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ORIGINAL PAPER

Microwave drying characteristics of microalgae (*Chlorella vulgaris*) for biofuel production

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Abstract Algal biofuels serve as a promising alternative energy source for liquid fuels. However, one of the bottlenecks in the conversion of microalgae to biofuels is the drying process. A moisture content of at most 10 % is desired for algal biomass prior to oil extraction to maximise biofuel yield. Conventional means of drying results to longer drying time and uneven drying of algal biomass. This study investigated the drying characteristics of microwave for microalgae (*Chlorella vulgaris*). Three microwave intensity levels (300, 600, and 900 W) were considered to dry 10, 20, and 30 of algal mass. Page model gave a better fit on the moisture ratio with time of microwave drying than the exponential model. Furthermore, the specific energy requirement was computed, and a relationship was found between moisture ratio with power and mass. Fourier transform infrared spectroscopy results showed significant reduction of infrared signal intensities of the functional groups present in the algae after drying at higher microwave power level. It was concluded that the 20 W/g microwave drying setting gave a lower specific energy requirement with good quality of remaining high lipid content qualitatively. Furthermore, it was recommended to use gas chromatography mass spectroscopy to further quantify the algal lipids and other functional groups.

Keywords Microalgae · Drying · Biofuel · Microwave · Page model · Chlorella vulgaris

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Introduction

Global oil demand from the transportation sector has been continuously increasing while this sector contributes one fifth of the overall carbon dioxide (CO_2) emissions (Balat and Balat 2010). Moreover, light vehicles are projected to grow to 2 billion by the end of 2050 which lead to an additional demand of fossil fuels. The eminent depletion of fossil fuels together with the projected increasing demand is forecasted to trigger an elevated fuel price (Rawat et al. 2013). Hence, biofuels are introduced to reduce the dependence on imported fossil-based fuels and to reduce CO_2 emissions (Zhu 2015). Experimentally, biofuels have less ecotoxicity than of petroleum fuels (Bulatov and Klemeš 2011). Experimental results from the study of Lapinskiene et al. (2006) showed that toxicity to soil organisms was greater by 3 % at petroleum diesel concentrations than of biodiesel. Biodiesel are categorised as first generation biofuels, which are harvested from edible

plant material, and second generation biofuels, which are produced from lignocellulosic or biomass material (Singh and Olsen 2011). There are numerous potential feedstocks that can be utilised for biofuel production: coconut, soybean, corn, canola, jatropha, and palm oil (Du et al. 2008). However, to meet 1 % of global fuel demand, 1 % of available land globally is used for first generation biofuels (Brennan and Owende 2010). To address the issue of food versus fuel and production per land area, third generation biofuels are proposed. Third generation biofuels are derived from microorganisms such as bacteria, yeast, and microalgae that produce oleaginous material. These microorganisms can grow in heterotrophic and photoautotrophic conditions (Singh and Olsen 2011).

Microalgae is one of the promising alternative energy sources due to its high oil yield per land area (Chisti 2007) while CO_2 is reduced for its cultivation (Glaser 2009). Microalgae are photosynthetic micro-organism that converts sunlight into different kinds of nutrients in various applications: nutraceutical, pharmaceutical, agricultural feed, and energy. There are benefits that microalgae can offer (Pokoo-Aikins et al. 2010). First, energy sources such as methane, bio-ethanol, bio-hydrogen (Brau et al. 2013), bio-oil (Grima 1994), and biodiesel (Gouveia and Oliveira 2009) can be produced from microalgae using various conversion methods (Koller et al. 2014). Second, the 60 % of dewatered microalgae's weight are pure biomass (Avagyan 2008), which prove that they are rich in oil prior to extraction process (Yanfen et al. 2012). Specifically, the production of biodiesel from microalgae involves stages from cultivation to harvesting, drying, oil extraction, and finally, transesterification (O'Connell et al. 2013). However, almost 60 % of the total energy consumed in the production of biofuels from microalgae is the drying process (Yanfen et al. 2012). It increases the effectiveness of the solvent-based extraction of oil (Iqbal 2012) and to prevent the formation of water and oil emulsion, resulting to higher quality of biofuels (Viswanathan et al. 2012). Moreover, to avoid spoilage due to hot climate environment, harvested biomass must be dried rapidly up to 5–10 % (dry solid) (Grima 2003).

