110) | 36.5    | (110) | Quartz       |
| 2.12   | (200) | 42.5    | (200) | Quartz       |
| 1.81   | (112) | 50.1    | (112) | Quartz       |
| 1.373  | (212) | 67.6    | (212) | Quartz triplet |
| 1.374  | (203) | 67.7    | (203) |              |
| 1.380  | (301) | 68.0    | (301) |              |
| 7.17   | (001) | 12.3    | (001) | Kaolinite    |
| 4.47   | (020) | 19.8    | (020) | Kaolinite    |
| 3.57   | (002) | 24.8    | (002) | Kaolinite    |
| 2.38   | (003) | 37.9    | (003) | Kaolinite    |
| 10.0   | (002) | 8.7     | (002) | Illite       |
| 5.02   | (004) | 17.6    | (004) | Illite       |
| 3.34   | (006) | 26.6    | (006) | Illite       |
| 3.84   | (012) | 23.1    | (012) | Calcite      |
| 3.04   | (104) | 29.4    | (104) | Calcite      |
| 2.83   | (113) | 39.5    | (113) | Calcite      |

Fig. 3. FTIR spectrum of the SA natural clay sample at different temperatures showing the main vibrations, (a) crude clay, (b) calcined to 250 °C, (c) calcined to 500 °C and (d) calcined to 850 °C.
Table 13
Infrared peak attributions for SA crude clay mineral and calcined to final temperature of 850 °C (CH spectra was similar).

| IR Frequencies of crude clay | IR Frequencies of calcined clay to 850 °C | Attributions |
|-----------------------------|-------------------------------------------|--------------|
| 3708                        | Disappeared because of calcination        | ν Si–OH external (SiO₂) |
| 3632                        | 3660                                      | ν Al–OH external (Al₂O₃) |
| 3406                        | 3412                                      | ν OH (H₂O) interlayer water |
| 1625                        | 1640                                      | δ OH(OH₂) [2] |
| 1437                        | Disappeared because of calcination        | ν CO₃ |
| 1031                        | 1034                                      | ν Si–O (SiO₂) |
| 909                         | Disappeared because of calcination        | δ(Al/Si/Mg/Ca)OH |
| 870                         | Disappeared because of calcination        | δ(CO₃) |
| 786                         | Disappeared because of calcination        | δ(CO₃) |

ν: stretching vibration, δ: bending vibration.

Fig. 4. Blue stains (spots) spotted on a Whatman 1441–055 Quantitative Filter Paper Circles, 20 Micron, Grade 41, 55mm Diameter.

Table 14
Thermal analysis (TGA/TDA) attribution of different phenomena of the SA and CH clay samples.

| TGA weight losses’ intervals (°C) | TDA Peaks | Phenomenon | Attribution |
|-----------------------------------|-----------|------------|-------------|
| 30–123                            | 90        | endothermic| departure of the water adsorbed on surface of the clay |
| 366–402                           | 390       | endothermic| Interlayer water departure |
| 465–587                           | 530       | endothermic| Kaolinite and illite decompositions |
| 674–760                           | 740       | endothermic| Calcite decomposition |
| 908–935                           | 925       | exothermic | Mullite crystallization |
2.4. Water absorption and porosity

Flat discs compacted and calcined to temperature equal or above 500 °C were immersed in degassed boiled distilled water for a period of 24 h, the discs were removed and dried in the open air for a period of 10 min then weighed and water porosity of the specimen were determined using Eq. (3).

2.5. Chemical resistance

The resistance of SA and CH compacted flat discs to drastic acidic or basic conditions were tested by measuring the weight loss of the two specimen after immersing them in HCl solution of pH = 5.0 and NaOH solution of pH = 10.0. The weight losses under acidic and based condition were determined using Eq. (4).

2.6. Oxides composition of the clay minerals

The elemental chemical analysis of the natural clay was performed by a Tescan VEGA-XM SEM spectrometer equipped with Oxford Instruments X-Max 50 EDX detector. The EDX measurements were acquired from the characteristic peaks of elements present in the clay (Na, K, Mg, Ca, Al, Si, S, Ti and Fe). Elements concentrations were determined after treatment of signals and the main outcome of the analysis is the (K-ratio). The concentration of the i-th element in the sample was calculated using Eq. (5) [4,5].

\[
\frac{C_i}{C_{i(0)}} = ZAF \times \frac{I_i}{I_{i(0)}} = ZAF \times K \text{- ratio} \tag{8}
\]

The oxides concentration was calculated by using the “Elements by difference” in advanced pan EDX software, oxygen concentration was calculated as difference between 100% and the sum of all other elements. Then, percentages of oxides were calculated by combining oxygen with all other elements that can form oxides [6]. Based on previous studies on clay oxides used were: Na₂O, MgO, Al₂O₃, SiO₂, SO₃, Cl₂O, K₂O, CaO, TiO₂ and Fe₂O₃ [7,8]. The compositions of the two clay samples SA and CH in oxides at different temperatures are given in Tables 5e10.

