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Influence of temperature on vacuum drying characteristics, functional properties and micro structure of Aloe vera (*Aloe barbadensis* Miller) gel

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Abstract

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Aloe vera possesses therapeutic, antioxidant and some other functional properties. These properties may be affected by processing operations. The present study investigated the influence of operating temperature on vacuum drying characteristics, functional properties and the inner solid structure of the fresh aloe vera gel. The gel was dried at a constant pressure of 720 mm Hg in the drying chamber at varying temperature of 30–60°C. The experimental data of moisture ratio of Aloe vera were used to fit different models and the effective moisture diffusion coefficients and activation energy were also calculated. The Page model was found to be the best fit to experimental data. The functional properties like water retention capacity, fat absorption capacity, and swelling of the dried product were studied and found to be decreased with increasing operating temperature. The damage to the inner solid structure was more pronounced at higher temperatures because of faster mass transfer through the pores of the solid. The best quality product was obtained when the temperature was maintained at 30°C.

Keywords: therapeutic; moisture ratio; activation energy

Aloe vera, the perennial tropical plant of African origin, belongs to the family of Liliaceae. There are more than 360 species known worldwide with different varieties i.e. Curacao Aloe (*Aloe barbadensis* or Aloe vera), Cape Aloe (*Aloe ferox*), and Socotra Aloe (*Aloe perryi*). Aloe vera (*Aloe barbadensis* Miller), recently named as Aloaceae (Eshun, He 2004), has been extensively used by human beings for its unique natural properties. It has thick, fleshy leaves covered with fleshy spikes and bears hanging, tubular, yellow flowers upon maturity (Martinez et al. 1996). It has low water requirements owing to its xerophytic adaptations. The thick mucilaginous transparent gel contained in the parenchymatic tissue of the aloe vera leaves is known as aloe vera gel. The gel is protected from evaporation by a film of a substance known as aloe, comprising mainly of resins and aloins (Vega et al. 2005). It contains over

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98—99% water and more than 60% of the dry matter is made of polysaccharides (McAnalley 1993; Femenia et al. 1999). The types of polysaccharides that are present in the gel are namely Acemannans (reserve polysaccharides rich in mannose). They are found inside the cell protoplasts, with a wide variety of polysaccharides that form the net of the cell wall, which consists of pectic substances, cellulose and also hemicellulose (Femenia et al. 1999, 2003; Chang et al. 2006).

Aloe vera is now grown as an industrial crop, for its immense nutritional and medicinal value. Aloe vera gel is found to possess immunomodulatory, anti-inflammatory, anti-allergic, anti-bacterial, and wound- and burn- healing properties apart from being an excellent hydrating and moisturizing agent for the skin (Chung, Choi 2006; Lee 2006; Ro 2006). The gel is a good source of vitamins C and E, protein, carbohydrate, fibre and contains low fat. It is now widely used in various food products, i.e. juices, yoghurts, marmalades, biscuits and cosmetics like lotion and shampoo, etc. It is also increasingly used as a natural coating agent owing to anti-oxidative properties. However, the major problem with aloe vera processing lies in its low shelf life and high handling cost because of its higher moisture content. The main issue confronting aloe vera processing industries has been its low shelf life and high handling costs because of its considerably higher moisture content. In such industries, drying has become the most favourable and commonly used technique to get rid of the problems associated with high moisture content. The purpose of drying operation is to allow longer storage period, minimize packaging requirements, and reduce shipping weight (Okos et al. 1992). Furthermore, products with low moisture can be stored for long periods of time at room temperature (Jayaraman, Das Gupta 1995). Thus, drying of aloe vera gel to a safe moisture level becomes very important to preserve its magnificent and diversified application.

However, improper heating during dehydration procedures may cause irreversible modifications in the polysaccharides, affecting their original structure, which may produce certain changes in the proposed physiological and pharmacological properties of the active ingredients (Eshun, He 2004). Vacuum drying of food materials was used for many years in order to introduce less temperature-induced changes. In comparison with a conventional atmospheric drying, vacuum drying allows for the moisture removal at low pressure. The high pressure gradient produced in vacuum drying allows a faster mass transfer in the drying chamber causing the material to dry rapidly with time. Vacuum expands air and water vapour present in the food material and creates a frothy or puffed structure, providing a large area-to-volume ratio for enhanced heat and mass transfer (Jaya, Das 2003). Consequently, with vacuum drying it is possible to have higher drying rate, lower drying temperature and an oxygen-deficient processing environment (Wu et al. 2007).