Previous studies showed the various alternative drying methods used in drying microalgae for biofuels such as convective dryer, solar drying, rotary drying, spray drying, cross-flow drying, vacuum shelf drying, and flashing drying. Viswanathan et al. (2012) employed convective drying with a constant parallel air flow velocity of 0.25 m/s subjected at different drying temperatures to determine the lipid yield in the solvent extraction method. Becker and Venkataraman (1982) utilised solar heat to assess its feasibility to dry *Spirulina*. Prakash et al. (1997) also used solar drying for *Spirulina* and *Scenedesmus* algal species. However, the disadvantages of solar dryer are dehydration and disintegration of algal chlorophyll which alter the quality of the final algal product, and it is weather-dependent technology (Show et al. 2015). Similarly, Culaba et al. (2013) conducted an experiment on the drying characteristics of *Tetraselmis sp* using solar dryer. Issues such as uneven drying and charring of product occurred during the study. Rotary drying showed impressive dried algal product when drying *Scenedesmus* algae (Soeder and Pabst 1975). However, using this method consumed high energy for different kinds of species such as *Botryococcus braunii* (Singh and Olsen 2011). Spray drying showed high efficiency in drying the algae for human consumption. However, it is costly to operate, and it degrades the quality of the microalgae biomass (Chen et al. 2011). To acknowledge such concern of drying method issues, the microwave drying characteristics of microalgae was investigated.

Microwave drying is known for its rapid and effective heat distribution in the sample, and its high yield output such as for orange slices (Diaz et al. 2003) and for apple slices (Feng and Tang 1998). Microwave is part of the electromagnetic spectra whose frequency ranges from 300 MHz to 300 GHz. The usual microwave frequencies that are used in domestic microwave application and for drying agricultural products are 915 MHz and 2.45 GHz (Datta and Davidson 2000). Based on the theory of electromagnetic waves, the microwave is absorbed by ions. In the case of drying, the microwave energy is absorbed by the water molecules in the sample (Nikolić et al. 2011). The dipolar water molecules align themselves constantly with the oscillating electric field generating heat caused by friction between molecules, hence, drying the sample (Chanrasekaran et al. 2013). The difference of microwave mechanism to other conventional heating mechanism is the non-surface heating that penetrates the overall target (Teo et al. 2014). On the contrary, conventional heating applies heat from the outside surface although mass transfer occurs from inside to the outside.

Other studies used microwave irradiation for different purposes (Maskan 2000, 2001; Özbek and Dadali 2007; Soysal et al. 2006). Lee et al. (2010) used microwave assisted extraction to *Chlorella vulgaris, Botryococcus sp., and Scenedesmus sp.* in solvent extraction using Bligh and dyer method. To extract lipids from microalgae, Cheng et al. (2013) used microwave treatment in *Chlorella* PY-ZU1 investigating its effect on the dynamic microalgal cell wall microstructures. Similarly, Iqbal (2012) used microwave to extract oil from *Nannochloropsis sp*. On the other hand, several computational studies were performed determining the effects of temperature and pressure on the water transport through lipid membrane (Manrique et al. 2014) and aquaporin (Ducut et al. 2014) of microalgae.

In this study, the microwave drying characteristics of *Chlorella vulgaris* were investigated, and its chemical composition were analysed by Fourier transform infrared

spectroscopy before and after drying. This algal strain was chosen because of its abundance (Shih-Hsin Ho et al. 2014) and high oil content of about 14–40 % of dried biomass (Koller et al. 2014).

Methodology

This study comprises of microalgae cultivation, harvesting, microwave drying, mathematical modelling, and characterisation by Fourier transform infrared spectroscopy. Details for each procedure are shown in the succeeding subsections.

Microalgae cultivation

The initial strain of *Chlorella vulgaris* was obtained from the culture collection of the Plant Biology division, Institute of Biological Sciences, University of the Philippines Los Baños, Los Baños, Philippines.

The algal strain was cultured in a sterilised 6 L bottle container embellished with BG 11 medium at a pH level between 7.0 to 7.5 (Stanier et al. 1971). It contained $NaNO_3$, $K_2HPO_4{\cdot}H_20$, $MgSO_4{\cdot}7H_2O$, $CaCl_2{\cdot}2H_2O$, Citric Acid, Ferric Citrate, Na_2 EDTA, Na_2CO_3, and trace metals specifically H_3BO_3, $MnCl_2{\cdot}4H_2O$, $ZnSO_4{\cdot}7H_2O$, $Na_2{\cdot}MoO_4{\cdot}2H_2O$, $CuSO_4{\cdot}5H_2O$, and $Co(NO_3)_2{\cdot}6H_2O$. The medium was sterilised in a batch bag using autoclave system at 15 psi for 15 min. During inoculation, the conditions were maintained at 23 ± 1 °C, under a fluorescent light of 100 μmol m^{-2} s^{-1} with 24 h of aeration.

Harvesting

The mature 6 L freshwater microalgae were dewatered by centrifugation method at 2000 rpm using Megafuge 1.0 R manufactured by Heraeus-Christ GMBH mode. A concentration of 3.33 g/L of microalgae were collected from the 6 L of cultured microalgae.

Microwave drying

The drying of biomass was carried out in a 2M265-M12WJ magnetron IBF Electronic microwave generator with the maximum incident power of 3,000 W at a frequency of 2,450 MHz.

