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Microwave power absorption profile of detergent surfactant agglomerates during microwave heating

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Abstract

The microwave energy absorption behaviour of the LAS (Linear Alkylbenzene Sulphonate) surfactant detergent agglomerates was studied while considering changes in the physical properties of the samples. Microwave heating was used to change the internal structure of the agglomerates to make a reduced density (fluffy) product. The absorption of energy within LAS samples indicated fluctuating trends as microwave heating progressed. This was associated with the dielectric properties of the material which are strongly dependent upon the nature ("free" or "bound") and quantity of water present in them at any instant which changes during heating. Therefore, complete profiles of the energy absorbed by the samples have been recorded to determine their actual power absorption behaviour / total energy consumption over time. The bulk density of the agglomerates decreased significantly when exposed to microwave fields. Hot air drying can be combined with microwave heating in order to reduce the total heating time. It has been observed that the pre-heating of the samples reduces the total heating time and microwave energy requirement. This is due to the temperature dependent decomposition of hydrates releasing more "free" water.

Keywords: Microwaves, bulk density, detergent agglomerates, microwave absorption, power consumption

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1. Introduction

The detergent industry makes a range of detergent powders ranging from compact, high bulk density products to low density, bulky products. The low dosage detergent products save resources and can be sold in small packages suitable for consumers. However, in many countries, consumers prefer to buy high dosage/ low bulk density products. The detergent granules are typically produced by either spray drying or agglomeration processes[1]. In spray drying processes, highly porous, low density granules are produced by drying aqueous detergent slurries in a spray drying tower by a counter or co-current flow of hot air[2]. The fine particles collected in exhaust air are typically added back in the dryer to generate larger particles[3]. Agglomeration processes produce high density particles by combining small, fine powders with a "binder" liquid (usually a surfactant) in some kind of mixer to produce larger "agglomerates". Typically agglomerates are higher in density than spray-dried powders and less suited for the high volume/low bulk density products preferred by some consumers[4].

Many dielectric materials are dipolar in nature. Therefore, when they are exposed to electromagnetic fields, dipolar components of molecules couple electrostatically and try to align with alternating field. This creates frictional force within the molecules and microwave energy is converted to heat[5]. Microwave heating has been extensively used in food, chemical and material processing due to its quick heating time, instantaneous on/off control and small footprint of the heating equipment[6-9]. Microwave heating can be used to change the internal structure of the materials. At optimal conditions, rapid, intense microwave heating can generate high internal pressures within the material by generation of steam[10]. This internal pressure can expand the material being heated resulting in a lower bulk density product. Thus, microwave heating can potentially be used to reduce the bulk density of high-density detergent agglomerates to produce the lower density products preferred by some consumers. This can be of interest in locations where a spray-drying tower would be uneconomic [11]. However, the materials within detergent agglomerate can have complex phase behaviors with a range of crystalline, liquid crystalline and amorphous phases being present dependent on factors such as concentration, available water level, presence of electrolytes etc. Only the "free" water contributes to the dielectric properties of the material [12]. Water tied up in crystalline hydrates is not able to interact with the microwaves and hence does not contribute to heating. During

microwave heating of the agglomerates, the bound water in some of these hydrates can become “free” due to thermal decomposition of the hydrates at elevated temperatures. As a result, the dielectric properties and electromagnetic energy absorption capability of many detergent products will change during the heating process.

This work presents the study of electromagnetic energy absorption behaviour of typical LAS (Linear Alkylbenzene Sulphonate) surfactant agglomerates during the period of microwave treatment. LAS is the most widely used surfactant globally, today domestic detergent formulas, either in powdered or liquid forms, contain a high proportion of LAS[13] . Domestic uses account for about 50% of the LAS production. Industrial uses include emulsion polymerization (polystyrene, polymethacrylate, PVC and other resins), agricultural self-emulsifying concentrates for seed and crop phytosanitary protection, production of elastomer of solid foams, emulsified paints, industrial cleaning and cleansing etc [13]. The instantaneous power absorbed by a sample is recorded along with its temperature history to understand the microwave heating curve of the detergent agglomerates.

