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Experimental and modelling studies of convective and microwave drying kinetics for microalgae

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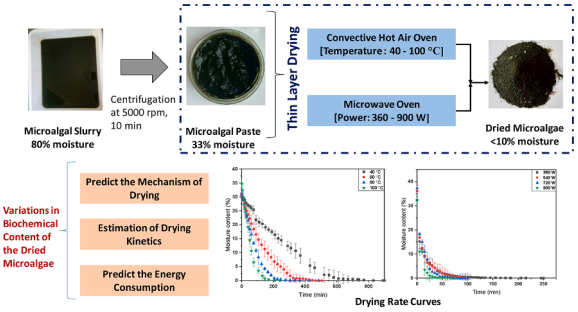
HIGHLIGHTS

- Algal drying characteristics of convective hot air and microwave oven were discussed.
- Microwave drying is faster with relatively lower specific energy requirements.
- Convective and microwave drying followed thin layer drying models.
- 14.4% higher lipid content was obtained in microwave dried algal biomass.
- Uniform volumetric heating in microwave drying preserves biochemical content.

GRAPHICAL ABSTRACT



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ABSTRACT

Conventional microalgal drying consumes huge time and contributes to 60–80% of downstream process costs. With the aim to develop an effective and rapid drying process, the present study evaluated the performance of microwave based drying (MWD) with a power range of 360–900 W and compared with the conventional oven drying (OD) at 40–100 °C. MWD was found to be efficient due to uniform and volumetric heating because of dipolar interaction, with an effective diffusivity of 0.47×10^{-9}–1.63×10^{-9} m^2 s^{-1}, comparatively higher than OD. Activation and specific energy of 32.43 W g^{-1} and 42.9–56.07 kWh kg^{-1} was projected respectively, and a falling rate period with best fit for Newton and Henderson-Pabis model was observed for MWD. Uniform heating from internal sub-surface avoided cell distress, resulting in 14.4% higher lipid yield and significant preservation of biochemical components that can be processed into bioenergy and valuable products in microalgal biorefinery.

1. Introduction

Microalgae have emerged as a promising feedstock for biofuel production in recent years through different biochemical and thermochemical processing routes (Mishra et al., 2019). These phototrophic solar bio-factories can sequester the atmospheric carbon dioxide (CO_2) into biochemically rich biomass that could be processed into several value-added products with simultaneous uptake and remediation of nutrients from wastewater (Nagarajan et al., 2020). Further, the algal cultivation facilities could be collocated inside industries providing fixation of flue gas generating additional revenues to the company (Behera et al., 2019a).

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Conversion of algal biomass into biofuel is a multi-step process involving cultivation, harvesting/dewatering i.e. separation of algal biomass from other media components, drying, cell disruption followed by a biochemical/thermochemical process conversion into biofuel (Dong et al., 2016). Apart from the upstream process of cultivation, the drying step is considered an essential intermediate point in the conversion of algal biomass into biofuel (Show et al., 2015). Also, the efficiency of the subsequent procedure for cell disruption, and the amount of solvent required during the biochemical conversion or the efficiency of the majority of thermochemical routes (pyrolysis, gasification) depends on the moisture content of algal biomass (Wang et al., 2021). Guldhe et al. (2014), reported that the quantity and viability of lipids during the transesterification is dependent on the quality of biomass obtained via drying since in-situ transesterification involving wet algal biomass is often inefficient. Dewatering and harvesting processes often reduce the moisture content of algal biomass to a level of 80%, which has to be brought to a level below 10% via drying to preserve the biochemical content of algal biomass, before subjecting them to any of the conversion routes (Al Rey et al. (2016)). Several researchers have evaluated the different drying techniques like solar and convective (Agbede et al., 2020), rotary bed (Silva et al., 2019) and spray drying (Biz et al., 2019) for dehydrating microalgal biomass. Conventional hot air oven-based drying involves an enormous amount of energy consumption i.e. almost 60–80% share of the total energy necessary during the conversion of algal biomass into biofuel (Agbede et al., 2020). Further, this process causes dehydration and shrinkage of cells, thus often negatively influences the functional and biochemical content of microalgae (de Farias-Neves et al., 2019). Solar drying though convenient and eco-friendly leads to unwanted contamination and is often time-consuming because of its dependency on weather conditions. Even though the above-mentioned issues can be overcome to certain extent via the use of closed solar dryers (Agbede et al., 2020), the process often results in a low yield of lipids compared to other drying process (Shekarabi et al., 2021). Culaba et al. (2013) experimented on drying characteristics of *Tetraselmis* sp. and reported charred biomass and uneven heating through solar radiation. Rotary, flash and spray drying though reported effective for several microalgae like *Spirulina* sp., (Silva et al., 2019), *C. pyrenoidosa* and *Nannochloropsis salina* (Ljubic et al., 2019), and *C. pyrenoidosa* (Biz et al., 2019), respectively however, the process is highly energy-intensive (Al Rey et al. (2016)).

Recently, microwave-based technology has gained attention as an efficient drying method due to more uniform and rapid volumetric heating caused by electromagnetic irradiation, without direct contact between the heating source and the sample (Teo et al., 2014). Microwave irradiation has been widely utilized to dry and preserve fruits in food industries (Feng et al., 2012). Even though exposure of cells to microwave irradiation as a pre-treatment or cell disruption procedure during solvent-based algal lipid extraction is a popular approach (Cheng et al., 2013; Sivaramakrishnan et al., 2020), the studies related towards the use of this approach for drying microalgal biomass are extremely limited. Mayol et al. (2015) varied the microwave power from 300 W to 900 W, and elucidated that 30 W g^{-1} was sufficient enough to obtain the bone dry mass for *C. vulgaris*. Al Rey et al. (2016) reported that microwave drying with 20 W g^{-1} of power setting could be a convenient approach to preserve the lipid content in *Chlorella vulgaris*. 76% drying efficiency could be achieved during the thermal treatment of 130 g *Nannochloropsis sp*., with 630 W microwave irradiation after 8.5 min, with no representation of the underlying drying kinetics (Mina et al., 2017). A recent study by Agbede et al. (2020) have analysed the drying kinetics of *Chlorella* sp., in a microwave oven by varying the power level in the range of 450 W − 700 W, and compared the drying kinetics with that of the process in open sun, solar dryer and convective hot air oven (40 °C − 70 °C). The study reported improved drying efficiency at higher temperature and power, but showed no effect of drying on quantitative lipid yields of microalgae. To the best of authors' knowledge, none of the studies done in literature so far, have altogether comparatively evaluated the convective and microwave drying based procedure for a mixed microalgal consortium in terms of the process efficiency considering the overall energy consumption and costs, along with the overall effects on the quantitative lipid yields. Also, in spite of the portrayed advantages of rapid heating rate, the industrial application of microwave drying technologies at large-scale commercial microalgal facilities is still at its nascent stage compared to convective dryers due to unresolved technological constraints. Studies related to analysing of the thin layer drying models for microwave based drying, are extremely limited, but is a necessary prerequisite for designing the industrial microwave dryers for microalgae.