The schematic diagram of the experimental setup is shown in Fig. 1a. The microwave equipment is composed of the magnetron, a directional coupler with a dummy load to prevent the microwave energy to reflect back to the source. The power level of the microwave was controlled using a programmable Logic controller (PLc) to ensure consistency of the microwave intensity. Based on the standard R26 waveguide set by the International Electrotechnical Commission (IEC), the dimensions used in designing the drying chamber are 86.36 mm × 43.18 mm x 87.14 mm as shown in Fig. 1b. The third dimension is about half the waveguide wavelength ($\lambda_g = 174.28$) of the microwave at 2.45 GHz.

The experiments were conducted for 10 g, 20 g, and 30 g of microalgae exposed to microwave at three (3) different power settings 300, 600, and 900 W. The instantaneous weight of the microalgae was monitored using (Denver Instrument XE-4100 analytical balance) every 2 min. The microalgae drying was performed until the microalgae reached its bone-dried mass.

Mathematical modelling of microwave drying curves

Several studies have mathematically modelled the drying curves of different drying methods as shown in Table 1. Two moisture ratio models namely, Newton' Model (exponential model) and the Page Model were used in this study. These models are derived from the Fick's second law by simplifying the general series solution. The exponential model was initially used to describe the drying of mint leaves (Park et al. 2002) and mulberry (Doymaz 2004a, b). This model is written as

$$\text{Moisture ratio (MR)}\,(t) = \frac{m_t - m_e}{m_i - m_e} = ae^{-kt}, \tag{1}$$

where m_i (g) is the initial mass, m_e(g) is the equilibrium mass or bone-dried mass, and m_t (g) is the mass at time *t, a*, and *k* are constants in the model.

The exponential model was modified resulting to the Page Model to accommodate nonlinear exponent of time, and given as

$$\frac{m_t - m_e}{m_i - m_e} = e^{-kt^y}, \tag{2}$$

where *k* and *y* are constants in this model. Page model was used in microwave drying such as for parsley by Soysal (2004), sardine fish by Darvishi et al. (2013), coriander leaves by Sarimeseli (2011), and sorbus fruits by Lule and Koyuncu (2015).

To evaluate the goodness of fit of these mathematical models, a regression analysis was performed calculating the coefficient of determination (R^2) and reduced Chi-square (χ^2) between the predicted and experimental values. An R^2 close to 1 (Özdemir and Devres 1999) and lower χ^2 values indicate the goodness of fit (Ertekin and Yaldiz 2004). The reduced Chi-square is given by

$$\chi^2 = \frac{\sum_{i=1}^{N}\left(MR_{exp,i} - MR_{pre,i}\right)^2}{N - z}, \tag{3}$$

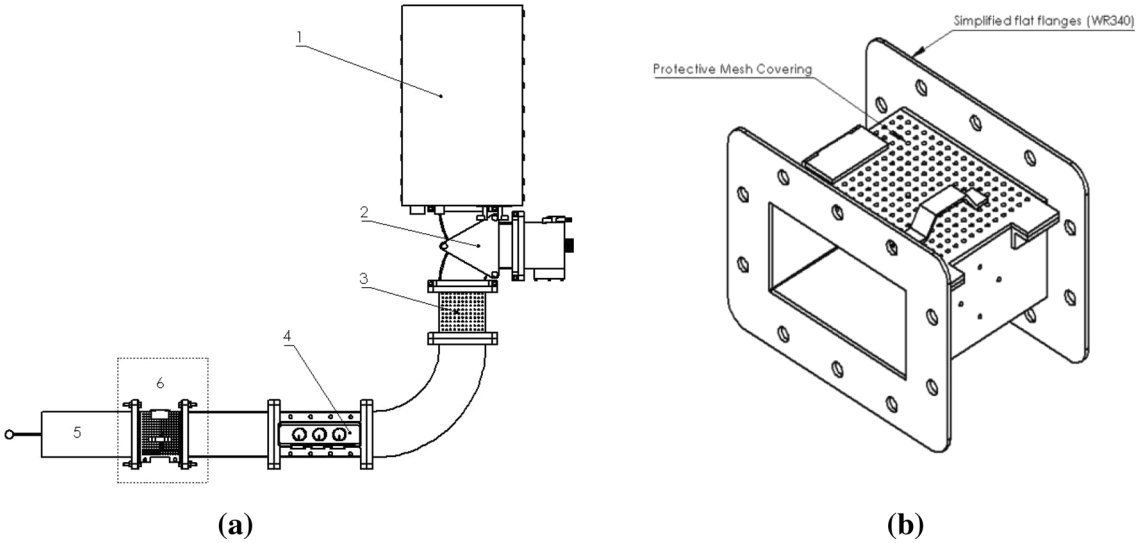

Fig. 1 The schematic diagram of the **a** microwave (magnetron) set up which includes the *1* magnetron head, *2* directional coupler with a dummy load, *3* quartz flash, *4* manual tuner, *5* sliding short, *6* chamber, and **b** design of microwave chamber

Table 1 Various mathematical models for kinetics of drying (Dissa et al. 2011)