2.7. X-ray diffraction

The clay powder was analyzed using XRD, two types of analysis were carried out on the clay samples. First, non-oriented analysis of fine enough powders was carried out in order to ensure the homogeneity of the sample. Second, 2.0 μm deposited clay fraction on a glass slide, which was extracted using the protocol for clay fraction extraction described later and left to dry for 24 hours at room temperature. In both analysis, non-oriented and oriented fraction, data was recorded using CuKα radiation (λ = 1.540 Å) on a PW1710 Philips Analytical diffractometer controlled by XPERT Quantify software (EA Almelo, The Netherlands) operating at 40 kV and 30 mA, with a copper anode and a graphite monochromator with a normal divergence (1.0°) and receiving slits of 0.1 mm dimensions. 1300 points were recorded using continuous scans in a 2θ range of 5.0° to 80°.

2.8. Clay fraction isolation

To separate elements with fraction less than 2 μm oriented preparation were made in 6 different steps: i- Grinding, it must produce a powder which is not too fine to preserve the clay minerals with a diameter of less than 2 μm [9]. ii- Discharge, it is done with distilled water, then the sample is subjected to magnetic stirring. The coarse material deposited at the bottom of the flask is removed by decanting. iii- Decarbonation, this involves the removal of carbonates (CaCO₃). This operation is necessary for several reasons [10], i) the carbonates include the clay minerals, and hence interfere with their deflocculating; ii) they dilute the clay fraction; obstruct the orientation of the preparations by
their non-lamellar form. Hydrochloric acid diluted to 10% is added dropwise to the clay suspension with magnetic stirring in order to avoid local overconcentration, while allowing a little time between each attack. pH is controlled with pH meter. When the solution becomes red, this indicate that the carbonates have been destroyed, at this stage it is necessary to stop the HCl addition and the agitation, then the suspension was allowed to settle. **iv- Washing,** its purpose is to free the sample from the excess of the hydrochloric acid, and to allow deflocculating of the clay fraction. If the supernatant becomes clear, it is enough to wash the sample without using the centrifuge, meaning, pour the supernatant, add distilled water, shake, leave it to decant, and so on until a neutral pH is obtained, pH was controlled by litmus paper. Otherwise, the suspensions were centrifuged at 2500 rpm for 5 min. Subsequently, the supernatant is removed, and the precipitate is re-suspended in distilled water. The precipitate is recovered, tested with pH paper, if it is not yet neutral, the centrifugation cycles must be re-instanted until the pH is neutral. **v- Suspended Sample,** the recovered precipitate is placed in a Beaker of 250 ml, to which distilled water is added, manually shaken, and left to decant for 1h 40mn. If, after 10 minutes, the supernatant is clear. This indicates that deflocculating is poorly performed. For this purpose, one to two drops of ammonia (NH₄OH) are added which reduces the pH to around 7.0 (the color of the pH paper becomes blue), this is an indication that deflocculating is promoted. **vi- Extraction of particles smaller than 2μm,** the supernatant must be cloudy so that the suspension is perfect. The contents of the upper 2 cm of the supernatant are recovered and placed in a Beaker of 100 mL. Distilled water was added and centrifuged at 3500 rpm for 40 minutes. The precipitate obtained is recovered by means of a spatula, deposited in glass slides, left to dry for 24 hours at room temperature and finally passed to analysis by X-ray diffraction.

2.9. Infrared spectroscopy

The FTIR analysis was carried out in the spectral range (400—4000) cm⁻¹ by a Bruker Platinum ATR tensor II spectrometer with a resolution of 4 cm⁻¹. A fine clay powder, raw or calcined to the required final temperature was kept in the oven at 70 °C, then taken in a desiccator to analysis in order to ensure absorbed waters are not present in the clay samples. The sample was squeezed between the swivel pressure tower and the diamond crystal of the ATR unit, then IR beam was stricken into the sample. Data was recorded in the transmittance mode. Only infrared of SA samples were recorded CH show identical spectra with SA, therefore was not shown.

2.10. Methylene blue (MB) stain test

Methylene blue stain test according to AFNOR was carried out using the following procedure [11]; 60.0 g of the clay sample was suspended in 500 mL of distilled water and stirred vigorously until homogenization. 5.0 mL of 10.0g/L of methylene blue solution were added to the homogenized solution using a burette. After each addition, spots (stains) were spotted on a Whatman 1441-055 Quantitative Filter Paper Circles, 20 μm, Grade 41, 55mm Diameter using a capillary. The sampling and spotting continued until the wet surface surrounding the deep blue spot is turned into light blue color. This represents the saturation of the clay by methylene blue.

**Acknowledgments**

The authors wish to express their gratitude to the Research Institute of Science and Engineering (RISE), University of Sharjah, Sharjah, UAE for the financial support provided through the seed grant number (1602142018).

**Conflict of Interest**

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.
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