The present paper thus, with the aim to bridge the knowledge gap on microwave assisted drying, analysed the thin-layer drying characteristics of a native mixed microalgal consortium via microwave assisted methods, with a power range of 360–900 W and further compared it with the commonly used convective hot air oven with a temperature variation of 40–100 °C. The performance of both the approach in two different independent instrumental set-up, in terms of the diffusivity constants, activation and, specific energy as well as costs was compared to evaluate their working efficiency. Further, the effect of both the methods over the lipid content of microalgae was evaluated and the characteristic changes in biochemical content, the functional groups were analysed through Fourier Transform Infrared Spectroscopy (FTIR). Such studies would aid in optimizing a suitable cost-efficient, eco-friendly drying technique to obtain biochemically rich algal biomass that can be processed into bioenergy and other valuable products in a sustainable algal biorefinery model.

2. Methodology

2.1. Microalgae cultivation and harvesting

A native mixed algal consortium mostly dominated by *Chlorella sp*., along with *Scenedesmus sp*., *Spirulina sp*., and *Synechocystis sp*., were collected from the wastewater ponds located at National Institute of Technology (NIT) Rourkela. The mixed consortium was grown under optimized culture conditions as described in the previous study by authors (Behera et al., 2020). At the end of an exponential growth phase of 10 days, the algal culture was harvested using the natural flocculants as detailed in the study by Behera et al. (2019b). The concentrated culture was further centrifuged and the cells obtained were evenly spread over a glass petri plate up to a 3 mm thin layer for subsequent drying experiments.

2.2. Drying of microalgal biomass

Thin layer drying of algal biomass was carried out in convective mode in a hot air oven with temperatures varying from 40 °C to 100 °C and in a domestic microwave oven (2450 kHz frequency) with the power level ranging from 360 W − 900 W. The convective drying was carried out using hot air oven (BD Instrumentation with inside cavity dimension of 450 mm [L] * 450 mm [B] * 500 mm [D]) employed with temperature sensor at the centre and parallel hot air velocity of 1 m s^{-1} at drying temperature ranging from 40 °C to 100 °C. The microwave assisted drying was conducted using domestic microwave oven (Electrolux C25K151 BG-CG) with rated output power of 900 W, having a frequency of 2450 Hz. The cavity dimensions of the microwave are of 190 mm (H) * 310 mm (L) * 300 mm (W).

An aliquot of 21.939 ± 0.35 g was evenly spread on a glass petri-plate with circular cross section having a diameter of 50 mm. The mass of the microalgae paste after being spread on the petri-plate with 3 mm thickness was measured before the commencement of the dehydration using a Shizmadu digital weighing balance (0.001 accuracy). The instantaneous weight during the drying process was measured every 5 min for an initial 30 min, followed by 10 min intervals up to the time until a constant weight i.e. bone dried biomass was obtained. The thin

layer drying protocol followed was similar to standard procedure as detailed in the ASABE Standard S448.1 (ASABE Standards, 2006). The storage and transfer of biomass for any measurement was carried out inside a desiccator having desiccant at the bottom to avoid error in mass loss measurements. Similar, process has also been employed in the study by Al Rey et al. (2016). The experimental mass-time data was then converted into moisture-time data employing the drying equations. The drying parameters were then fitted into the model equations as mentioned to calculate the drying kinetics using Microsoft Office Excel 2016 (Version 16.0.4266.1001) which also has been used by Rueda-Ordonez and Tannous, (2018) to calculate drying kinetics of sugarcane straw.

The initial moisture content of the microalgal slurry was 79.43 ± 0.54%. The slurry was centrifuged at 5000 rpm for 10 min, reducing the moisture content of the wet microalgal pellet moisture content to 33.13 ± 2.31%.

The sources of uncertainty in the present study could be linked with the deviations associated during the measurement of weight of sample in the analytical balance, the variation in the performance of hot air oven due to changes associated with the air velocity and drying temperature, as well as fluctuations in the power setting of microwave. Uncertainty analysis as detailed by the Holman, (1994) has now been included for each of the parameter calculated for estimating the drying performance. The drying data presented in this study represents the mean along with the range of standard deviation to ensure the level of uncertainty.

2.3. Data analysis and mathematical modelling

The data obtained from the drying experiments were analysed and the drying parameters were calculated based on the set of equations as mentioned in the subsequent sections to compare the performance of convection-based and microwave-based drying techniques. Further, the data were fitted into a mathematical model to study the drying kinetics of microalgae.

2.3.1. Evaluation of moisture ratio, effective diffusivity, and drying rate

The instantaneous moisture content of microalgae was calculated by converting the mass versus $X_t = \frac{m_t - m_o}{m_o}$ time data recorded into moisture content variation with time. The moisture content of algal biomass (X_t) (dimensionless) after a time t in min was calculated using Eq. (1).

$$X_t = \frac{m_t - m_o}{m_o} \tag{1}$$

where m_t represents the mass of microalgae (g) after a time (t) [min], m_o represents the mass of the absolutely dried microalgae (g).

The drying rate of microalgae (D_R) can be calculated from the moisture content of microalgae by employing Eq. (2).

$$D_R = \frac{X_{t+\Delta t} - X_t}{\Delta t} \tag{2}$$

where, $X_{t+\Delta t}$ is the moisture content of microalgae after a time $(t + \Delta t)$in min and Δt is the increment in time (min).

Conveniently, the moisture content could be converted and expressed as a dimensionless number termed as moisture ratio (MR) (ratio of water to be removed at any time (t) to total water available initially) (Eq. (3)) utilizing the initial and equilibrium moisture content represented by X_{t0} and X_e respectively.

$$MR = \frac{X_t - X_e}{X_{to} - X_e} \tag{3}$$

Considering long drying time, X_e will be negligible compared to X_t and X_{t0}, the above equation could be reduced to a simpler form (Eq. (4)) as given below.

$$MR = \frac{X_t}{X_{to}} \tag{4}$$

During the thin layer drying the loss of moisture occurs from the internal microalgal surface towards the outside over time, thus following Fick's law of diffusion. Assuming internal mass transfer as the dominant mechanism, the variation in MR as a function of time ($\frac{dMR}{dT}$)could be expressed in the form of Eq. (5) (Vega-Galvez et al., 2010).

$$\frac{dMR}{dT} = D_{eff}\frac{d^2MR}{dx_2} \tag{5}$$

where D_{eff} represents the effective diffusivity expressed in the units of $m^2\ s^{-1}$ and x denotes the spatial distance in m.