	Name of model	Equation	References
1	Newton	$MR = e^{-kt}$	El-Sebaii et al. (2002)
2	Henderson and Pabis	$MR = ae^{-bt}$	Henderson and Pabis (1961)
3	Page	$MR = e^{-kt^y}$	Koua et al. (2009)
4	Modified Page	$MR = e^{-(kt)^y}$	Toğrul and Pehlivan (2002)
5	Logarithmic	$MR = ae^{-(kt)^y} + c$	Yaldiz et al. (2001)
6	Two-term model	$MR = ae^{-(k_0t)} + be^{-(k_1t)}$	Lahsasni et al. (2004)
7	Two-term exponential	$MR = ae^{-(k_0t)} + (1-a)e^{-(k_0at)}$	Midilli and Kucuk (2003)
8	Verma et al.	$MR = ae^{-(k_0t)} + (1-a)e^{-(gt)}$	Doymaz (2005)
9	Approximation of Diffusion	$MR = ae^{-(k_0t)} + (1-a)e^{-(k_0bt)}$	Usub et al. (2010)
10	Wang and Singh	$MR = 1 + at + bt^2$	Koua et al. (2009)

where $MR_{\text{pre},i}$ is the predicted moisture ratio, $MR_{\text{exp},i}$ is the experimental moisture ratio, N is the number of observations, and z is the number of constants.

Fourier transform infrared (FTIR) spectroscopy

The microalgae were characterised with FTIR spectroscopy using a NICOLET 6700 FTIR spectrophotometer in order to identify the chemical compounds present before and after microwave drying. The 20 g samples were prepared using potassium bromide (KBr) pellet method, in a 1:10 ratio of the dried microalgae and potassium bromide. It was ground and mixed using mortar and pestle, and pressed to form a thin pellet sample. The samples were scanned 32 times between the 4000 and 400 cm^{-1} region. This was conducted on the 20 g samples before and after exposure to microwave at three different specific power settings: 300, 600, and 900 W.

Results and discussions

In this section, the results on microwave drying, mathematical modelling, and FTIR spectroscopy are presented.

Microwave drying

Influence of power level

The effect of three power levels on the drying curve with different initial mass of microalgae is shown in Fig. 2. Generally, it shows that the drying curves are all in the falling period. There is no constant rate of drying period. The decay of the microwave drying curve is faster than that of the other drying methods performed by Gogus and Maskan (1999) and Gupta et al. (2002).

Figure 2 also shows that the drying power level caused an important increase in the drying rate leading to a shorter drying time. It means that the time required to reduce the

