

# Extraction of Silica from Three Varieties (Dista, Makalioka, Tsemaka) of Rice Bales of Madagascar

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## Abstract

Madagascar is ranked among the largest consumers of rice in the world. The annual per capita consumption of rice in rural Malagasy is 138 kg, and in urban areas 118 kg. Urban households eat 85% of their meals with rice. Rice cultivation is practiced throughout the island. The overall annual rice production is estimated at 4 680 630 t in 2020. For each tonne of paddy milled, 200 kg of rice bales (outer husk of rice) are obtained from husking, which have an energy content equivalent to 60 L of fuel oil. The rice husk contains a non-negligible amount of silica varying between 26% and 88% depending on the extraction methods. Two methods of silica extraction are used: the thermal method and the soft method. The silica content extracted from the rice husk is different in both methods. The extraction was carried out in three varieties of Malagasy rice: Makalioka, Tsemaka and Dista. The percentage of silica obtained varies from 82% to 85%. It was compared to that synthesised by the sol-gel process using the X-ray fluorescence technique and spectrophotometer infrared analysis. This

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study is of economic and environmental interest: recovery of agricultural waste, reduction of pollution and the circular economy in Madagascar. The extraction of nanometric silicon dioxide, commonly known as silica ( $\text{SiO}_2$ ), from rice bales allows for the transformation of abundant agricultural waste into a material of high added value.

**Keywords:** rice; bio-based materials; sol–gel process; nanosilica

## 1 Introduction

Madagascar is ranked among the largest consumers of rice in the world. The annual per capita consumption of rice in rural Malagasy is 138 kg, and in urban areas 118 kg. Urban households eat 85% of their meals with rice [1].

Rice cultivation is practiced throughout the island, with the exception of the extreme-south where the arid climate does not allow it. In the highlands, rice crops occupy almost 45% of cultivated areas, bringing two to three harvests per year, depending on the different rice varieties. The rice growing seasons are determined by the harvesting period. In general, 70% of the rice harvest of the entire territory occur between April and June.

Malagasy rice cultivation is done between April and June. There are 2 million small producers of rice in Madagascar. Equipment of the Malagasy rice farmers is limited. The overall annual rice production is estimated at 4 680 630 t in 2020 [2]. For every tonne of paddy milled, 200 kg of bales of rice are obtained from husking (removing the dry outer covering from the rice), which have an energy content equivalent to 60 L of fuel oil [1]. However, in Madagascar, rice bales are usually available in small quantities and distributed in tens of thousands from small rice mills. Because they are expensive to transport, they are often reused on site for baking building bricks. After the baking process, a tiny amount of the rice husk ash (RHA) [3], [4] is recovered to clean and polish pots and pans, but the majority are thrown into the wild, which contributes to environmental degradation. The valorisation of these combustion residues is therefore necessary and desirable.

Rice bale ash has a large amount of silica depending on the nature of the paddy and the method of combustion. Ash produced by a high-temperature incinerator laboratory can generally reach a high percentage of silica, which can range from 86.98% to 96.94% [5], [6]. The general objective of this study is to determine the yield of constituent silica in the three varieties of rice husk: Makalioka, Tsemaka and Dista, which are obtained by the thermal method [7], [8]. In Madagascar, rice bales are mainly used to bake building bricks. This study tries to value rice husks as a supplier of silica. The extraction of silica from rice husks has attracted considerable attention owing to its environmental and economic benefits. Conventional silica production relies on mining quartz and other mineral resources, which can be energy-intensive and environmentally damaging. In

contrast, rice husks represent a renewable and abundant biomass resource that can be converted into high-purity silica through chemical and thermal treatments [9].