The glass petri-plate utilized for drying the algal paste had a circular cross-sectional area. Assuming an internal mass transfer mechanism with a uniform diffusivity, limited cell shrinkage, and external resistance, Eq. (5) could be solved according to the formula (Eq. (6)) as provided by Crank, (1975).

$$MR = \frac{8}{\pi^2}\sum_{i=0}^{\infty}\frac{1}{(2i+1)^2}\exp\left[-\frac{(2i+1)^2 D_{eff}\pi^2 t}{4L^2}\right] \tag{6}$$

where, L is the half-thickness (m) and t is the drying time (min). Assuming longer drying times, the first part of the equation provides a good approximation, thus Eq. (6) can be simplified in the form of Eq. (7) as

$$MR = \frac{8}{\pi^2}\exp\left[-\frac{D_{eff}\pi^2 t}{4L^2}\right] \tag{7}$$

The above expression could be linearly written in the form of Eq. (8).

$$\ln(MR) = \ln\left(\frac{8}{\pi^2}\right) - \left[\frac{D_{eff}\pi^2 t}{4L^2}\right] \tag{8}$$

Thus, from the above-mentioned equation the slope of the straight line obtained by plotting $ln(MR)$ versus t (min), provides the value for effective diffusivity (D_{eff}) ($m^2\ s^{-1}$).

2.3.2. Evaluation of activation energy and specific energy

The effective diffusivity that governs the rate of drying is dependent on the temperature, which can be described based on the Arrhenius equation (Tunde -Akintunde and Ogunlakin, 2011; Doymaz and Ismail, 2011) (Eq. (9)). This equation provides a relationship between the effective diffusivity (D_{eff}) [$m^2\ s^{-1}$] and the Arrhenius factor (D_o) ($m^2\ s^{-1}$) as a function of the activation energy (E_a)(kJ mol^{-1}) and the temperature (T) (K).

$$D_{eff} = D_o\exp\left(-\frac{E_a}{RT}\right) \tag{9}$$

where R represents the universal gas constant (8.314 J $mol^{-1}\ K^{-1}$).

Transforming Eq. (9) into a linear form (Eq. (10)) considering logarithm on both sides, the activation energy (E_a) (kJ mol^{-1}) can be determined from the slope of straight line on the plot of $ln(D_{eff})$ and 1/T.

$$\ln(D_{eff}) = \ln(D_o) - \frac{E_a}{RT} \tag{10}$$

Similar to the drying studies in the oven, in the case of microwave drying process, a modified Arrhenius reaction (Olanipekun et al., 2015) (Eq. (11)) can be utilized to study the activation energy (E_a) [W g^{-1}].

$$D_{eff} = D_o\exp\left[\frac{E_a m}{P}\right] \tag{11}$$

where, D_o is the Arrhenius factor [$m^2\ s^{-1}$], P is the microwave power (W), m is the weight of the algal sample (g).

The activation energy (E_a) [W g^{-1}] can be calculated from the slope of the straight line obtained via plotting $ln(D_{eff})$ versus m/P (Eq. (12))

$$\ln(D_{eff}) = \ln(D_o) - \frac{E_a m}{P} \tag{12}$$

The specific energy (E_{SO}) [kWh kg^{-1}] can be calculated from the total energy (E_{TO})[kWh] as mentioned in Agbede et al. (2020) using Eq. (13)

$$E_{SO} = \frac{E_{TO}}{m_i - m_d} \quad (13)$$

where, m_i represents the initial mass of microalgae (g) and m_d denotes the absolutely dried algal biomass (g).

The specific energy (E_{SM}) [kWh kg^{-1}] can be computed from the total energy consumed in the microwave oven (E_{TM}) [kWh] as mentioned in Agbede et al. (2020) considering the initial algal biomass (m_i) [g] and the absolutely dried algal biomass (m_d) [g] using Eq. (14).

$$E_{SM} = \frac{E_{TM}}{m_i - m_d} \quad (14)$$

2.3.3. Curve fitting and modelling of the algal drying process in hot air and microwave oven

The data obtained from the drying experiments in a hot air oven at different temperatures (40–100 °C) and microwave oven at a power range of 360–900 W were fitted into the Page, Newton and Henderson-Pabis models respectively as represented in Eq. (15), (16) and (17) respectively.

$$MR = \frac{X_t - X_e}{X_{to} - X_e} = \exp(-k_1 t^n) \quad (15)$$

$$MR = \frac{X(t) - X_{eq}}{X_o - X_{eq}} = \exp(-k_2 t) \quad (16)$$

$$MR = \frac{X(t) - X_{eq}}{X_o - X_{eq}} = A\exp(-k_3 t) \quad (17)$$

where k_1, k_2, k_3 represent the drying constant for the respective equation, n represents the Page constant and A is constant related to the shape of biomass spread.

The coefficient of determination (R^2), the sum of square error (SSE) (Eq. (18)), root mean square error ($RMSE$) (Eq. (19)), and *Chisquare* (Eq. (20)) were used to determine the goodness of fit for each of the models utilized in two different drying set-ups.

$$SSE = \frac{1}{N}\sum_{i=1}^{N}(MR_{Exp,i} - MR_{Pred,i})^2 \quad (18)$$

$$RMSE = \frac{1}{N}\sum_{i=1}^{N}[(MR_{Exp,i} - MR_{Pred,i} \quad (19)$$

$$Chisquare = \frac{\sum_{i=1}^{N}(MR_{Exp,i} - MR_{Pred,i})^2}{N - z} \quad (20)$$

where, $MR_{Exp,i}$, and $MR_{Pred,i}$ represents the experimental and predicted values of moisture ratio respectively. N and z denote the number of observations and number of constants respectively.

2.4. Influence of convective and microwave drying on microalgal lipids

The lipid content of the oven-dried and the microwave-dried sample at each temperature and power level was estimated using Bligh and Dyer Method (1959) with slight modifications. 1 g of dried sample was mixed with 10 ml of Chloroform: Methanol (1:2 ratio). The sample was homogenized for 2 min, then soaked for 20 min, followed by the addition of 5 ml of Chloroform: Methanol (1:2 ratio) mixture. The samples were centrifuged at 5000 rpm for 5 min. The supernatant was transferred to a separate Eppendorf tube and mixed with 6 ml of Chloroform: Methanol (1:2 ratio) mixture along with 1.6 ml of 1% Potassium chloride solution. The mixture was vortexed for 2 min followed by centrifugation at 5000 rpm for 5 min. The supernatant was again transferred to a separate tube, mixed with 4 ml of Chloroform: Methanol (1:2 ratio) mixture along with 2.4 ml of 1% potassium chloride, and was kept stagnant until two separate phases were formed. The chloroform phase or the lower layer was pipetted out and passed through a sterile filter into a pre-weighed vial (W_1). The chloroform phase was allowed to evaporate and the final weight of the vial with lipids (W_2) was noted. The lipid content in % dry cell weight (Dcw) was obtained by subtracting W_1 from W_2.

2.5. Effect of drying method on chemical components and functional groups of microalgae

The functional groups and other biochemical compounds present in the microalgal biomass showing the maximal lipid content post drying were analysed via Fourier Transform Infrared Spectroscopy (FTIR). The scan rate was kept at 400 cm^{-1} to 4000 cm^{-1} in attenuated transmission mode to detect the peaks corresponding to specific biochemical constituents.