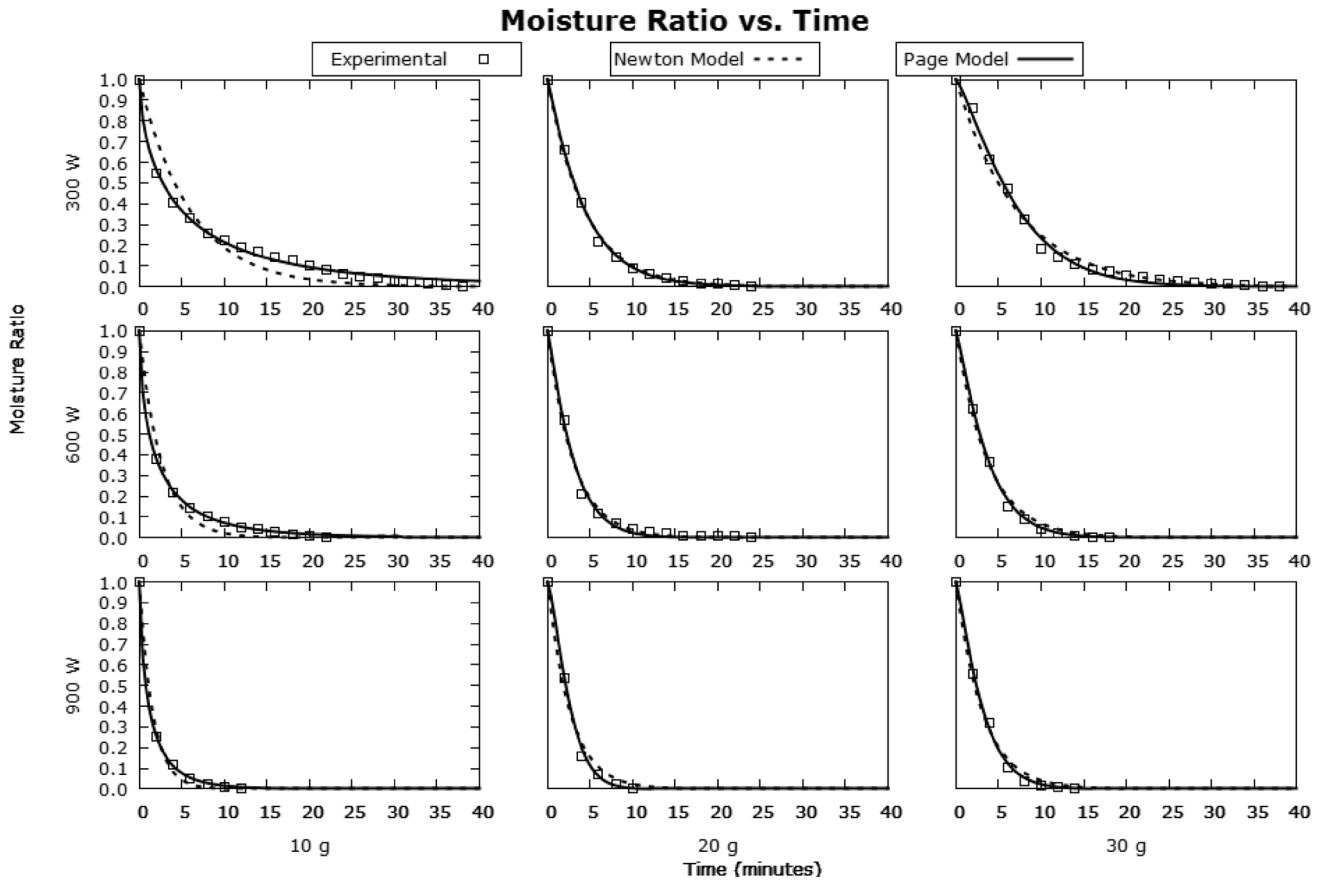

Fig. 2 Moisture content versus drying time with best fit line using Newton's model and Page model

moisture ratio is dependent on the power level of the microwave.

Mathematical model of the microwave drying characteristics

The drying curve shown in Fig. 2 can be generally characterised as an exponential function with time as modelled by the Newton model and Page model. It shows that the Page Model is a better fit than the Newton for the moisture ratio as a function of time at different power settings and initial mass. This can be validated by a higher coefficient of determination and lower Chi-square as shown in Fig. 3. The previous models are further evaluated by considering the power level and initial mass of the sample being dried.

Figure 4 shows the plot of the linear fit of the natural logarithm of moisture ratio as a function of time. Generally, it shows a good fit as indicated by the coefficient of determination (R^2). It can be seen that as the power level is increased, the linear fit becomes more accurate and the slope becomes steeper. This clearly shows the dependence of the slope on the power level. On the other hand, the initial mass of the sample appears to be insignificant to the drying curve since there is no variation in the slopes of the drying curve except on the moisture ratio curve of the 20 g at 300 W power level data.

As seen in Fig. 4, the microwave drying curve of moisture ratio is best modelled as

$$\mathrm{MR} = e^{-k(P,m_i)t^y}, \tag{5}$$

where k is dependent on the power level and initial mass of the sample, which resembles the Page model. This model takes consideration of the curvature at lower power levels.

Using nonlinear regression calculations, the best fit parameters for all data corresponding to the highest coefficient of determination of 0.9744 and reduced Chi-square (χ^2) of 0.002 is given by

$$y = 0.998; k = -\frac{P^{0.78}}{182.36 m_i^{0.32}}. \tag{6}$$

Table 2 shows the coefficient of determination for each power level using Eqs. (5) and (6). It shows a good fit for all dataset. Based on the model, the power level needed for drying the microalgae to the desired 10 % moisture ratio with a time requirement t_r (in min) is given by

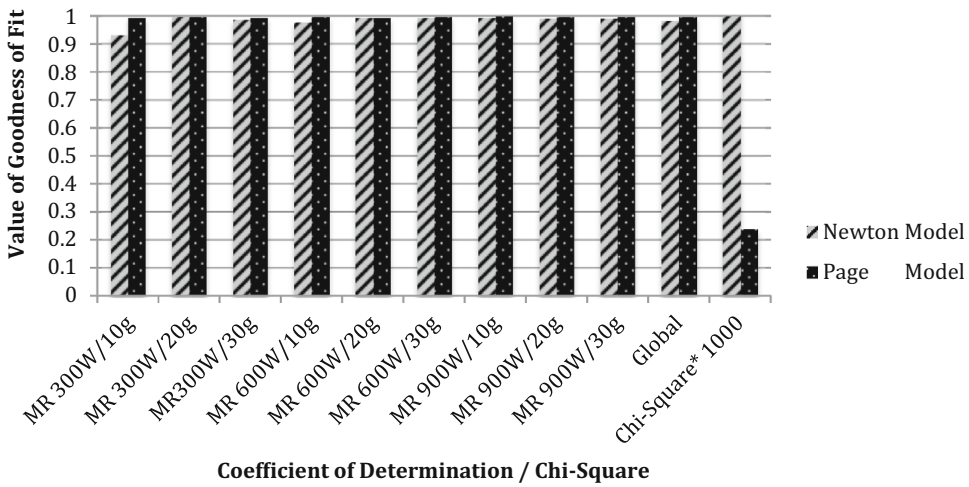

Fig. 3 Coefficients of determination and Chi-Square of Newton's model and Page model