The aim of this work was to study the vacuum drying characteristics of fresh aloe vera gel as well as the influence of temperature on the functional properties and inner solid structure of the gel.

MATERIAL AND METHODS

Sample preparation. Fresh aloe vera leaves were harvested by separating the leaves from the stem of 2–3 years old Aloe vera (*Aloe barbadensis* Miller) plant grown in the aloe vera field of the department with a stainless steel knife. The leaves were selected maintaining the homogeneity in their maturity, size and colour. The length of the leaves varied from 0.2–0.3 m. Aloin, a yellow coloured liquid layer between the outer rind and the inner gel of the leaf was drained out by cutting the base of the leaves and keeping them in a slanted position for an hour. Rinds of the freshly harvested aloe vera leaves were then manually removed using the knife and the inner gel was cut into cubical pieces of 10 mm side. The sample pieces of the aloe vera gel were immediately put into the refrigerator at 4°C for the drying within 24 hours.

Drying experiments. Drying was carried out at four temperature levels of 30, 40, 50 and 60°C while the drying chamber was maintained at 5 kPa. Aloe vera gel cubes were dried in the lot of 70 g each. The cubes were spread in a single layer on a tray and placed inside the vacuum dryer (Rivotek TM Vacuum Oven (Round) – STD Model; RG Private Ltd., Mumbai, India). The dryer had an inbuilt Ohmic heating mechanism, a thermostat to maintain the temperature at the pre-set value and a Bourdon gauge for displaying an instantaneous vacuum level (mm of Hg) inside the chamber (Fig. 1). The surface was heated mainly by conduction and radiation as convection is very poor in vacuumised region. The

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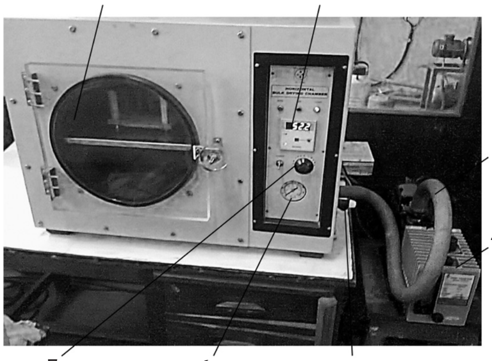

Fig. 1. Set-up for vacuum drying of aloe vera gel
1 – drying chanber; 2 – thermostat; 3 – connecting tube; 4 – vacuum pump; 5 – vacuum release vent; 6 – bourdon gauge; 7 – heating control knob

door of the chamber was properly closed after applying vacuum sealing gel on the contacting surface to prevent any leakage into the chamber as the drying proceeded. Drying started when the vacuum pump was switched on to bring the vacuum level from 0 to 720 mm Hg (95.99 kPa). At the intervals of 1 h each, the vacuum pump was switched off and the vacuum inside the drying chamber was relieved by operating the vacuum release vents. Thereafter, the samples were taken out, cooled in desiccators and weighed. Drying was stopped when two consecutive readings matched, indicating the equilibrium conditions.

Mathematical modelling of drying curves. Modelling of the drying data was done using various models which relate moisture ratio with temperature, which in turn relates the gradient of sample moisture in real time with initial and equilibrium levels.

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

$$MR = \frac{X_t - X_e}{X_0 - X_e} \tag{2}$$

where:
X_t – moisture content at any time (g water/g d.m.)
m_t – mass of the sample at any time (g)
m_g – absolute dried mass of sample (g)
MR – moisture ratio (–)
x_0 – initial moisture content (g water/g d.m.)
x_e – equilibrium moisture content (g water/g d.m.)

The drying rate (DR) aloe vera was calculated using Eq. (3):

$$DR = \frac{X_{t+dt} - X_t}{dt} \tag{3}$$

where:
DR – drying rate (g water/g d.m.)
X_{t+dt} – moisture content at $t + dt$ (g water/g d.m.)
t – time (s)
dt – time incrementW

The drying data were fitted to six thin layer drying models detailed in Table 1 using nonlinear regression analysis by using the ORIGIN 8.5 software (OriginLab Corporation, Northampton, USA). The coefficient of determination (R^2) was the primary criteria for selecting the best equation to describe the drying curve in addition to root mean square analysis (*RMSE*). The higher value of R^2 and lower value of *RMSE* was considered for justifying the better goodness of fit.

$$RMSE = \left(\frac{1}{n}\sum_{i=1}^{n}\left(MR_{i,\mathrm{cal}} - MR_{i,\mathrm{exp}}\right)^2\right)^{\frac{1}{2}} \tag{4}$$

where:
$MR_{i,cal}$ – ith calculated moisture ratio
$MR_{i,\mathrm{exp}}$ – ith expected moisture ratio
n – number of samples
i – positive integer (i = 1,2,3, ...)