Furthermore, the effect of pre-heating samples prior to microwave treatment is investigated. A comparison of energy consumption, residence time (the amount of time for which sample interacts with microwave fields) and bulk density between non-heated and pre-heated samples is presented.

2. Materials and methods

I. Microwave heating experimental equipment

A 1.8 kW continuous wave microwave waveguide heating experimental setup has been designed for laboratory use. The system is designed using WR-340 standard waveguide sections. The block diagram of the in-line heating equipment is shown in Figure 1 consisting of the following components.

Microwave Generator: A SM 845 (MKS Instruments Ltd) microwave power magnetron head and power supply is used to generate continuous power up to 1.8 kW at 2450 MHz. The output power generated from the magnetron is then fed via a WR-340 waveguide flange. Stable output power can be adjusted from 0.1 kW to 1.8 kW by using a DC (0-10 V) reference signal.

Isolator: Microwave isolator is a 2-port microwave device which allows electromagnetic waves to travel in one direction only. Its purpose is to protect the magnetron from the damaging effects of reverse microwave power due to mismatched load. A 6 kW GA116 microwave isolator from Gerling Applied Engineering (GAE) is connected to the

output of the microwave power generator. Microwave power generated from the magnetron travels in the forward direction to the load. The reflected power signal travelling in the opposite direction is dissipated in a water load connected to the isolator.

Power Meter: A bi-directional digital microwave power meter from GAE is connected to the output port of the isolator. This device measures the electromagnetic signals travelling in both forward and reverse direction. The bi-directional power meter consists of a bi-directional waveguide coupler, diode detector and a digital voltage meter.

Waveguide Tuner: A waveguide tuner (GA1002 from GAE) is used to maximize the forward power by matching the load impedance. The 3-stub waveguide tuner consists of a waveguide section with 3 movable tuning stubs placed at a distance of quarter wavelength from each other.

Waveguide Applicator: GA6004A universal waveguide applicator from GAE is used for the experiments. It is a standard WR-340 waveguide section with 5 cm diameter removable adapter ports on the top and bottom walls and a mounting boss on one side for attaching an infrared sensor head or a camera. This allows use of this applicator either in batch or continuous mode. There are a number of 3 mm diameter holes in one side wall of the applicator. These are used to insert fibre-optic temperature probes in the sample. There is a 2 cm diameter pipe connected to the other side wall of the applicator. This has been used to put the digital video recorder cam and a pipe to remove the evaporated water vapour. The photograph of the waveguide applicator with a powder sample in it is shown in Figure 3. An acrylic sample holder is used to place the samples inside the waveguide applicator.

Waveguide Water Load: At the end of the waveguide setup a waveguide dummy water load is connected. It consists of a short circuited waveguide with a flowing water glass pipe through it. This unit is connected to dissipate all of the remaining microwave power which is not being utilized by the sample. This helps to minimize reverse power in order to protect magnetron.

Fiber Optic Thermometer: A four channel unit (FOB-100 from Omega Engineering) is used to continuously monitor the temperature of the sample when exposed to microwave radiation. The fibre-optic probe is placed inside the sample through one of the 3 mm holes in the side wall of the applicator.

The photograph of the complete microwave heating unit is shown in Figure 2.

Figure 1 1.8 kW microwave heating equipment block diagram

Figure 2 Waveguide heating experimental setup

Figure 3 Waveguide applicator

II. Materials

The starting material used in this study was a detergent agglomerate containing 33% LAS surfactant, 63.5% inorganics (sodium carbonate, sodium sulphate, zeolite) and ~ 3.5% water and miscellaneous. 18 g white colored powder agglomerate samples with bulk density of 866 g/L were used for the microwave heating experiments.

III. Experiment procedure

Separate samples of the above agglomerate were conditioned to different moisture levels by exposure to a range of ambient humidity. After conditioning the sample had an eRH of 33% which corresponds to a free water level of ~ 3 wt % in the agglomerate. Following conditioning, the dielectric properties of the different agglomerate samples were measured and the samples then exposed to microwave heating. The loss factor and dielectric constant of the LAS agglomerate sample were measured using an open circuited microstrip stub partly loaded with the sample as described in [12]. The measurement process consists of measuring the scattering-parameters of a loaded and unloaded bandstop microstrip open circuit stub. The complex permittivity of the material is calculated from the shift in resonant frequency of the stub and dielectric absorption.