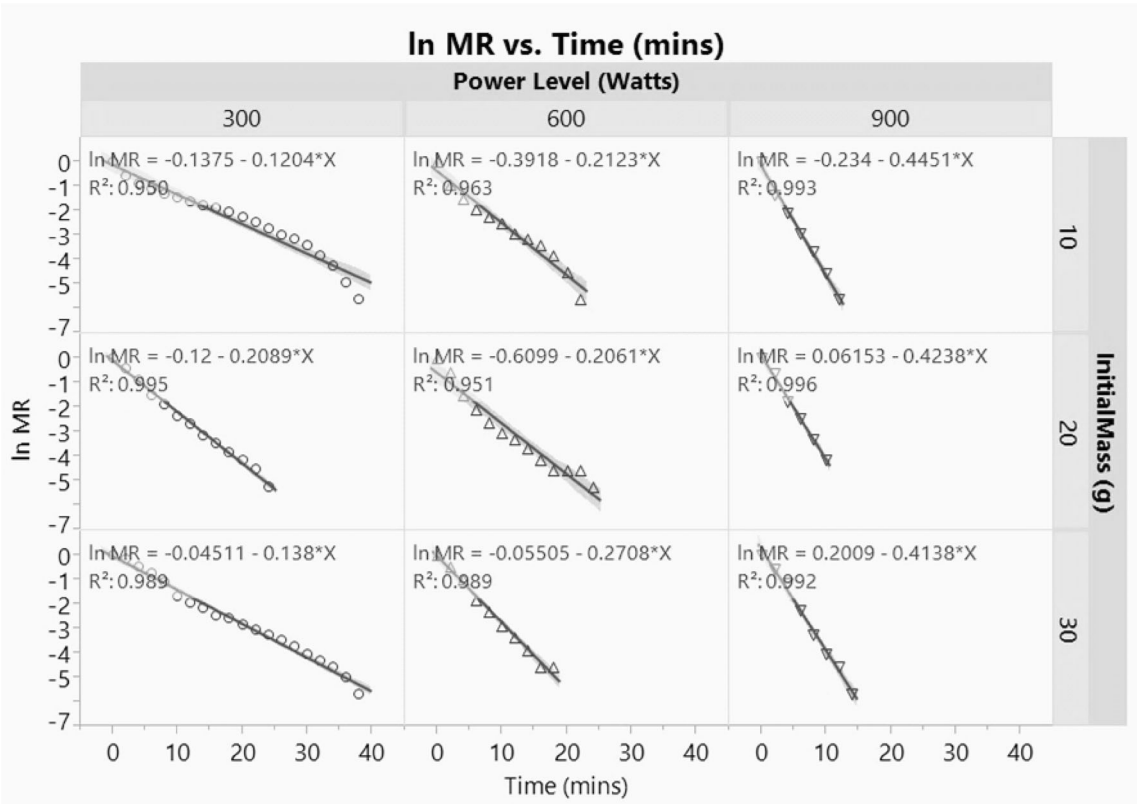

Fig. 4 Natural logarithm versus time plot where x in the equation is time

$$P = \left(419.9\frac{m_i^{0.32}}{t_r}\right)^{1.29}. \tag{7}$$

Energy of drying

The data were further analysed to determine the energy consumption in drying the algae. The specific energy requirements during the drying time were calculated. The specific energy requirement based on the study of Varith et al. (2007) and Soysal (2004), defined as the amount of energy required to remove a unit mass of moisture, were computed using Eq. (8):

$$\text{Specific energy requirement} = \frac{P \times t_i}{(m_i - m_t) \times m_e}, \tag{8}$$

where P is the power setting of the microwave oven in W, and t is the elapsed time in seconds.

The specific power settings were determined based on the power settings and microalgae sample size. The following specific power settings are 10, 15, 20, 30, 45, 60,

Table 2 Coefficient of determination

Power level (W)	Mass (g)		
	10	20	30
300	0.9025	0.9572	0.9837
600	0.9769	0.9935	0.9952
900	0.9819	0.9902	0.9907

and 90 W/g. Note that for 30 W/g, the combined results of the following power settings/sample size were used: 300 W/10 g, 600 W/20 g, and 900 W/30 g.

Figure 5 summarises the specific energy requirements for the various specific power settings as the samples are being dried. It shows that the 15 W/g setting requires the least energy to dry the sample until it reached its 42.5 % moisture content. Lower than its 42.5 % moisture content, the 20 W/g has the lowest specific energy requirement.

Derived from Eqs. (5) and (6), the specific energy requirement (joules) to dry the microalgae to 10 % moisture ratio is given by

$$E_s = 27,993 \frac{P^{0.22}}{m_i^{1.68}}. \quad (9)$$

Equation (9) indicates that the specific energy requirement decreases when large amount of algae is used in drying the algae at a given constant power level. This result agrees well with Fig. 4 which shows that the specific energy requirement is lower for higher initial mass given a constant power level.