Determination of the effective moisture diffusivity. Fick's second law of diffusion equation was used to fit the experimental drying data for the determination of the effective moisture diffusivity coefficient.

$$\frac{\partial X}{\partial t} = D_{\mathrm{eff}}\frac{\partial^2 X}{\partial x^2} \tag{5}$$

where:
X – moisture content (g water/g dry matter)
t – time (s)
x – thickness of the material (m)

The solution of diffusion equation Eq. (7) for slab geometry is solved by Crank (1975), and supposed uniform initial moisture distribution, negligible external resistance, constant diffusivity and uniformity in the physical dimension.

$$MR = \frac{8}{\pi^2}\sum_{n=1}^{x}\frac{1}{(2n-1)^2}\exp\left(\frac{-(2n-1)^2\pi^2 D_{\mathrm{eff}}t}{4H^2}\right) \tag{6}$$

where:
D_{eff} – effective moisture diffusivity (m^2/s)
t – drying time (s)
H – half thickness of the sample (m)
n – positive integer

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Table 1. Thin layer models applied to the aloe vera drying

Sr. No.	Model name	Model	References
1	Newton	$MR = \exp(-kt)$	Westerman et al. (1973)
2	Henderson and Pabis	$MR = a\exp(-kt)$	Bhattacharya et al. (2015)
3	Logarithmic	$MR = a\exp(-kt) + c$	Bhattacharya et al. (2015)
4	Page	$MR = \exp(-kt^n)$	Bhattacharya et al. (2015)
5	Modified page	$MR = a\exp(-(kt^n))$	Yaldiz et al. (2001)
6	Wang and Singh	$MR = 1 - at+bt^2$	Ozdemir, Devres (1999)

Sr. No. – serial number; MR – Moisture ratio; a, b, c – equation constants; k –drying rate constant, t – time

Only the first term of equation can be used for long drying time (Lopez et al. 2000)

$$MR = \frac{8}{\pi^2}\exp\left(\frac{-\pi^2 D_{\text{eff}} t}{4H^2}\right) \tag{7}$$

The slope of the plot ln(MR) vs. time is given:

$$\text{Slope} = \frac{-\pi^2 D_{\text{eff}} t}{4H^2} \tag{8}$$

Computation of the activation energy. The effective moisture diffusivity (D_{ef}) and temperature relation is generally described by the Arrhenius equation:

$$D_{\text{eff}} = D_0\exp\left(\frac{-E_a}{R(T + 273.15)}\right) \tag{9}$$

where:

D_0 – pre-exponential factor (m²/s)

E_a – activation energy (kJ/mole)

R – universal gas constant (kJ/mol·K)

T – temperature (°C)

Functional properties. Functional properties measured of each dried sample included hydration properties such as water retention capacity (WRC), swelling (SW) and fat absorption capacity (FAC).

WRC was expressed as the amount of water retained by the dry matter (Thibault et al. 1992). Each of the dried gel samples weighing 0.25 g was suspended for 24 h in 5ml of phosphate buffer solution and was then centrifuged at 15,000 rpm for 15 minutes. Residual solids in the supernatant were recovered by filtration over a 1.2 µm glass microfiber filter paper (GF/C) and recombined with the pellet. The pellet was weighed as P_1 and dried at the 102°C for 24 h in an air oven. After cooling, the dry weight was determined as P_2. Finally, the WRC was calculated:

$$WRC = \frac{P_1 - P_2}{P_2 - k} \tag{10}$$

where:

k = $a(P_1 - P_2)/\rho$, with $a = 2.8 \times 10^{-2}$ g salt (phosphate)/ml

ρ – water density at standard temperature and pressure (STP) (g/ml)

a – constant

SW was measured as bed volume after equilibration in excess solvent (Kuniak, Marcessault 1972). Samples in 0.5 g each were weighed into a graduated conical tube with an excess of buffer. The suspension was stirred and after equilibration in 16 h, the volume was recorded and expressed as ml/mg d.m.