The samples were heated at three input power levels of 0.5 kW, 1 kW and 1.6 kW. The input power, power absorbed by the sample and the sample temperature profile were recorded over time. The equipment designed to heat the detergent powder agglomerates uses a bi-directional power meter between the signal generator and heating applicator. This measures forward power transmitted towards the heating applicator and any of the reverse power travelling back towards the generator due to mismatched load. Another unidirectional power meter is connected between the heating applicator and the water load. This measures the remaining forward power which has not been absorbed by the sample in the heating applicator. In this way, by combination of these two power meters we were able to quantify the input power, power absorbed by the sample, remaining power absorbed by the water load and any reflections travelling in the backward direction. There are no reflections in the current setup due to the water load connected at the end of the system. The reflected power measured by the power meter is always zero. The bulk

density of each sample was measured before and after microwave heating. Each bulk density measurement was repeated 3 times for accuracy. The following section discusses the results in detail.

3. Results

I. Dielectric loss measurement

The loss factor of a dielectric material is the key parameter which defines its behaviour of interaction with electromagnetic energy. It is the measure of how well a material absorbs the microwave power. It can be defined as the ratio of energy dissipated per cycle to energy stored per cycle [14]

$$Loss\ tangent = tan\delta = \frac{\varepsilon''}{\varepsilon'} \qquad (1)$$

Where ε'' and ε' represent the imaginary and real part of the complex permittivity of the material. Dry LAS agglomerates have very low loss factors; therefore, they are transparent to microwave energy. In order to increase the loss factor of the agglomerates, the samples are humidified by exposure to a high ambient equilibrium Relative Humidity (eRH). After conditioning the sample had an eRH of 33% which corresponds to a free water level of ~ 3 wt % in the agglomerate. The sample must have an appropriate level of initial moisture content in order to puff efficiently. Too little or too much water is not preferable as this can make the sample either microwave transparent or needing excessive drying. The agglomerate sample at 33% eRH had a dielectric constant of 2.21 and loss factor of 0.045.

II. Sample power (absorbed power)

Three different input power levels of 0.5 kW, 1 kW and 1.6 kW were used to modify the internal structure of the agglomerates. Increasing the input power significantly decreased the residence time required to achieve a given temperature of 150°C. Due to the small volume of the sample and the low loss factor of the sample only a fraction of the total incident power was actually absorbed by the sample. Increasing the input power from 0.5 kW to 1.6 kW slightly increased the instantaneous power absorbed by the sample but reduced the residence time significantly. This is due to the higher power density (electric field per unit area) when higher power input is selected [15]. The dissipated power is a function of loss factor and electric field intensity i.e.[16]

$$p = \omega\varepsilon_{\circ}\varepsilon'' E_i^2 \qquad (2)$$

The power absorption by the sample increases slowly until its temperature starts to approach 100°C. This suggests that the loss factor increases slowly with the temperature until it approaches 100°C. The power absorbed by the sample has peak absorption at around 100°C. This is consistent with the breakdown of sodium carbonate monohydrate which is known to thermally decompose at ~ 100°C to form anhydrous sodium carbonate and free water. This free water can then absorb more microwave energy. Shortly after this peak, power absorption starts to decrease because of evaporation of the free water to leave the dry powder.

The following Figure1, Figure 2 and Figure 3 show input power, power absorption and temperature profiles of the samples at input power levels of 0.5 kW, 1 kW and 1.6 kW, respectively. The graphs suggest that the power absorbed by the samples is temperature and time dependent due to the changes in the dielectric properties of the material during heating. It is observed that the residence time required raising the sample temperature to 150°C at 1.6 kW decreases to one-fourth of the time needed at an input power level of 0.5 kW.