3. Results and discussion

3.1. Drying characteristics

3.1.1. Hot air oven drying characteristics

The plot of moisture content versus time concerning drying at different temperatures in the conventional hot air oven has been illustrated in Fig. 1a. It could be inferred that the moisture content of microalgal paste with 3 mm thickness dried at all temperature decreases with time. The moisture ratio (MR) was also found to decline with temperature, thus indicating that increased drying air temperature promotes the drying rate of microalgae where less time is enough to complete the drying process at higher temperatures. For instance, the moisture ratio reached 0 after 740 ± 20 min when dried at 40 °C however, the drying time of 183 ± 23 min was enough to reach MR of 0 at 100 °C. The time taken for complete drying of microalgal paste was 458 ± 11 min and 405 ± 34 min at 60 °C and 80 °C respectively. Agbede et al. (2020) reported a decrease in drying time from 350 min to 190 min for *Chlorella sp.*, dried in a convective hot air oven with increase in temperature from 50 °C to 70 °C. Similar conclusions were also observed for *C. pyrenoidosa* by Damayanti et al. (2020) where a stabilized moisture content was reported after 1140 min via heating at 50 °C which declined to 640 min at 70 °C. With an increase in treatment temperature, more heat energy is being utilized resulting in an increase in water activity, thereby improving the drying behaviour in shorter time-period (Agbede et al., 2020). The difference in minimum temperature required to achieve a stable moisture content below 10% as observed in the above-mentioned studies might be attributed to the differences in the initial moisture content of the sample, apart from other instrumental operational conditions. Further, non-linear relationships were observed between the moisture ratio and drying time in hot air oven as represented in Fig. 1b. This might be because of the heterogeneous distribution of initial moisture, shrinkage of the product, and the temperature changes in the microalgal sample under different drying conditions. A similar trend of decline in moisture ratio with the progress of drying was also observed by Agbede et al. (2020) for *Chlorella sp.*, in a convective hot air oven.

The effect of drying rate with respect to time at different temperatures in the hot air oven has been given in Fig. 1c. The drying rate of microalgae in hot air oven was found to increase with drying temperature as expected, however, it was observed to decrease with an increase in drying time. This could be corroborated with the fact that at a higher temperature, the temperature difference between the microalgae and surrounding medium (air) increases the heat transfer co-efficient which enhances the heat and mass transfer rate (Agbede et al., 2020). After a time of saturation, the diffusivity of moisture is limited at all temperatures resulting in a decrease of drying rate and this corresponds to the falling rate period. During the falling rate period, often a decline in moisture content of the solid sample increases the internal resistance to

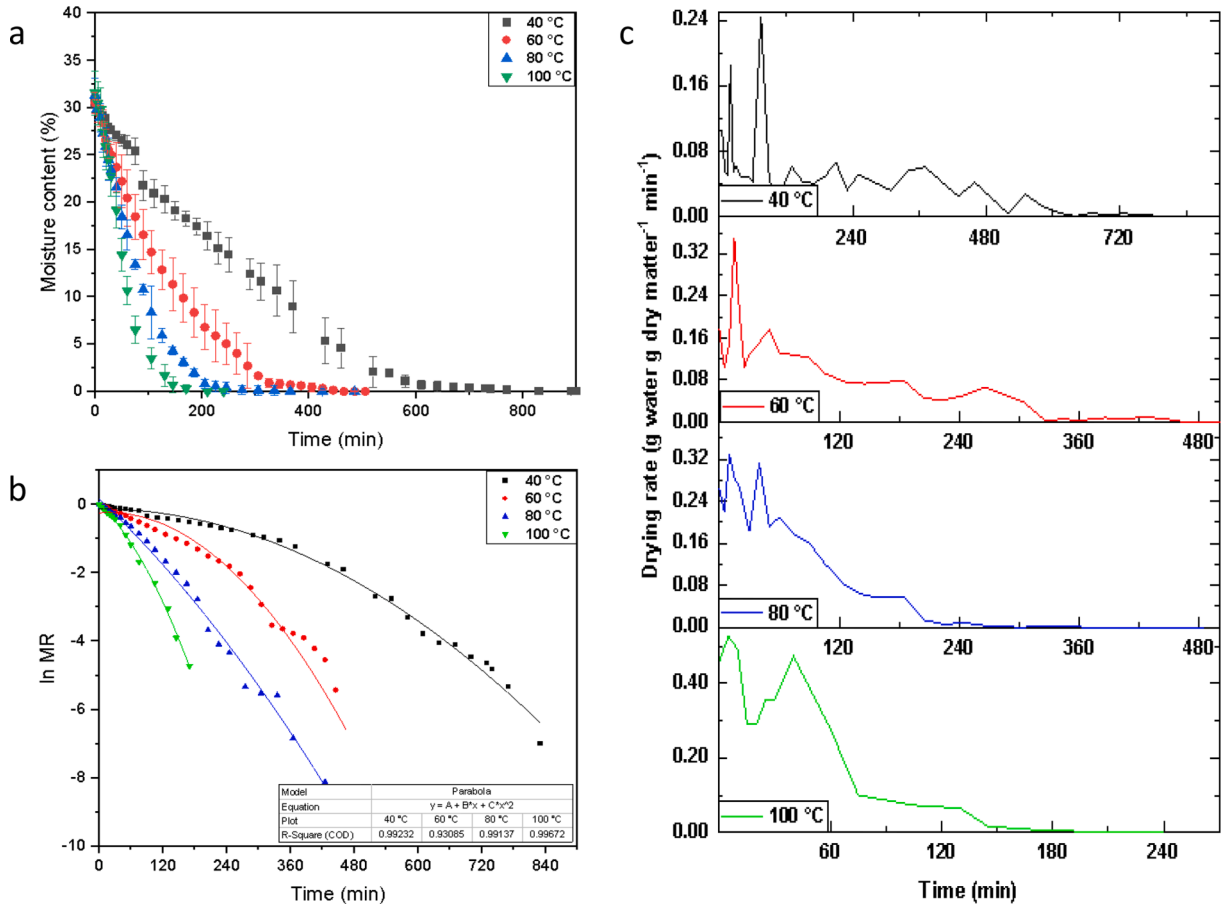

Fig. 1. a). Effect of drying time on moisture content of microalgae; b). Plot of ln *MR* vs drying time for different temperatures; c). Drying rate curves of microalgae at different temperatures in hot air oven.

the migration of moisture (Simioni et al., 2019). In the present study, the drying rate curve at all the temperatures in the conventional hot air oven depicted the falling rate period rather than the constant rate period. Similar to the present study, Viswanathan et al. (2011, 2012) also reported the drying of a mixed microalgal consortium in a convective hot air oven to be influenced by diffusion and following a falling rate period. Simioni et al. (2019), during the drying of *Scenedesmus sp.*, in a convective hot air oven at 50 °C and 60 °C, with an air velocity of 1–2 m s^{-1} described the drying curve to consist of an initial warm up period where the bulk water was evaporated followed by a sharp variation in moisture content with temperature and subsequently the falling rate period. Similar falling rate drying curves for *Spirulina sp.*, was also observed in the studies by Oliveira et al. (2009) affirming the mechanism of drying in a convective oven to be governed by moisture diffusion phenomenon.