Comparing the energy consumption with the other studies, microwave drying has shown a big potential for efficient and low cost drying method as shown in Table 3.

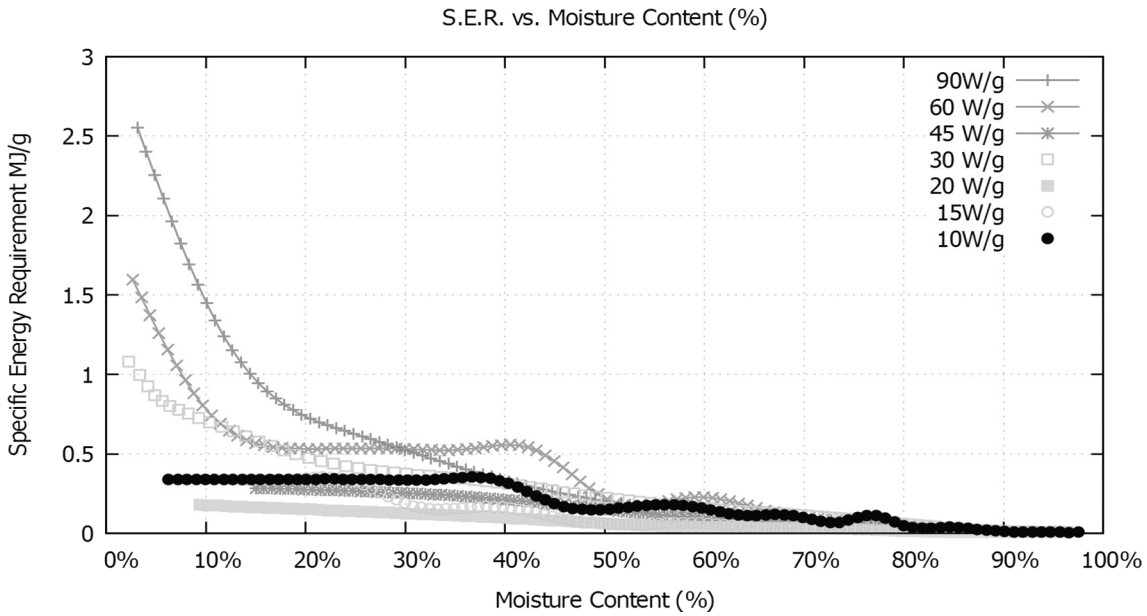

Fig. 5 Specific Energy Requirement vs. Moisture Content

Table 3 Energy consumption for drying

Reference	Method	Energy (MJ/kg)
Bennion et al. (2015)	Freeze drying	19.01
	Rotary drum drying	7.76
	Pyrolysis unit	10.21
	Hydrothermal Liquefaction	6.51
Minowa and Sawayama (1999)	Centrifuge	23
Xu et al. (2011)	Spray drying	80.9
Current study	Microwave drying	0.69 @100 W
		0.88 @300 W
		1.03 @600 W

