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A modified model for alumina membranes formed by gel-casting followed by dip-coating

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Abstract

α-Al_2O_3 membrane coating (top layer) was formed by dip-coating and investigated based on full factorial design of experiments. Statistical analysis showed that the effects of two variables; dipping time and withdrawal speed of the substrate, were highly significant in determining the thickness of the top layers ($\alpha < 0.001$). A modified model was developed to interpret quantitatively the formation process of the top layers onto the porous substrate during dip-coating. In this model, the effects of all variables affecting the top layer thickness were considered simultaneously. The experimental data corresponding to α-Al_2O_3 membranes obtained from suspensions with different volume fractions of ceramic powder showed good agreement with the model. The root mean square deviation was less than 0.02.

Keywords: Dip-coating; Membranes; Al_2O_3; Gel-casting; Modelling

1. Introduction

Gel-casting process is a novel forming method for fabricating complex three-dimensional ceramic parts. Recently, this process has been extensively used in producing porous ceramic parts employed in drug delivery systems, bio-ceramics and sensors,[1,2] ceramic foams,[3] and porous support systems for membranes.[4,5]

Generally, a ceramic-composite membrane is built up of three distinct components; a porous support system, with a few millimeters thickness and pore size in the range of 0.5–10 μm, which provides mechanical strength to the membrane; an intermediate layer, with 10–100 μm thickness and pore size in the range of 0.05–0.5 μm, which acts as transition phase and prevents the top layer from penetrating into the porous structure of the support; and finally a top layer, with 1–10 μm thickness and pore size in the range of 2–50 nm, which plays the main role in membrane filtration processes, see Fig. 1.[6]

The intermediate and top layers are often deposited on the porous support system by a dip-coating procedure. When a dry porous support is dipped into a ceramic powder suspension and subsequently withdrawn from it, a wet and more or less dense cake of well-defined thickness can be formed on the substrate surface. After being dried and sintered, a ceramic-composite membrane is obtained. Careful control of the thickness of the membrane top layer is a critical stage of the dip-coating process and has a significant effect on membrane quality and properties. Therefore, full analysis and control of this so-called "dip-coating" process is necessary.

Numerous experimental and theoretical studies of the dip-coating process are to be found in the literature. Meng and coworker used a model to quantitatively describe wet membrane formation on a porous substrate by capillary filtration during the dip-coating process.[7] The model is derived on the basis of the slip-casting process, and the effect of the withdrawal speed of the substrate on the thickness of the top layers has not been considered. Krozel et al.[8] investigated the fluid mechanical aspects of halted substrate motion using gravimetric means, based on the following two basic theoretical models: (1) Jeffrey's solution for transient coating and drainage, where capillary forces were considered to be negligible;[9] (2) the Landau–Levich solution for steady state coating,[10] which was modified by White and Tallmadge.[11] In the latter model the effect of

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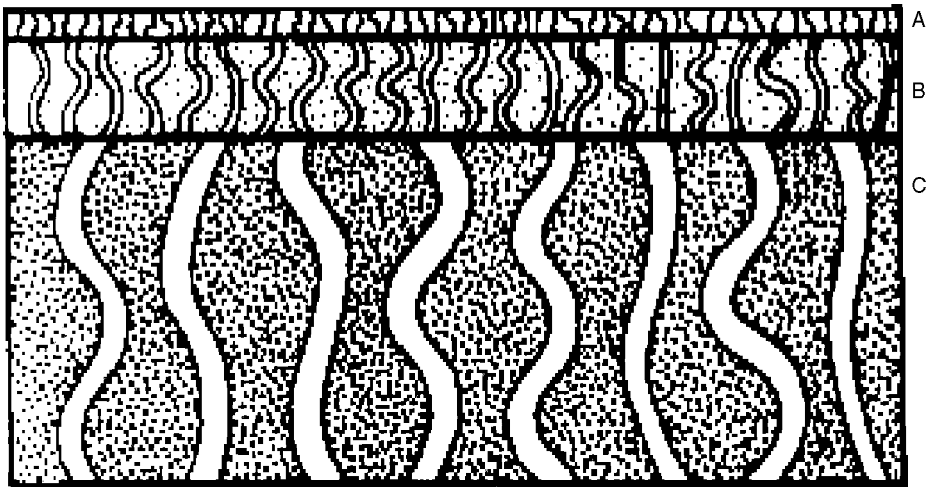