The FAC was measured as the amount of vegetable oil retained by the dry matter (Caprez et al. 1986). Each sample (of 0.25 g each) was mixed with 5 ml of sunflower oil and left overnight at room temperature. The samples were then centrifuged at 15,000 rpm for 10 minutes. The excess supernatant was then decanted and FAC for each sample was determined and expressed in g oil/g dry matter.

Scanning Electron Microscopy. Scanning electron microscopy of each of the vacuum dried aloe vera gel samples was carried out with a scanning electron microscope (JEOL JSM-5800 scanning microscope; JEOL Ltd., Tokyo, Japan). The dried samples were gold plated before the scanning process because of their non-conducting nature of electrons. The obtained micrographs of vacuum dried aloe vera gel through scanning electron microscopy (SEM) were analysed and studied to understand the structural changes like structural collapses, stretches over the surfaces.

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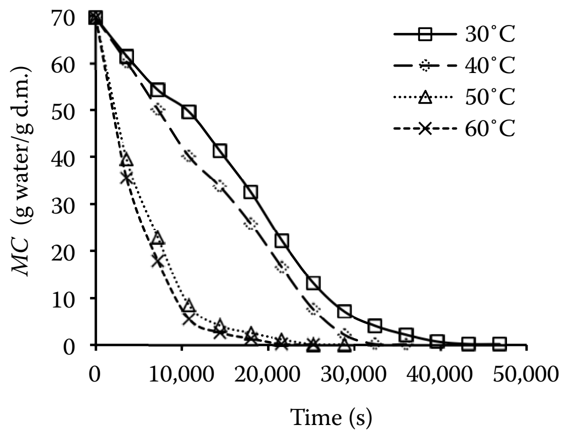


Fig. 2. Drying curves; moisture content (MC) versues time at different temperatures

RESULTS AND DISCUSSION

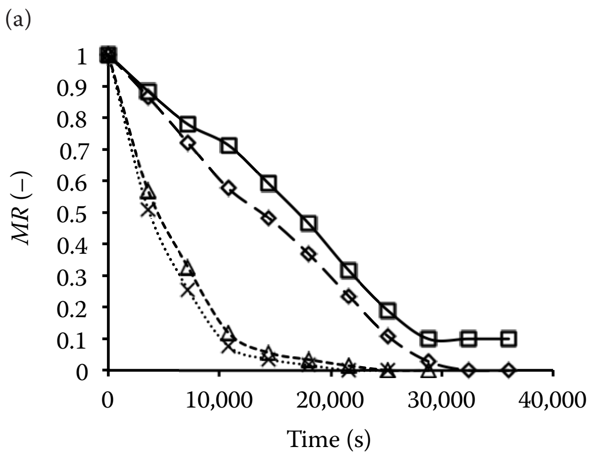
Drying characteristics

The fresh aloe vera gel was dried from 69.592 to 0.207, 0.184, 0.173, and 0.162 g water/g d.m. at 30, 40, 50, and 60°C under vacuum and the drying times were 13, 10, 8, and 7 h, respectively. Fig. 2 shows that moisture content sharply decreased at 50 and 60°C in comparison to moisture decreased at 30 and 40°C which were due to faster moisture diffusion from the centre of aloe vera gel to the surface and consequently drying time decreased. Moisture ratio was estimated by using Eq. (2) and its changes with time are shown in Fig. 3a. Drying rate estimated based on equation Eq. (3) and its change with temperature is shown in Fig. 3b. A peculiar influence of drying temperature on drying rate could be observed in these curves (Fig. 3b) and it shows that drying rate decreases continuously with the increase in time. The drying rate was initially very high at 50 and 60°C but after some time it crossed the drying rate curve at 30 and 40°C and become almost constant. The major drying operation occurs in the falling rate period as the most of the agricultural products are dried in the falling rate drying period, and in this condition the movement and diffusion of moisture in interior of products controls the entire drying process (Sahay, Singh 2001). These results are in good agreement with the observations of various other agricultural products like peach (Zhu, Shen 2014).