To answer the question posed and to achieve the objectives, we will focus on the extraction of silica from the three varieties of rice husks. The size of the silica synthesised by sol–gel process is measured in nanometres. It was used as a reference for the analyses by X-ray fluorescence spectrometry and Fourier transform infrared spectroscopy (FTIR). Nanostructured silica obtained from rice husks has numerous applications [10] in agriculture, environmental remediation, catalysis, electronics, construction materials, and biomedical fields. In agriculture, nanosilica can improve plant resistance to pests and diseases, enhance nutrient uptake, and increase tolerance to abiotic stresses such as drought and salinity. Consequently, the valorisation of rice husk waste into high-value silica products contributes to both waste management and sustainable development. Silica nanoparticles can be used as nanofertilisers [11], protection against water stress [12], and disease control.

## 2 Materials and Methods

### 2.1 Silica Extraction [13]

#### 2.1.1 Materials

The main materials used in this study are the three varieties of rice husks that exist in Madagascar, namely, Makalioka, Tsemaka and Dista (Figure 1).



Makalioka



Tsemaka



Dista

**Figure 1:** Different varieties of rice husk

These three varieties of rice husk (Makalioka, Tsemaka, Dista) are treated in the same way for the extraction of silica.

#### 2.1.2 Calcination and Acid Pretreatment

The rice bales are washed with distilled water, dried at 104 °C and then grounded to increase the contact surface. They are calcined at 1 000 °C for 5 h to remove the organic matter and to obtain an ash rich in silica. Acid demineralisation consists of immersing

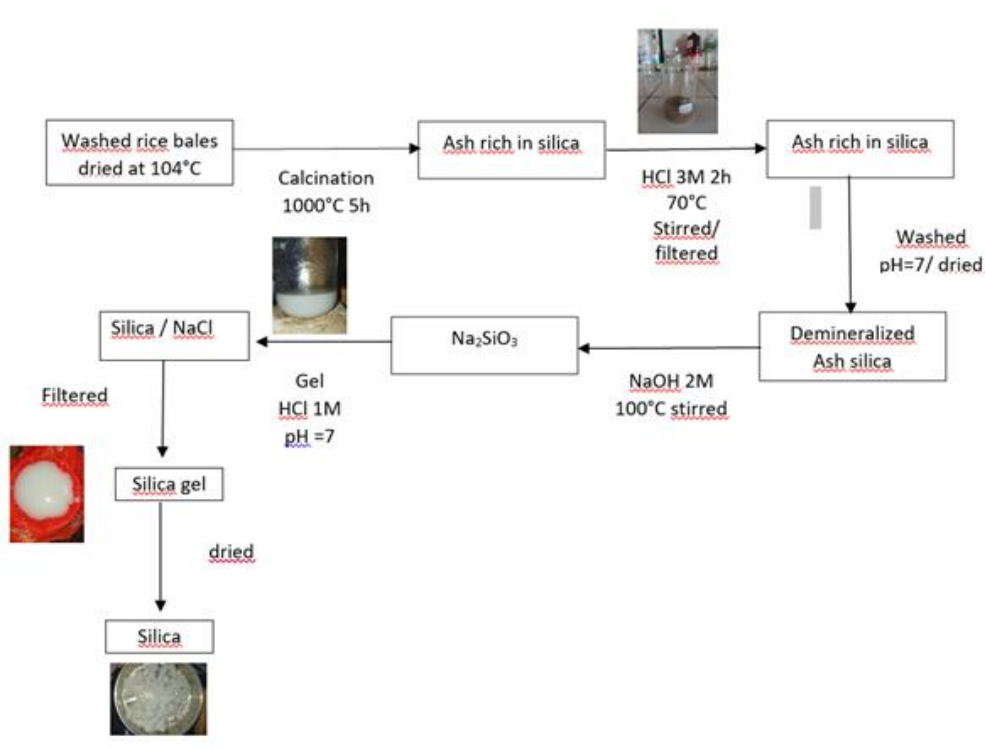
the ash of the rice husk in a solution of HCl (3M) with a ratio (m/v) of 1:10. The solution is stirred for 2 h at a temperature of 70 °C, then filtered and washed to neutrality (pH = 7), and dried.

### 2.1.3 Alkaline Extraction

After the acid demineralisation, the ash is treated with a solution of NaOH (2M), with a ratio of 1:10, at 100 °C, with stirring for 2 h to produce a solution of sodium silicate ( $\text{Na}_2\text{SiO}_3$ ). The mixture is allowed to settle and filtered to recover the liquid solution (sodium silicate).