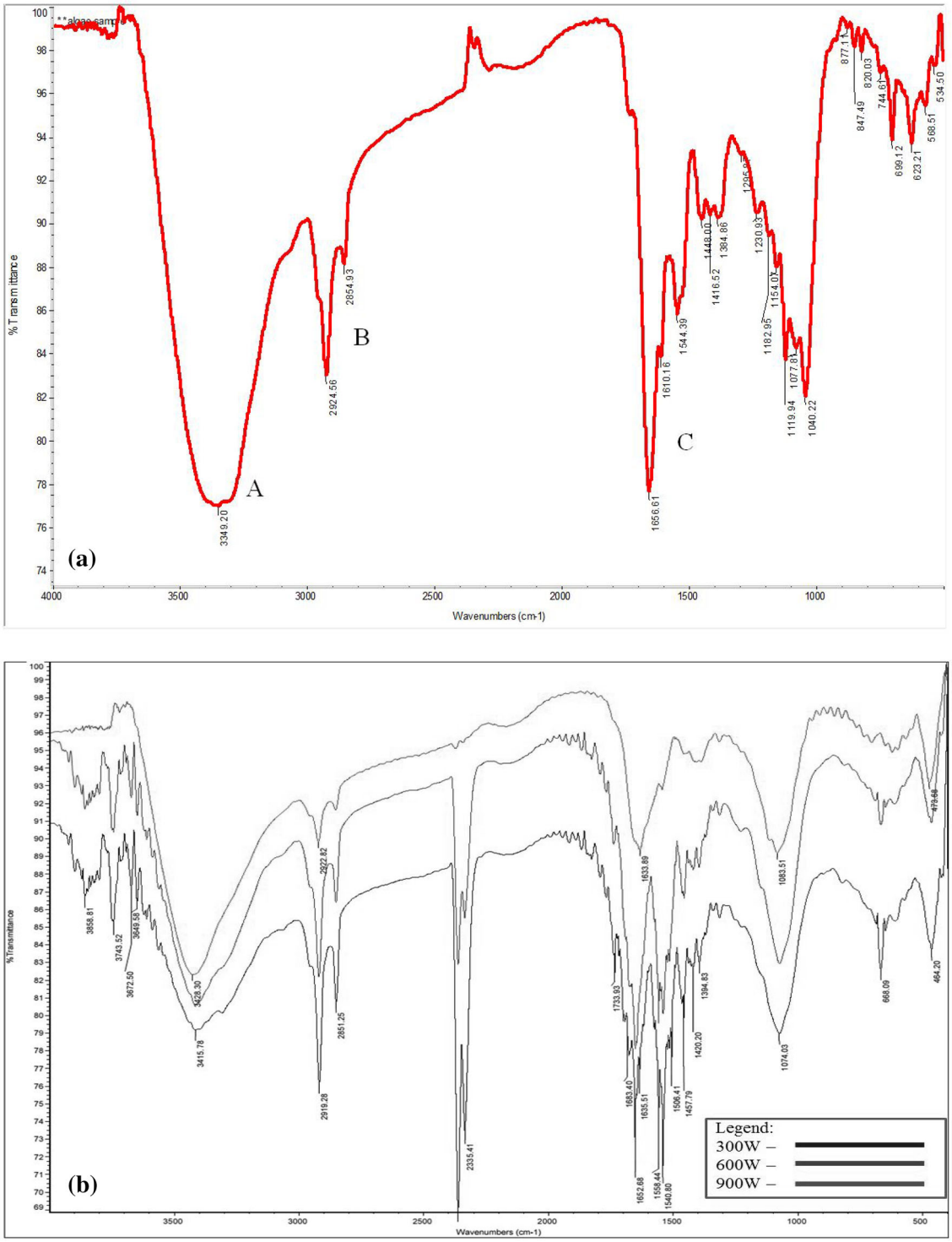

Fig. 6 FTIR spectroscopy of Chlorella vulgaris of **a** pre-drying and **b** post-drying at different microwave power. (*a* Water ν(O–H) stretching, Protein ν(N–H) stretching (amide A), *b* Lipid–hydrocarbons mainly due to C–H stretching, *c* Protein amide I band Mainly (C=O) stretching *d* Nitriles and Alkynes due to (C≡N) and (C≡C) adsorption.)

Fourier transform infrared spectroscopy results

The FTIR spectroscopy result for the undried sample is provided in Fig. 6a. The strong peaks at point C (1536 and 1422 cm^{-1}) corresponds to the bending modes of protein methyl groups. The intensity of the peak at point B (2924.56 cm^{-1}) suggests high content of lipid and carbohydrate in the sample indicating that *Chlorella vulgaris* is a

good biofuel feedstock. It is interesting to note that these spectral peaks are consistent with the findings of a similar investigation (Ponnuswamy 2013). Moreover, nitriles and alkynes intensity peaks appeared in the post-drying of microalgae.

To investigate the effects of microwave irradiation on the quality of the algal samples, IR spectra were also recorded after irradiation as shown in Fig. 6b. The quality of the algae was checked by monitoring the changes in the IR peak intensities, specifically on the lipid–hydrocarbons (2809–3639 cm^{-1}) and the protein amide (1583–3012 cm^{-1}) signals, at different power levels. Results clearly demonstrate a significant change in the IR spectra obtained after irradiation. Note that a lower percentage of transmittance is more desirable as this indicates higher lipid content, thus a better biofuel feedstock which corresponds to the power settings 20 and 15 W/g. Moreover, it shows that there is a higher percentage transmittance at higher specific power settings indicating a decrease in lipid content, most probably due to thermal degradation. This is particularly true for the 45 W/g setting where the percentage transmittance is higher than that of the undried sample as shown in Fig. 6a.

Conclusions

The effects of microwave drying on the cultivated microalgae *Chlorella vulgaris* were analysed for its suitability in biofuel production. Based from the FTIR spectroscopy results, the cultivated algal species has a high content of lipid and carbohydrate indicating that Chlorella vulgaris is a good source of biomass for biofuel production. Microwave drying results suggested the usage of power settings 20 W/g and 15 W/g in drying *Chlorella vulgaris* for biofuel production. Page model shows a good fit better than Newton's model for the drying curve. A mathematical model of the microwave drying curve was formulated as a function of microwave power and mass of the algae. Specific energy requirement and power level requirement were also modelled to obtain a 10 % moisture ratio. FTIR spectroscopy results revealed a significant reduction of infrared signal intensities as microwave power is increased. Further investigation will be pursued to quantify the algal oil, and the effects of microwave using Gas Chromatography Spectroscopy (GC–MS) method and microwave design for large-scale application.

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