The moisture removal from the aloe vera gel at higher temperature influenced faster than the lower temperature because temperature influences the migration of moisture from the interior to the surface. At lower temperatures, moisture migration to the surface of the cube-shaped aloe vera gel and evaporation rate from the surface to vacuum chamber decreases with the decrease of moisture in the aloe vera sample and thus increased drying rate (Fig. 3a). This increase is due to the increased heat transfer potentials between the local atmosphere (vacuum chamber) and aloe vera cubes which supports the evaporation of moisture from the surface.

Fitting of drying curves

The drying data obtained from the experiments were fitted to six thin layers drying models mentioned in Table 1. Non-linear regression analysis was used to estimate the parameters of all the models. The model expression and statistical results at different temperatures are summarized in Table 2. The best model describing the thin layer drying characteristics of Aloe vera gel was set on



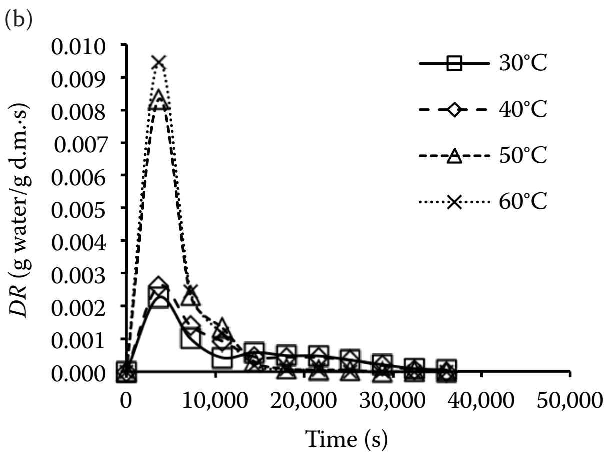


Fig. 3. Drying curves (a) moisture ratio (MR) versus time and (b) drying rate (DR) versus time at different temperatures

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Table 2. The fitting models and statistical results at different drying temperatures

Type of model	T (°C)	Models	R^2	$RMSE$
Newton	30	$MR = \exp(-0.0000550518t)$	0.92274	0.10139
	40	$MR = \exp(-0.0000647745t)$	0.93806	0.089331
	50	$MR = \exp(-0.00017069t)$	0.99495	0.024495
	60	$MR = \exp(-0.000200047t)$	0.99649	0.021078
Henderson and Pabis	30	$MR = 1.11342\exp(0.0000605781t)$	0.93673	0.091752
	40	$MR = 1.08606\exp(-0.0000699746t)$	0.94766	0.082116
	50	$MR = 1.01344\exp(-0.000172593t)$	0.99516	0.023953
	60	$MR = 1.00917\exp(-0.000201523t)$	0.99659	0.020736
Page	30	$MR = \exp(-0.000000170182t^{1.58004})$	0.98737	0.040988
	40	$MR = \exp(-0.000000890064t^{1.43828})$	0.98233	0.047703
	50	$MR = \exp(-0.0000415287t^{1.15822})$	0.99811	0.014958
	60	$MR = \exp(-0.0000565547t^{1.1436})$	0.99874	0.012593
Modified Page	30	$MR = 0.99634\exp(0.000001147481t^{1.59401})$	0.98562	0.043745
	40	$MR = 1.00043\exp(-0.000000886317t^{1.43876})$	0.98015	0.050572
	50	$MR = 0.99903\exp(-0.0000407474t^{1.16026})$	0.99749	0.017224
	60	$MR = 0.99946\ \exp(-0.000055951t^{1.14476})$	0.99824	0.014900
Logarithmic	30	$MR = 1.46691\exp(-0.0000313501t)-0.41322$	0.97261	0.060378
	40	$MR = 1.66455\exp(-0.0000290848t)-0.64248$	0.98955	0.036691
	50	$MR = 1.03145\exp(-0.000161519t)-0.02306$	0.99551	0.023094
	60	$MR =1.02681\exp(-0.000189391t)-0.02153$	0.99701	0.019391
Wang and Singh	30	$MR = 1 - 0.0000386701t + 0.000000000346598t^2$	0.98562	0.041883
	40	$MR = 1 - 0.0000440814t + 0.00000000041576t^2$	0.98015	0.047679
	50	$MR = 1 - 0.000101361t + 0.00000000241151t^2$	0.99749	0.015946
	60	$MR = 1 - 0.000116514t + 0.00000000316977t^2$	0.99824	0.013601

MR – moisture ratio; t – time; T – temperature; $RMSE$ – room mean square error; R^2 – coefficient of determination

the basis of the highest R^2 values and the lowest $RMSE$ values. The associated statistical parameter estimations for different temperature showed that R^2 and $RMSE$ values were varied from 0.92274 to 0.99874 and 0.012593 to 0.0125, respectively. The highest value of R^2 was found to be 0.99874 and the lowest value of $RMSE$ was 0.012593 in case of the Page model and hence the model may be selected as a best fit model for temperature to represent the thin layer drying.