### 2.1.4 Silica Gel

The Na silicate solution is neutralised with HCl (1M) dropwise to pH  $\approx$  7, which forms a white precipitate of silica and salt (NaCl) dissolved in water. The mixture is filtered to recover the silica precipitate and washed with distilled water (hot if possible) to remove any trace of salt. The gel is dried at 120 °C and then calcined again at 1 000 °C to remove the organic residues. The silica obtained is of high purity. The process is shown in Figure 2.



**Figure 2:** Silica extraction process

## 2.2 Sol–Gel Process for the Synthesis of Silica

Using a syringe, 8 ml of the metal precursor tetraethyl orthosilicate (TEOS) Sigma-Aldrich and 5.4 ml of ethanol, which is the solvent, are poured into a 50 ml beaker. The mixture is stirred for 10 min with magnetic stirring at a constant speed. The solution becomes homogeneous. Then, 0.3 ml of nitric acid ( $\text{HNO}_3$ ) is added. The system is stirred for 10 min. and then 12 ml of demineralised water are added dropwise to initiate the hydrolysis. Finally, the mixture is heated to 60 °C until a gel is obtained. A sample is taken every 15 min. After a few days of ageing of the solution, the gel hardens and releases the syneresis liquid.

## 2.3 Analyses of Samples

The X-ray fluorescence spectrometry analysis technique was used to analyse silica samples obtained by extraction in rice bales and by the hydrolytic sol–gel process (Figure 3).



**Figure 3:** Portable X-ray fluorescence spectrometer

## 3 Results

### 3.1 Silica Extraction in Rice Bales

#### 3.1.1 Percentage of Ash Yield

To measure the percentage of ash yield (PAY), a dried quantity of rice husk was calcined at 1 000 °C in the oven at a high temperature for 5 h to remove the organic matter. Then, the ash obtained rich in silica was weighed on a precision balance. The percentage ash yield is measured using the following relationship:

$$\text{PAY}(\%) = \frac{m_{\text{ash}}}{m_{\text{sample}}} \times 100$$

$m_{\text{ash}}$ : mass of ash remaining after combustion (g)

$m_{\text{sample}}$ : initial mass of the dry sample (g)

### 3.1.2 Percentage of Volatile Carbon

The percentage of volatile carbon (PVC) (Table 1) shall be calculated from the following formula:

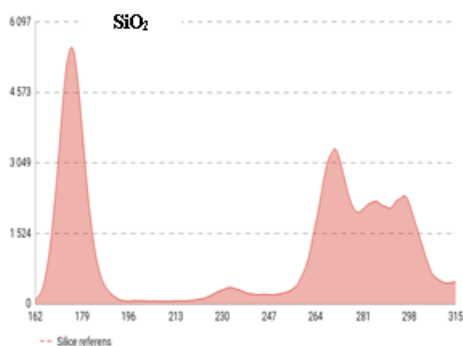
$$\text{PVC (\%)} = 100\% - \text{PAY}$$

**Table 1:** Percentages of ash yield and volatile carbon

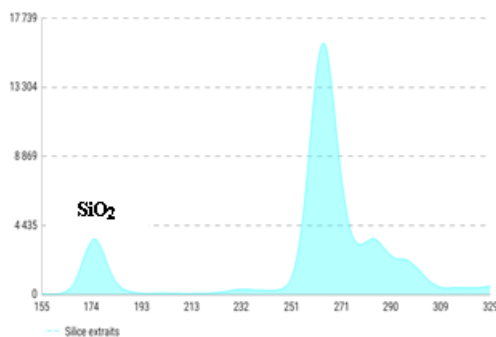
| Rice bale | Weight (g) | Ash weight (g) | PAY (%) | PVC (%) |
|-----------|------------|----------------|---------|---------|
| Makalioka | 30         | 9,2            | 30,6    | 69,3    |
| Tsemaka   | 18         | 5,45           | 30,2    | 69,8    |
| Dista     | 40         | 12,5           | 31,2    | 68,8    |