The moisture diffusivity is a characteristic of mass transfer of inherent moisture that includes molecular diffusion and a few other mass transport mechanisms (Show et al., 2015). The measured effective moisture diffusivity for the microalgae dried under hot air oven was found to increase from $0.94 \times 10^{-10} \pm 0.77 \times 10^{-11}$ m^2 s^{-1} at 40 °C to $4.13 \times 10^{v10} \pm 0.45 \times 10^{-10}$ m^2 s^{-1} at 100 °C as shown in Table 1 The values reported are of the similar order of magnitude as obtained in the study by Simioni et al. (2019). Hosseinizand et al. (2018) also obtained ten times higher effective moisture diffusivity for *Chlorella* sp. biomass, when the drying temperature is increased from 40 °C to 140 °C in a hot air oven. Even-though the effective diffusivity increases with increase in

Table 1
Effective diffusivities, activation and specific energy for convective and microwave drying of microalgae.

	Hot air oven drying				Microwave drying		
Drying Temperature (°C)	Effective Diffusivity (D_{eff}) (m^2 s^{-1})	Specific Energy (E_{SO}) (kWh kg^{-1})	Activation Energy (E_a)(kJ mol^{-1})	Microwave Power (W)	Effective Diffusivity (D_{eff}) (m^2 s^{-1})	Specific Energy (E_{SM}) (kWh kg^{-1})	Activation Energy (E_a) (W g^{-1})
40	$0.94 \times 10^{-10} \pm 0.77 \times 10^{-11}$	58.27 ± 2.4	24.37 ± 2.33	360	$0.47 \times 10^{-9} \pm 0.13 \times 10^{-9}$	56.07 ± 2.54	32.43 ± 2.7
60	$1.64 \times 10^{-10} \pm 0.51 \times 10^{-10}$	55.79 ± 2.61		540	$0.72 \times 10^{-9} \pm 0.24 \times 10^{v9}$	53.58 ± 2.6	
80	$2.90 \times 10^{-10} \pm 0.14 \times 10^{-10}$	51.06 ± 1.82		720	$1.15 \times 10^{-9} \pm 0.36 \times 10^{-10}$	49.63 ± 2.48	
100	$4.13 \times 10^{-10} \pm 0.45 \times 10^{-10}$	47.39 ± 2.23		900	$1.63 \times 10^{-9} \pm 0.76 \times 10^{-10}$	42.98 ± 3.4	

Table 2
Statistical results of thin layer models for convective drying of microalgae.

Temperature (°C)	Model	Model Constants	R^2	*RMSE*	*SSE*	*ChiSquare*
40	Page	$n = 1.1608$				
$k_1 = 0.0016$	0.9743	0.0561	0.0031	0.00332		
	Newton	$k_2 = 0.0062$	0.9412	0.1508	0.0227	0.02337
	Henderson-Pabis	$k_3 = 0.0062$				
$A = 1.4425$	0.9412	0.1861	0.0346	0.03661		
60	Page	$n = 1.1788$				
$k_1 = 0.0033$	0.9849	0.0625	0.0039	0.00416		
	Newton	$k_2 = 0.0104$	0.9661	0.0881	0.0077	0.01164
	Henderson-Pabis	$k_3 = 0.0104$				
$A = 1.2925$	0.9661	0.1036	0.0107	0.01689		
80	Page	$n = 1.1847$				
$k_1 = 0.0054$	0.9958	0.0181	0.0003	0.00036		
	Newton	$k_2 = 0.0191$	0.9862	0.1146	0.0131	0.01367
	Henderson-Pabis	$k_3 = 0.0191$				
$A = 1.4500$	0.9862	0.1430	0.0204	0.02217		
100	Page	$n = 1.2858$				
$k_1 = 0.0060$	0.9916	0.0763	0.0058	0.00659		
	Newton	$k_2 = 0.0272$	0.9800	0.1159	0.0134	0.01428
	Henderson-Pabis	$k_3 = 0.0272$				
$A = 1.3185$	0.9800	0.1097	0.0120	0.01364		

*k_1, k_2, k_3 represent the drying constant for the respective model, n represents the Page constant and A is constant related to the shape of biomass spread. R^2, RMSE, SSE represents regression coefficient, root mean square error and sum of square error respectively

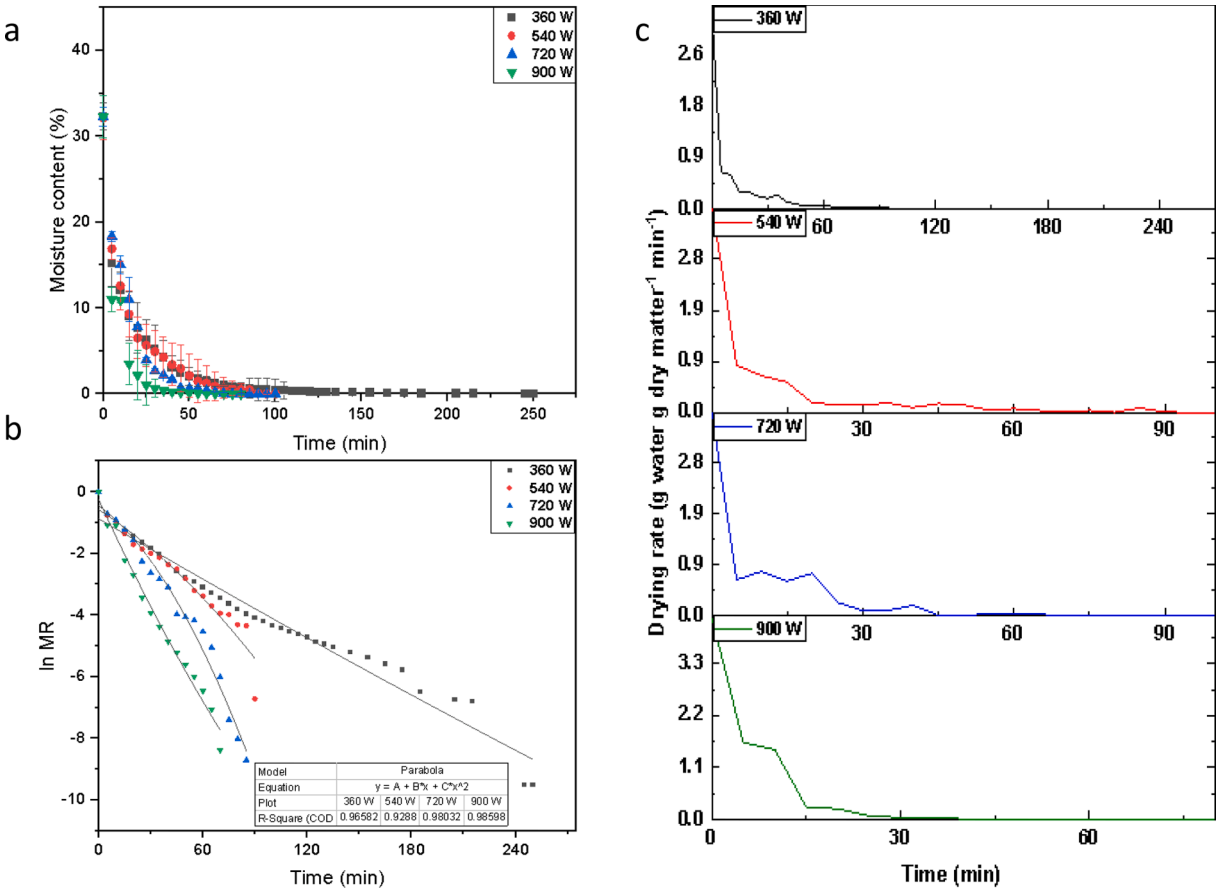

Fig. 2. a). Effect of drying time on moisture content of microalgae; b). Plot of ln *MR* vs drying time for different microwave power; c). Drying rate curve of microalgae for different power in microwave oven.