Effective moisture diffusivity

The values of the effective moisture diffusivity were determined by using Eq. (8) and are shown in Table 3. The Eq. (8) is a slope determined by plot between ln(MR) vs. time. The values of effective moisture diffusivity were varied in the range of 3.042×10^{-9} to 7.0997×10^{-10} m^2/s at 30–60°C. The sample thickness was kept uniform in every experiment. It was observed that the effective moisture diffusivities were increased with increasing drying temperature, which is due to the increase in thermal energy. The latter increased the activity of water molecules leading to the higher moisture diffusivity, but especially at higher temperatures it seemed to be constant because of the relation of moisture concentration to effective moisture diffusivity.

The values of D_{eff} obtained from this study lie between the range of 10^{-12} to 10^{-8} m^2/s for the drying of food materials and these results have been sup-

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Table 3. The effective moisture diffusion coefficient of different temperatures

T (°C)	$1/(T + 273.15)$(K^{-1})	D_{eff} (m^2/s)	lnD_{eff}
30	0.003298697	7.0997×10^{-10}	–21.066
40	0.003193358	2.0228×10^{-9}	–20.0188
50	0.003094538	3.042×10^{-9}	–19.6101
60	0.003001651	3.042×10^{-9}	–19.6101

T – temperature; K – kelvin; D_{eff} – effective moisture diffusivity

Table 4. Functional properties of vacuum dried aloe vera gel at different temperatures

Sample type	WRC (g/g d.m.)	SW (ml/mg d.m.)	FAC (g/g d.m·s)
S_1	13.72	57.41	06.37
S_2	12.93	49.24	04.92
S_3	11.35	37.81	04.28
S_4	09.41	30.92	03.66

S_1, S_2, S_3, S_4 – samples are dried at 30, 40, 50 and 60°C; WRC – water retention capacity, SW– swelling; FAC – fat absorption capacity

ported by other medicinal crops like garlic (Pardeshi et al. 2009).

Activation energy

A linear graph between lnD_{eff} vs. $(T + 273.15)^{-1}$ was plotted and shown in Fig. 4. The slop ($-E_a/R$) of the straight line was obtained and by using the Arrhenius relationship, the activation energy was found to be 40.63 KJ/mol. The value is somewhat similar to the D_{eff} value of other agricultural product like peach i.e. 42.53 kJ/mol (Zhu, Shen 2014).

Influence of temperature on functional properties of vacuum dried Aloe vera gel

Functional properties measured in this study included WRC, SW, and FAC. It was observed that all the three hydration-related functional properties of vacuum dried aloe vera gel samples reduced (Table 4) with temperature. This might be because of the faster fluid diffusion in vacuum drying, resulting in more shrinkage which, in turn, left shrunk the pore spaces to be filled with water or oil. The decreasing effect of temperature on swelling might be possible at higher temperature, as the solid got more shrunk and rigid.

Micro-structure of dried product

Changes in the inner solid structure of vacuum dried aloe vera gel samples were studied and compared through their SEMs micrographs (Fig 5). Analysis of such changes at the cellular level is important as these explain the variations in other properties of the gel like the kinetic parameters and the functional properties of the dried product. For vacuum dried products, the structural collapses, breakages, scratches and more thread formation

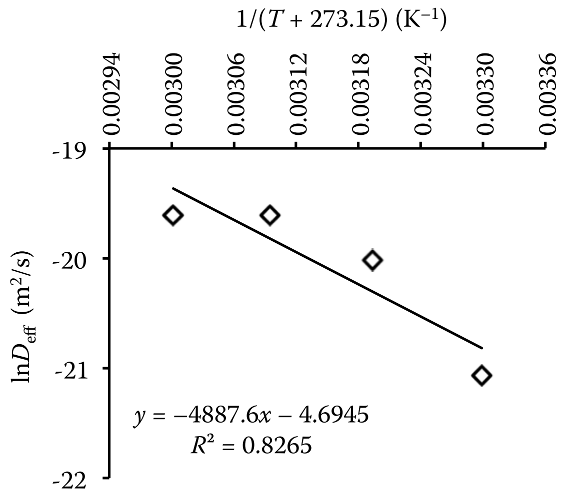

Fig. 4. Relationship of lnD_{eff} and $1/(t + 273.15)$ at vacuum level of 95.99 kPa