### 3.2 Analysis by X-Ray Fluorescence Spectrometry

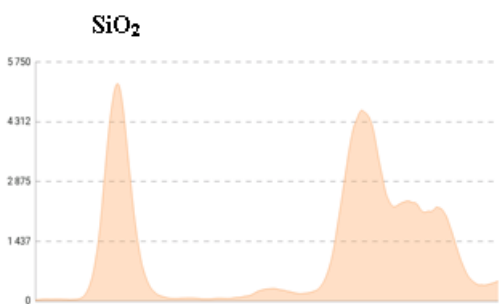
The crude samples and the ash (calcined crude) were analysed by X-ray fluorescence spectrometry for the two samples Dista and Tsemaka to evaluate the silica yield. Figures 4, 5 and 6, respectively, show the spectra of the reference silica obtained by the hydrolytic sol–gel process, the silica yield in the rice bale powder and the silica yield after calcination.



**Figure 4:** Silica reference spectrum (obtained by the sol–gel process)

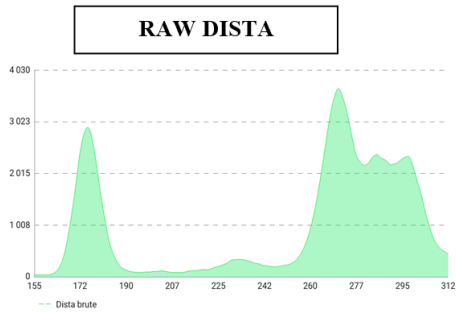


**Figure 4:** Silica spectrum after extraction

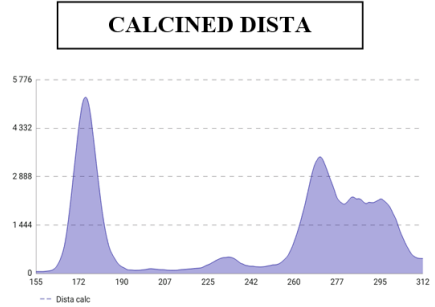


**Figure 6:** Silica spectrum after calcination at 1 000 °C

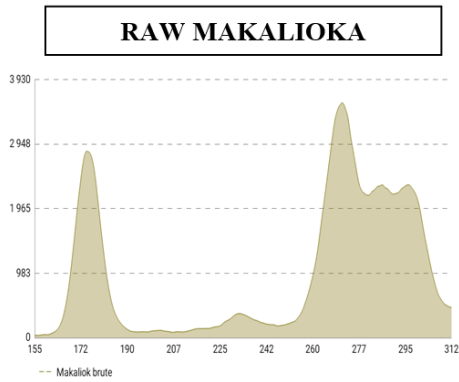
Figures 7–12 show the silica peaks in each of the raw and calcined samples of rice bales.



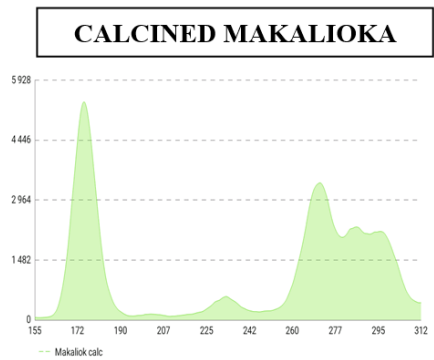
**Figure 7:** Silica spectra of raw Dista



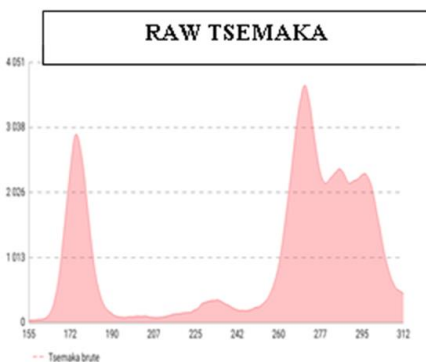
**Figure 8:** Silica spectra of calcined Dista