Figure 4 Absorbed power and temperature vs time at input power of 0.5 kW

Figure 5 Absorbed power and temperature vs time at input power of 1 kW

Figure 6 Absorbed power and temperature vs time at input power of 1.6 kW

The Table 1 presents the bulk density data of the agglomerates before and after heating at various input power levels. It is evident that highest input power results in the lowest bulk density. This is due to higher power density which generates higher pressures inside the sample to make it puff rather than just dry.

III. Effect of pre-heating

From Figure 4 and Figure 5, it is observed that a significant period of the time is taken to increase the temperature of the sample to the point where water vapour begins to be formed inside the material. To minimise the microwave exposure time of the sample further, samples were pre-heated. Sealed samples were heated in an oven up to 70°C. The sample holders were also placed inside the oven so as to be at the same temperature. The samples were then transported to microwave applicator. Figure 7-9 present the power and temperature profile of the pre-heated agglomerate samples treated with 3 different microwave input power levels of 0.5 kW, 1 kW and 1.6 kW,

respectively. It is evident that at each of three input power levels the sample residence time decreases very significantly as compared to non-heated samples. For instance, at 1.6 kW input power, it takes only 24 sec to raise the sample temperature to 150°C as compared to 70 sec without pre-heating. Similarly, at 0.5 kW it reduces from 350 sec to 110 sec. Table 2 presents the bulk density and residence time data of the non-heated and pre-heated samples. The results show that the pre-heating adds the benefits of reduced residence time and further reduced bulk density as compared to non-heated samples.

Therefore, microwave heating can be combined with other sources of heating in order to minimize the residence time of the samples in the microwave applicator and further reduce their bulk density. This is important to minimise energy requirements and maximise throughput in any industrial process.

Figure 7 Absorbed power and temperature vs time at input power of 0.5 kW

Figure 8 Absorbed power and temperature vs time at input power of 1 kW

Figure 9 Absorbed power and temperature vs time at input power of 1.6 kW

Table-1 Bulk density of only microwave heated and pre-heated samples

IV. Total power consumption

In a batch process, the total microwave energy (E) supplied to the workload over a time period of T seconds can be calculated as[16]

$$E = \int_0^T P dt \qquad (3)$$

where p represents the incident power supplied to the samples. To calculate total power absorbed (E_{abs}) by the sample during its microwave exposure, total power p in equation3 should be replaced by the P_d (dissipated power).

$$E_{abs} = \int_0^T P_d dt \qquad (4)$$

A comparison of the total absorbed powers of non-heated and pre-heated samples is given in Figure 10. In addition to the reduction of residence time, pre-heating of the samples saves significant amount of microwave power. For example at an input power of 0.5 kW, pre-heated samples needed only 37% of the microwave energy as compared

to non-heated samples to raise its temperature to 150°C. This is due to the fact that with pre-heated samples all of the slow heating time associated with making bound-water free is reduced. The microwaves interact quickly with any available free water. Pre-heating of the samples helps to further reduce the bulk density of the sample due to a more rapid interaction of microwaves with free water inside the material thus generating increased pressure inside the agglomerates. The difference between the energy requirements of non-heated and pre-heated samples reduces with increasing input power. This is because the higher power density reduces the residence time of the sample required to make bound water free. But even at elevated input power of 1.6 kW, pre-heating saves 44% of microwave energy to raise the sample temperature to 150°C.

Figure 10 Power absorption of non-heated and pre-heated samples

Table 2 presents the comparison of residence time, bulk density and total absorbed power between pre-heated and only microwave heated samples.