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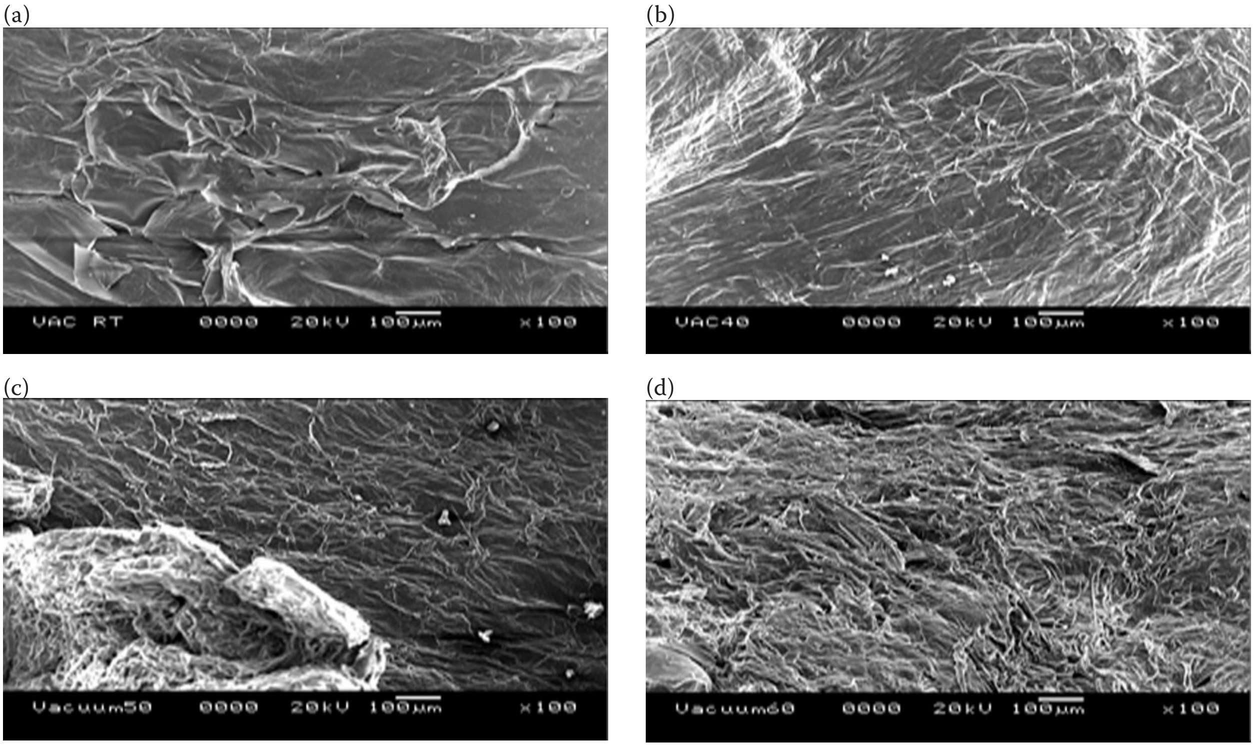

Fig. 5. SEM of vacuum dried product at (a) 30°C, (b) 40°C, (c) 50°C and (d) 60°C

at higher drying temperature were observed. However, such collapses were the least when the drying took place at 30°C. This could be attributed to the faster moisture transfer through the solid pores at a higher temperature in vacuum which also led to the shrinkage of the cell wall.

CONCLUSION

The operating temperature during the vacuum drying of Aloe vera gel at 5 kPa influenced both the drying time as well as the final moisture content of the dried product. The Page model showed a good fit with the drying data. Evaluation of the three functional properties showed decrements in them over the higher temperatures. The inner solid structure of dried product was damaged over drying. This damage was more severe at higher operating temperatures. Thus, from the study, it can be recommended that the vacuum drying of the gel be conducted at 30°C to obtain the best quality product.

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