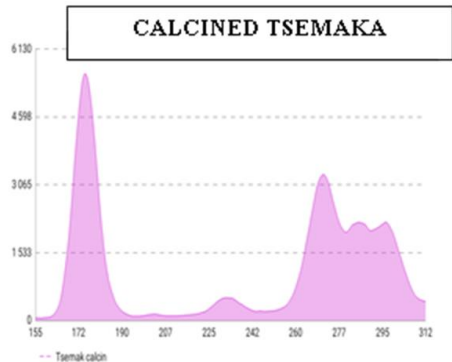
**Figure 9:** Silica spectra of raw Makalioka



**Figure 10:** Silica spectra of calcined Makalioka



**Figure 11:** Silica spectra of raw Tsemaka



**Figure 12:** Silica spectra of calcined Tsemaka

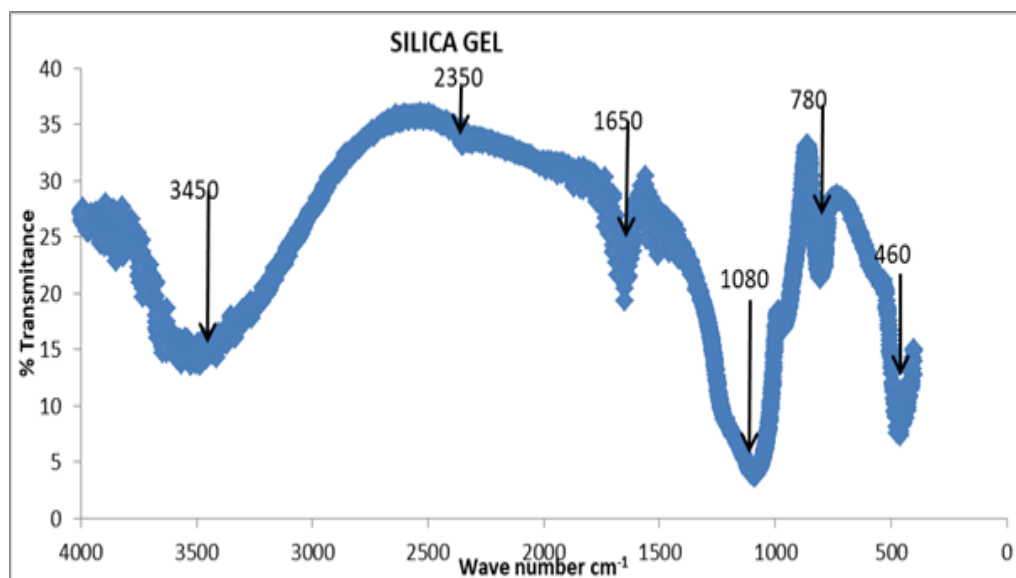
Table 2 presents the percentage of silica in each sample calcined at 1 000 °C.