Table-2 Comparison of residence time, bulk density and absorbed power between pre-heated and non-heated sample

4. Conclusion

The experimental results show that the microwave power absorbed by the material sample increases slowly until the sample temperature reaches 100°C, which is consistent with the breakdown temperature of sodium carbonate monohydrate. At this stage, the available free water absorbs more electromagnetic energy and a peak appears on the energy absorption curve. The residence time required to raise the sample temperature to 150°C at 1.6 kW decreases to one-fourth of the time needed at an input power level of 0.5 kW. In addition to reduction of the residence time, higher MW power levels also decrease the total energy consumption. For example, at an input power level of 1.5 kW, 60% less power is required to raise the temperature of the sample to 150°C as compared to 0.5 kW input power level. Pre-heating of the samples makes the microwave heating process more efficient and effective so the required residence time is shorter. For example, at an input power level of 0.5 kW, pre-heated samples needed only 37% of the microwave energy as compared to not pre-heated samples to raise their temperature to 150°C. Applying microwaves only when it is most effectively utilized will also minimise the use of electricity, which is often in short supply in some locations. The optimum combination of higher power levels (1.5 kW) and pre-heating

of the drying material reduces 78 % energy consumption as compared to not pre-heated sample dried at low power level of 0.5 kW in order to raise the material temperature to the required 150°C.

5. Acknowledgement

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6. References

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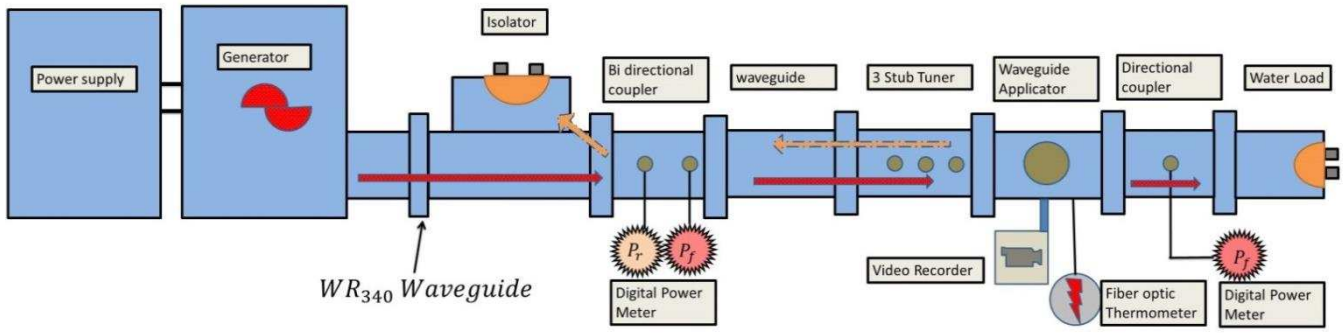