temperature, the magnitude of moisture diffusion from microalgal sample is relatively low because of the cell shrinkage (Viswanathan et al. 2011).

The activation energy (E_a)required for drying microalgae in a hot air oven was estimated by plotting the graph between $ln(D_{eff})$ and 1/T (see supplementary material). The activation energy was found to be 24.37 ± 2.33 kJ mol^{-1} and, this represents the minimum energy needed to overcome the barrier for the drying process to occur. E_a found in this study is similar to the activation energy of 24.81 kJ mol^{-1} for convective drying of *Chlorella vulgaris* as observed by Hosseinizand et al. (2018). The activation energy obtained in this study was also found to be lower than 28.7 kJ mol^{-1} for *Chlorella* sp., as reported by Agbede et al. (2020) and 31.8 kJ mol^{-1} for a mixed microalgal consortium represented by Viswanathan et al. (2011) dried in a convective hot air oven. The variation obtained might be attributed to the differences in instrumental operational setting of the convective heating process. The value obtained in the present study is also in range with those reported for the agricultural products based convective hot-air drying process (Erbay and Icier, 2010).

3.1.1.1. Microwave drying characteristics. The effect of drying time on moisture content of microalgal samples under the influence of microwave radiation varying from 360 W to 900 W has been given in Fig. 2a. As evident from the above-mentioned figure, the moisture removal rate was increased with an increase in time and microwave power. The rate was found to be higher compared to convective drying, thus less time was needed to achieve significant moisture removal. At 360 W, 137.5 ± 24 min was needed to remove the moisture, while only 40 min was required to achieve an equilibrium moisture content at 900 W, which was relatively lower than that required during hot air oven drying. Uniform and volumetric heating of microwave radiation improvise the drying process, thereby resulting in the removal of moisture at an enhanced rate (Al Rey et al. (2016)). Also, at higher microwave power setting, more amount of heat is generated due to the frictional movement of dipoles, resulting in a more rapid evaporation of moisture from inside to outside (Agbede et al., 2020). Compared to the conventional hot air oven, a relatively linear relationship was observed in the case of microwave drying between the moisture ratio and the drying time as represented in Fig. 2b. This might be attributed to a more uniform and volumetric heating rate caused by exposure to microwave irradiation. Similar trends in moisture ratio and drying time were also projected in the studies by Viswanathan et al. (2011, 2012) and Agbede et al. (2020).

From the drying rate curve shown in Fig. 2c, it is evident that there was almost no or a very short initial activation or warming-up stage in the case of microwave-assisted drying process, compared to the convective hot air oven based drying. This signifies the fact that a much faster drying rate could be obtained under the influence of microwave irradiation with less energy required for initiating the drying via crossing the energy barrier (Agbede et al., 2020). The drying rate mostly followed a falling rate period being dependent on the diffusion of moisture from inside to outside surface of microalgae as similarly reported in the study by Viswanathan et al. (2011). The rate of drying under the microwave was found to be much faster compared to the hot air oven, which could be attributed to higher temperature induced by volumetric heating that possibly promoted better diffusivity of moisture. The measured effective moisture diffusivities were found to increase with the increase in microwave power level from $0.47 \pm 0.13 \times 10^{-9}$ m^2 s^{-1} at 360 W to $1.63 \times 10^{-9} \pm 0.76 \times 10^{-10}$ m^2 s^{-1} at 900 W as shown in Table 1. The gradual decline in moisture content at higher power increases the permeability of vapours from the porous surface of the microalgal sample. In the initial stage of drying, the product temperature increases rapidly and possesses a higher loss factor which decreases with moisture content. This in turn increases the water vapour pressure inside, resulting in the pressure-induced opening of pores. Thus, liquid diffusion of moisture might be the key mechanism of moisture transport during the initial stage of microwave-assisted drying, whereas vapour diffusion would probably be predominant during the latter part of the process (Agbede et al., 2020). Furthermore, the effective diffusivity in case of drying carried out in the microwave oven was found to be higher compared to that of the conventional oven, owing to a faster heating rate due to the phenomenon of more uniform loss of water from the inside to the outside subsurface (Teo et al., 2014). To the best of author's knowledge, most research until now on microwave assisted drying of microalgae have not much focussed on the aspects of effective diffusivity during microwave assisted drying, except the recent study by Agbede et al. (2020). The authors have reported an effective diffusivity ranging from 1.83×10^{-9} m^2 s^{-1} to 3.91×10^{-9} m^2 s^{-1} during the microwave drying of 5 mm thick layer of *Chlorella sp.*, with power levels varying from 450 W to 700 W. The activation energy (E_a) for the microwave drying process was determined from plot of m/P and $ln(D_{eff})$ (see supplementary material) and found to be 32.43 ± 2.7 W g^{-1} in the present study, which was higher than the value of 21.4 W g^{-1} for microwave based drying of *Chlorella* sp., as projected by Agbede et al. (2020). The difference might be due to the variation in the thickness of algal paste as well as the power intensities of microwave utilized in both the studies.

3.2. Modelling the drying kinetics of conventional and microwave oven

The drying data obtained for different temperatures and power used in the convective hot air oven and the microwave oven respectively were fitted into the three most common models (i.e.) i). Page model ii). Newton model and the iii). Henderson-Pabis model utilized for studying the drying kinetics.