**Table 2:** Silica yield

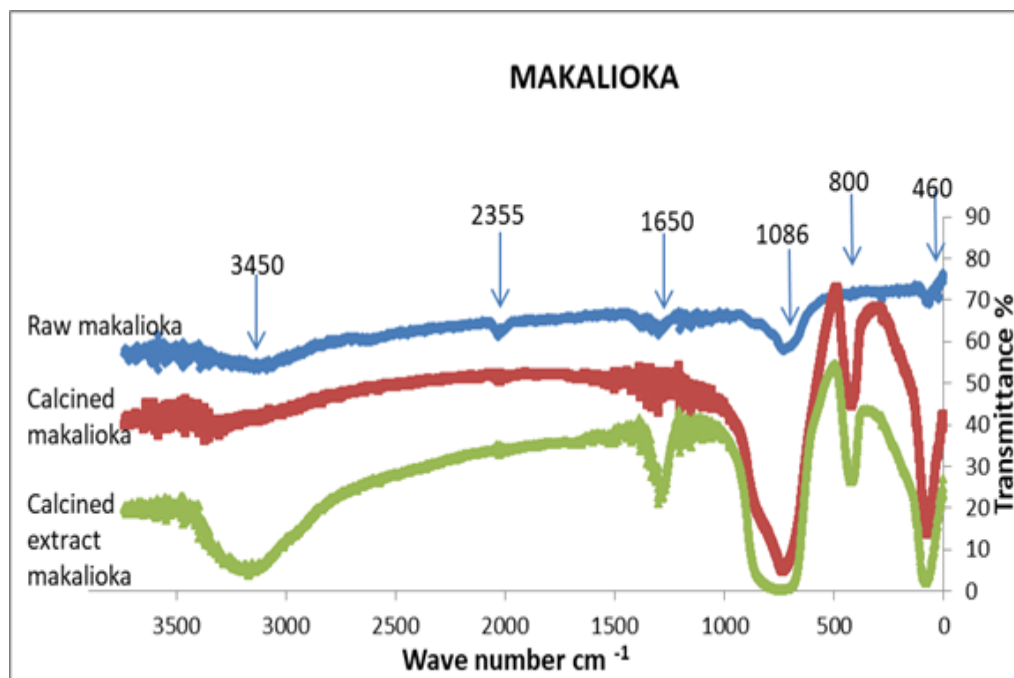
| Rice bale | Weight (g) | Ash weight (g) | Silica (%) |
|-----------|------------|----------------|------------|
| Makalioka | 30         | 9,2            | 85,5       |
| Tsemaka   | 18         | 5,45           | 83,85      |
| Dista     | 40         | 12,5           | 82,35      |

### 3.3 Fourier Transform Infrared Spectroscopy Analyses

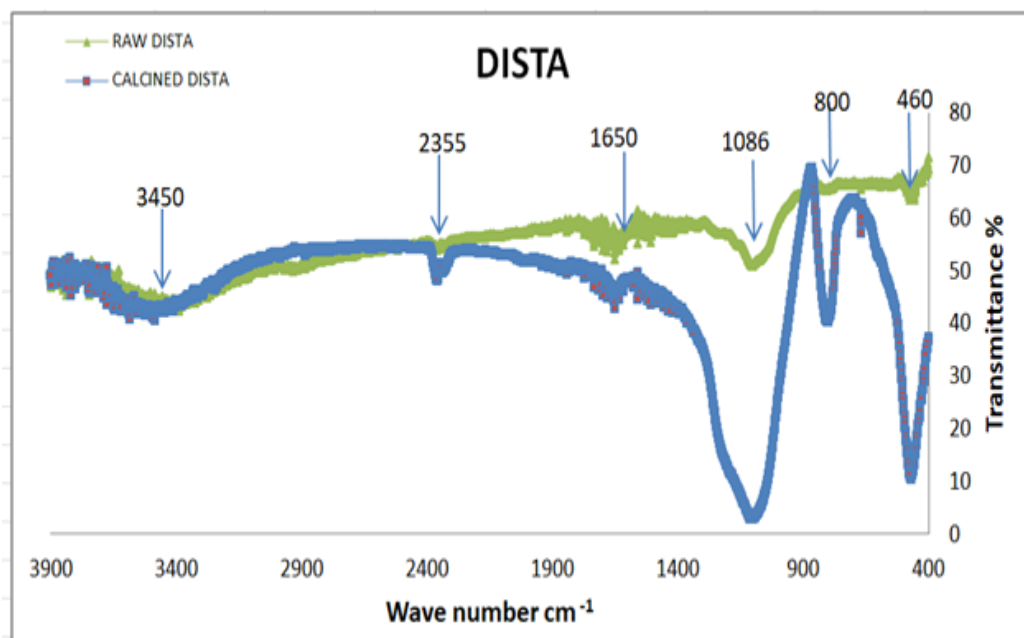
The samples were also analysed by FTIR to demonstrate the presence of Si–O–Si bonds which characterise the silica lattice and the bonds of the carbon residues (Figures 13–16).