Fig 1 1.8 kW microwave heating equipment block diagram

Fig. 2 Waveguide heating experimental setup

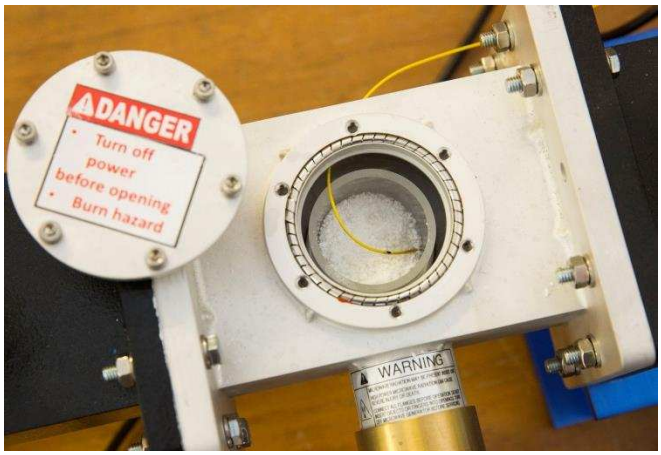

Fig 3 Waveguide applicator

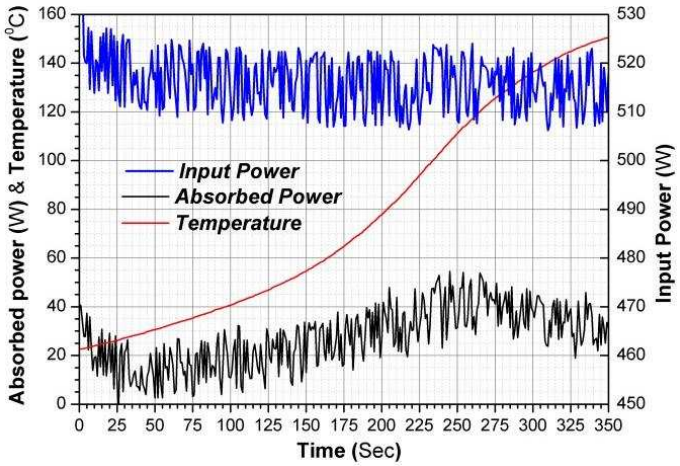

Fig 4 Absorbed power and temperature vs time at input power of 0.5 kW

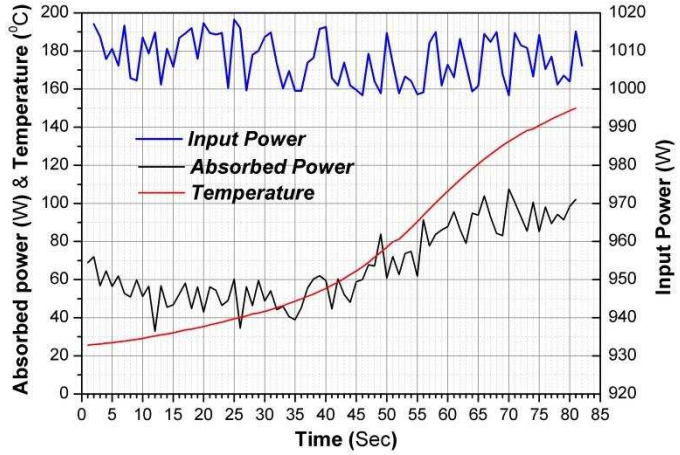

Fig 5 Absorbed power and temperature vs time at input power of 1 kW

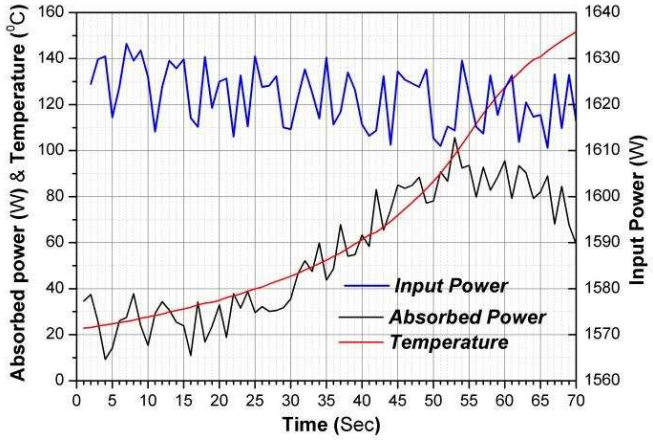

Fig 6 Absorbed power and temperature vs time at input power of 1.6 kW

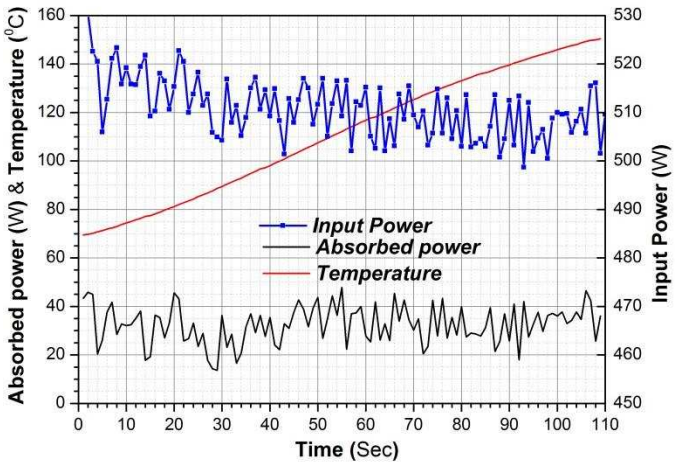

Fig 7 Absorbed power and temperature vs time at input power of 0.5 kW

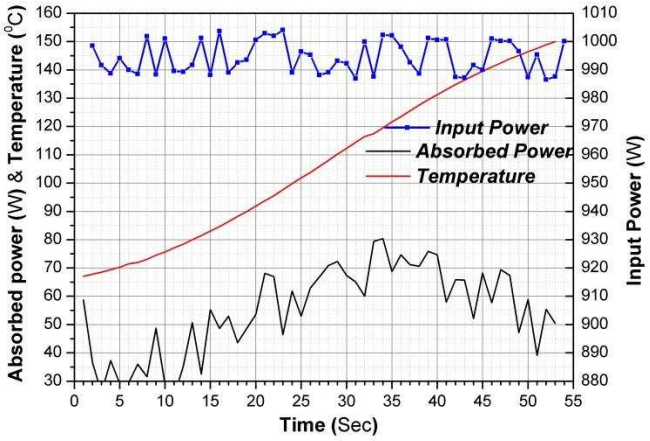

Fig 8 Absorbed power and temperature vs time at input power of 1 kW

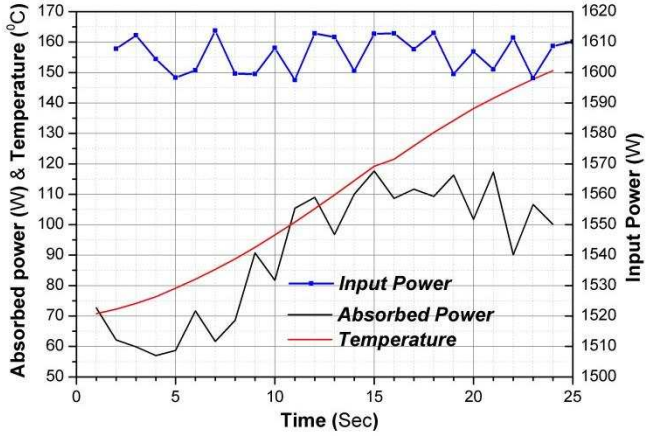