3.2.1. Drying kinetics of microalgae in conventional hot air oven

Table 3 shows the curve fitting results and statistical inference obtained by kinetic modelling of the experimentally obtained drying data at different temperatures in the hot air oven. In general, the drying data fitted well into all the three models considered for the study with (R^2) almost equal to or more than 0.95, with a low *RMSE*, as evident from the above-mentioned table. At all the selected temperatures, the Page model was found to provide the best fit statistic results at a 95% confidence interval in terms of higher R^2 and low *RMSE*, *SSE* and *Chisquare*. Regression coefficient (R^2) of 0.9743, 0.9849, 0.9958, 0.9916 with *RMSE* of 0.056, 0.0625, 0.0181, 0.0763 was obtained at temperature value of 40 °C, 60 °C, 80 °C and 100 °C respectively. There was no significant difference between the statistical estimates provided by Newton model and the Henderson-Pabis model as highlighted by the same regression coefficient observed in both cases ($p < 0.05$). The Page model describes the drying characteristics as a function of the air velocity and the thickness of the microalgal paste (Hosseinizand et al., 2018). Thus, with an increase in temperature as evident from Table 2, the value of the constant k_1 increases, showing an enhanced and faster drying rate at a higher temperature. Similarly, the constant k_3 obtained from the Henderson-Pabis model corresponds to the effective air diffusivity. An increase in the value of k_3, with the increase in oven temperature, can be ascribed to the enhancement in the effective diffusivity, thereby resulting in an efficient drying mechanism. The study by Hosseinizand et al. (2018) with the thin layer drying of *Chlorella vulgaris* in a hot air oven having a temperature variation of 40 °C – 140 °C also showed a better fit of the data with the Page model compared to the Newton and Henderson-Pabis model. Best fit with R^2 of 0.99 and *SSE* varying from 0.28 to 0.04 was projected for the Page model, compared to the Newton and Henderson-Pabis model for a mixed microalgal consortium dried in a convective dryer with temperature varying from 30 °C to 90 °C by Viswanathan et al. (2012). Also, the convective oven based drying parameters of the green mixed microalgal consortium studied by Viswanathan et al. (2011) showed better predictive results with the Page model compared to several other kinetic models described in the study.

Table 3
Statistical results of thin layer models for microwave drying of microalgae.

Microwave Power (W)	Model	Model Constants	R^2	*RMSE*	*SSE*	*ChiSquare*
360	Page	$n = 0.6430$				
$k_1 = 0.2227$	0.9865	0.0776	0.0060	0.00017		
	Newton	$k_2 = 0.0314$	0.9655	0.1458	0.0212	0.02250
	Henderson-Pabis	$k_3 = 0.0314$				
$A = 0.3922$	0.9655	0.1039	0.0107	0.01175		
540	Page	$n = 0.6713$				
$k_1 = 0.2218$	0.9535	0.0946	0.0089	0.00047		
	Newton	$k_2 = 0.0475$	0.9828	0.1236	0.0152	0.01730
	Henderson-Pabis	$k_3 = 0.0475$				
$A = 0.6054$	0.9828	0.0879	0.0077	0.01891		
720	Page	$n = 0.8300$				
$k_1 = 0.1510$	0.9748	0.1696	0.0287	0.00151		
	Newton	$k_2 = 0.0938$	0.9582	0.0345	0.0011	0.00490
	Henderson-Pabis	$k_3 = 0.0938$				
$A = 1.2918$	0.9582	0.0968	0.0093	0.01725		
900	Page	$n = 0.9131$				
$k_1 = 0.1664$	0.9733	0.3971	0.1577	0.01051		
	Newton	$k_2 = 0.1070$	0.9844	0.0655	0.0042	0.00456
	Henderson-Pabis	$k_3 = 0.1070$				
$A = 0.6600$	0.9844	0.0877	0.0076	0.00872		

*k_1, k_2, k_3 represent the drying constant for the respective model, n represents the Page constant and A is constant related to the shape of biomass spread. R^2, RMSE, SSE represents regression coefficient, root mean square error and sum of square error respectively.

3.2.1.1. Drying kinetics of microalgae in the microwave oven. The curve fitting results including the drying constants and statistical parameters, for the thin layer drying done at different power combinations in a microwave oven using various models, are presented in Table 3. As evident from the table, the drying data obtained at distinct power levels fitted well in all the selected models. The drying data at 360 W showed the best fit with the Page model showing R^2 of 0.9865, with *RMSE* of 0.0776 and *SSE* of 0.0060. For 720 W, even-though highest R^2 was obtained with the Page model, Newton model with R^2 of 0.9582 and *RMSE* of 0.0345, and *SSE* of 0.0011 can be considered as the best fit. There was no significant difference between the best fit results at different power settings for the Newton and the Henderson-Pabis model which showed a similar R^2 in all the cases. However, the drying curve fit at 540 W showed a lower *RMSE* and *SSE* with the Henderson-Pabis model compared to the Newton model, while at 900 W, the Newton model showed the least *RMSE* and *SSE*. The drying constants for microwave drying for Page model did not show a consistent increase with an increment in power levels, however, an increasing trend was seen for the drying constants in the case of the Newton and Henderson-Pabis model. Thus, it can be concluded that Newton and Henderson-Pabis models describe the mechanism of microwave-based drying with that of the increase in power, better than that of the Page model. The best fit obtained for a set of drying data is dependent on the mechanism of the drying process involved. In convective drying in a hot air oven, the drying rate is the function of the air velocity and thickness of the microalgal paste, thus could be appropriately described by the Page model (Hosseinizand et al., 2018). The microwave drying process involves non-surface heating, where the heat energy propagates within the entire surface (Teo et al., 2014), which is dependent on the effective diffusivity constant. As microwave irradiation causes volumetric water evaporation from inside to outside, and thus could be better described mostly by the Henderson-Pabis model.

3.3. Comparative evaluation of the specific energy consumption, costs involved, and the associated emissions

The specific energy consumption (kWh kg^{-1} of water removed) was evaluated under different temperatures and power consumption during the drying in a hot air oven and microwave oven respectively as represented in Table 1. The specific energy consumption for hot air oven ranged between 47.39 kWh kg^{-1} to 58.27 kWh kg^{-1}, whereas in the case of microwave oven, the specific energy was found to be within the range of 42.98 kWh kg^{-1} to 56.07 kWh kg^{-1}. It was seen that with an increase in temperature or power in the hot air oven and the microwave oven respectively, the specific energy consumption was found to decrease, due to a more efficient and faster drying rate. Furthermore, the specific energy consumption was found to be relatively lower in the microwave oven compared to that of the hot air oven, due to more uniform and faster heating (Agbede et al., 2020). Assuming the cost of 1 kWh electricity to 0.075 US $, a total cost for hot air oven drying will be ranging between 3.55 US $ kg^{-1} to 4.37 US $ kg^{-1}. Also, a similar amount of costs will be incurred varying from 3.22 US $ kg^{-1} to 4.20 US $ kg^{-1} in the case of microwave drying. The drying process in a microwave oven will have a net greenhouse gas (GHG) emission of 40.83–53.26 kg (CO_2) equivalent per kg of water removed, compared to the 45.02–55.36 kg (CO_2) equivalent per kg of water removed in hot air oven (Assuming 0.95 kg [CO_2] equivalent per kWh of electricity consumption). Thus, it could be concluded that microwave drying is an eco-friendly and efficient process requiring less time, specific energy emissions as well as costs, compared to conventional oven drying.