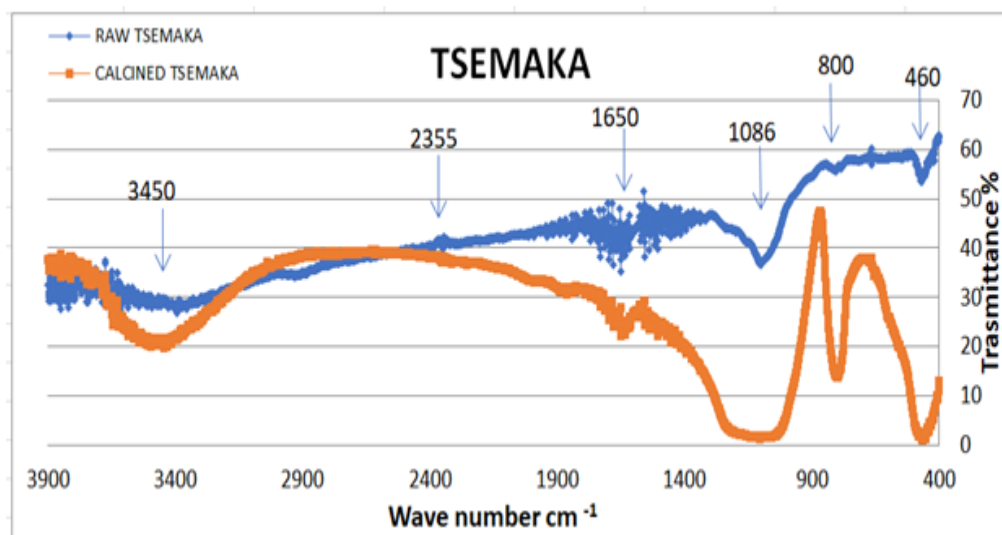
**Figure 5:** FTIR spectrum of silica synthesised by sol-gel



**Figure 14:** FTIR spectrum of Makalioka



**Figure 15:** FTIR spectrum of Dista



**Figure 6:** FTIR spectrum of TsemaKa

## 4 Discussion

The amount of silica obtained varies according to the rice varieties. As presented in Table 1, the variation of the percentage yield of ash in the three rice varieties is little (between 30.2 and 31.2%), as for the PVC. The analyses carried out by X-ray fluorescence spectrometry determined the percentages of silica in the three samples which vary from 82.35% to 85.5%. Analyses by FTIR (Figures 14, 15 and 16) confirmed the presence of silica in rice husk extracts. The peaks that correspond to the Si–O–Si bonds [14] in rice bales are at 1 086 cm<sup>-1</sup> [15], 800 cm<sup>-1</sup> and 460 cm<sup>-1</sup> and the peak at 3 450 cm<sup>-1</sup> corresponds to the Si–OH [16] bond and adsorbed water, silica being a very hygroscopic material and that at 2 355 cm<sup>-1</sup> would be CO<sub>2</sub>. The analysis by FTIR of the silica prepared by sol–gel process (Figures 13 to 16) shows the absorption of the different peaks. The residual carbon groups are approximately 1 650 cm<sup>-1</sup>. The peak (large band) at 1 070 cm<sup>-1</sup> to 1 086 cm<sup>-1</sup> can be assigned to the TO<sub>3</sub> vibration mode of silica [16].

## 5 Conclusions

Rice husk biomass from Madagascar represents an abundant and valuable source of silica. In this study, silica (SiO<sub>2</sub>) was successfully extracted and converted into nanosilica using an appropriate chemical treatment process. The silica extracted is compared to silica obtained by the sol–gel process. The characterisation results confirmed the successful synthesis of nanosilica. Fourier transform infrared spectroscopy analysis revealed the characteristic Si–O–Si and Si–OH vibrational bands,

indicating the formation of silica structures. X-ray fluorescence analysis demonstrated a high silica content and confirmed the purity of the extracted product. These findings indicate that rice husk ash can be effectively valorised into high-value nanosilica, providing a sustainable approach for using agricultural waste in Madagascar. The obtained nanosilica has potential applications in materials science, environmental technologies, catalysis, and industrial processes.

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