Fig 9 Absorbed power and temperature vs time at input power of 1.6 kW

Table-1 Bulk density of only microwave heated and pre-heated samples

Sample	Sample mass (g)	Bulk density before (g/L)	Bulk density after (g/L)	Power Incident (kW)
Non-Heated	18	866	825	0.5
	18	866	799	1
	18	866	783	1.6
Pre-Heated	18	866	822	0.5
	18	866	791	1
	18	866	780	1.6

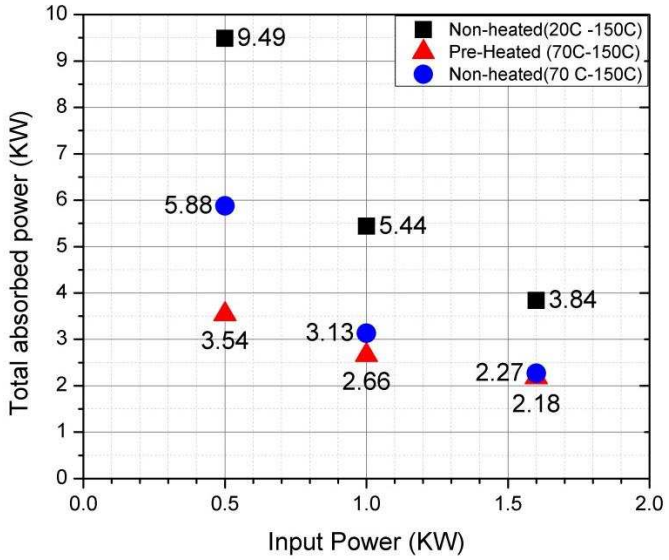

Fig 10 Power consumption of non-heated and pre-heated samples

Table-2 Comparison of residence time, bulk density and absorbed power between pre-heated and non-heated samples

Sample mass= 18 g Bulk density =866 g/L	Residence time (Sec)	Bulk density after (g/L)	Power incident (kW)	Total absorbed power (kW)
Non-Heated	350	825	0.5	9.49
	82	799	1	5.45
	70	783	1.6	3.84
Pre-Heated	108	822	0.5	3.54
	53	791	1	2.66
	24	780	1.6	2.18