It is noteworthy to mention that the microwave assisted drying of microalgae in the present study was as efficient as that of other drying techniques. Similar to the drying data obtained in the present study, Stramarkou et al. (2017) investigated the freeze drying efficiency of *C. vulgaris* subjected to −30 °C, 3 mBar pressure for 4.5 h, projecting a reduction in moisture content from 70.38% (wet basis) to 0.88% (wet basis), with a drying rate of 2.19 * 10^{-6} min^{-1}. Ansari et al. (2018) reported that freeze drying/lyophilisation of microalgal biomass requires 0.9 kW electricity for 24 h, resulting in 78.5 MJ kg^{-1} of energy consumption. Biz et al. (2019) through spray drying of *C. pyrenoidosa* reported that moisture diffusion followed Fick's second law and had the effective diffusivity of 1.4 * 10^{-13} m^2 s^{-1}, which was less than that obtained during microwave assisted drying. Also, spray dryers are reported not to be efficient in terms of energy consumption. Bennion et al. (2015) reported an energy consumption of 19.9 MJ kg^{-1} and 7.76 MJ kg^{-1} for freeze drying and rotary vacuum drying respectively, while Xu

et al. (2011) reported 80.9 MJ kg^{-1} for spray drying process. All the above-mentioned values were reportedly higher than that of the energy consumed during microwave assisted drying as shown by Viswanathan et al. (2011). Similar to the specific energy consumption mentioned in the present study, Al Rey et al. (2016) projected 0.69 MJ kg^{-1} to 1.03 MJ kg^{-1} for microwave power variation from 100 W to 600 W for a mixed microalgal consortium, which was relatively lower than that of the energy required during spray and freeze drying process.

3.4. Effect of drying on the lipid content of microalgae

The process of drying influences the microalgal cell structure, thereby the total lipid yield. It was observed that with an increase in the drying temperature from 40 °C to 60 °C, the lipid yield was found to slightly increase from 21.61 ± 0.59% (w/w) to 24.78 ± 0.20% (w/w). With a further increase in drying temperature, the lipid yield was found to decline to 23. 11 ± 0.71% w/w and the minimum lipid content of 19.14 ± 0.50% (w/w) was obtained at 100 °C. An increase in temperature often results in thermal degradation and cracking, thereby having detrimental effects on the biochemical constituents of microalgal biomass. Similar to the present study, Viswanathan et al. (2011) observed a slight decline in lipid content of a green microalgal consortium consisting of *Scenedesmus sp*., *Chlamydomonas* sp., and *Chlorella sp*., at 90 °C. The study by Hosseinizand et al. (2018) reported lipids in *Chlorella vulgaris* biomass to be preserved best at drying with a temperature of 60 °C and 80 °C, which was found to decline with further increase in temperature.

In case of microwave irradiation exposure, the maximum yield of 28.35 ± 0.35% (w/w) was obtained at 540 W which was slightly higher than the lipid content (23.78 ± 0.58% w/w) at 360 W. On increase in microwave irradiation intensity to 720 W, the lipid yield of 24.4 ± 0.20% w/w was obtained and with further increase in 900 W, there was a decline in the lipid content to 15.69 ± 0.11% (w/w). A decline in lipid content with higher intensity of microwave irradiation can be attributed to the degradation of the biochemical constituents and charring of algal biomass due to intensive thermal energy. Also, a 14.4% increase in lipid yields was observed through microwave drying at 540 W compared to the drying in a conventional hot air oven at 60 °C. Thus, it could be concluded that drying in the microwave until an optimum power setting cause uniform volumetric heating, and faster release of water molecules, thereby maintaining its biochemical constituents intact. However, during oven drying the time lag involved in the diffusion of the water molecule inside the cell to outside often causes a collapse of the cellular structure, thus altering the biochemical content (Hosseinizand et al. 2018). Singh et al. (2020) reported that a variation in drying process can affect the amount of lipid extracted from the microalgal biomass. Shekhrabi et al. (2021) reported that lipid content is significantly influenced by the drying method reporting 13.77% and 11.68% lipids from *Isochrysis galbana* subjected to freeze drying and sun drying respectively. Even though freeze drying can preserve the biochemical and lipid content of microalgae, higher energy requirements makes the process unfeasible. Thus, microwave based drying with no cell shrinkage could definitely act as a sustainable cost efficient method to obtain desirable yield from microalgae.

3.5. Influence of drying technique on the functional and biochemical composition of microalgae

Often the drying and the heat transfer techniques influence the biochemical content of microalgal biomass. To evaluate the effect of convective air drying and microwave irradiation on the quality of the algal samples to be processed into biofuel and other value-added bio-based products, the FTIR spectra of the oven and microwave dried microalgal samples were analysed (see supplementary material). The peaks were observed at 1017 cm^{-1} corresponding to the presence of C-O-C functional groups in polysaccharides. Further, the peaks at 2923 cm^{-1} were also evident relating to the $-CH_2$ stretching in lipids. In general, for detecting the basic biochemical composition of microalgal biomass, the FTIR spectral regions of interest are between the wavenumbers of 2809 cm^{-1} to 3639 cm^{-1} corresponding to the lipid fraction, 1583 cm^{-1} to 3012 cm^{-1} specific to amides of proteins, and between 700 cm^{-1} to 1100 cm^{-1} representing the carbohydrate content (Al Rey et al. (2016)). Higher the area covered by the peaks and lower the transmittance value, the better is the quantity and quality of biochemical constituents obtained. Thus, in the present study, the FTIR spectral peaks for microwave dried biomass showed lower transmittance of the characteristics peaks, showing the better quality of biochemical constituents compared to that of the oven-dried algal biomass. Also, the larger area covered by the spectral peaks for lipids, carbohydrates, and proteins in microwave dried samples compared to that of the oven-dried samples, showed the biochemical content to be possibly more in terms of quantity in former compared to the latter. To the best of the authors' knowledge, there have been no studies on the comparative yield of biochemical constituents obtained via convective oven-based and microwave-based drying of microalgal biomass. The study by Mina et al. (2017), compared the FTIR spectrum of the microwave dried and the freeze dried sample of *Nannochloropsis sp*., reporting the presence of similar functional groups in both cases. Further, it could also be affirmed that microwave based drying could preserve the biochemical constituents of microalgal biomass as good as freeze-drying, which is also time-consuming and energy intensive. It can be well concluded that the algal biomass subjected to microwave drying will have a well-preserved and desirable biochemical content to be processed into biofuel or other bio-based products in an algal biorefinery.

4. Conclusion

The study compared the efficiency of thin layer drying of microalgae through OD and MWD showing the later process to be much faster and efficient with high effective diffusivity of 0.47×10^{-9}–1.63×10^{-9} m^2 s^{-1}. Kinetic modelling showed best fit with Newton and Henderson-Pabis model as MWD followed volumetric heating due to internal sub-surface moisture diffusion. 14.4% higher lipid content with appreciable functional groups corresponding to carbohydrates and proteins was observed in MWD due to better heating and low cell shrinkage. Thus, with less enviro-economic impacts, MWD could be easily scaled up facilitating commercialization of microalgal technologies.

CRediT authorship contribution statement

Bunushree Behera: Conceptualization, Writing – original draft. **Paramasivan Balasubramanian:** Conceptualization, Investigation, Funding acquisition, Writing – review & editing.

Declaration of Competing 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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Appendix A. Supplementary data

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