Fig. 1. Schematic representation of three distinct components of a membrane: (A) top layer, (B) intermediate layer, and (C) porous support system.

dipping time on the thickness of the top layers was also not considered. Other researchers have used these models for ceramic membrane formation by the dip-coating processes. Xia et al. used Meng model to investigate the thickness growth of yttria stabilised zirconia (YSZ) membranes on porous substrates,[12] and McDonagh et al., used the Landau–Levich solution to characterise a sol–gel derived silica film as a function of the withdrawal speed of the substrate.[13] Although these models have been proposed to describe membrane formation by dip-coating, the effects of both dipping time and withdrawal speed of substrate have not been considered simultaniously. Developing a model to interpret these coupled effects is the aim of this work.

2. Theory

2.1. Dipping time theory

Dipping time theory has been recently developed for ceramic membrane formation by the dip-coating process.[7] In this model, the growth of film thickness (h_{ave}) is interpreted as a function of substrate permeability (K_{s}) and of the permeability of the film (K_{m}) previously deposited on the substrate:

$$h_{\mathrm{ave}} = 2\left[\frac{\varepsilon_{\mathrm{s}}\sigma}{\eta\alpha R[(1/K_{\mathrm{m}}) + (\alpha/\varepsilon_{\mathrm{s}})(1/K_{\mathrm{s}})]}t\right]^{1/2} \tag{1}$$

where ε_{s} is the substrate porosity, t is the dipping time (s), η is the viscosity of suspension ($\mathrm{N\,m^{-2}\,s}$), σ is the surface tension of the liquid (solvent) in the pores of the support ($\mathrm{N\,m^{-1}}$), R is the radius of support pores (m), and α is defined as:

$$\alpha = \frac{\phi_{\mathrm{m}}}{\phi_0} - 1 \tag{2}$$

where ϕ_0 and ϕ_{m} are the volume fractions of the particles in the suspension and in the wet membrane, respectively.

Usually, the permeability of the substrate (K_{s}) is much bigger than that of the membrane (K_{m}), therefore, Eq. (1) is simplified as follows:

$$h_{\mathrm{ave}} = 2\left(\frac{\varepsilon_{\mathrm{s}}\sigma K_{\mathrm{m}}}{\eta\alpha R}t\right)^{1/2} \tag{3}$$

Considering the membrane shrinkage during the drying and sintering processes, the thickness of the sintered membrane ($h_{\mathrm{ave,s}}$) can be expressed as:

$$h_{\mathrm{ave,s}} = \beta h_{\mathrm{ave}} = 2\beta\left(\frac{\varepsilon_{\mathrm{s}}\sigma K_{\mathrm{m}}}{\eta\alpha R}t\right)^{1/2} \tag{4}$$

where β is defined as:

$$\beta = \frac{\phi_{\mathrm{m}}}{1 - \varepsilon_{\mathrm{m}}} \tag{5}$$

ε_{m} is the porosity of the sintered membrane.

The film permeability is determined by the following equation:

$$K_{\mathrm{m}} = \frac{\varepsilon_{\mathrm{m}}^3}{K_0 K_\tau S_{\mathrm{v}}^2 (1 - \varepsilon_{\mathrm{m}})^2} \tag{6}$$

where K_0 is a particle shape factor and K_τ accounts for the tortuosity of the porous medium. The product of K_0 and K_τ was generally put at about 5 for particle packings.[7] S_{v} is the surface area of the particles per unit volume of the solid ($\mathrm{m^{-1}}$).

2.2. Withdrawal speed theory

For Newtonian liquids, the principle considered in the prediction of the Landau–Levich model is the limiting film thickness (h_∞):[8]

$$h_\infty = 0.944\frac{(\eta U)^{2/3}}{\sigma^{1/6}(\rho g)^{1/2}} \tag{7}$$

where ρ is the density ($\mathrm{g\,m^{-3}}$), g is the acceleration due

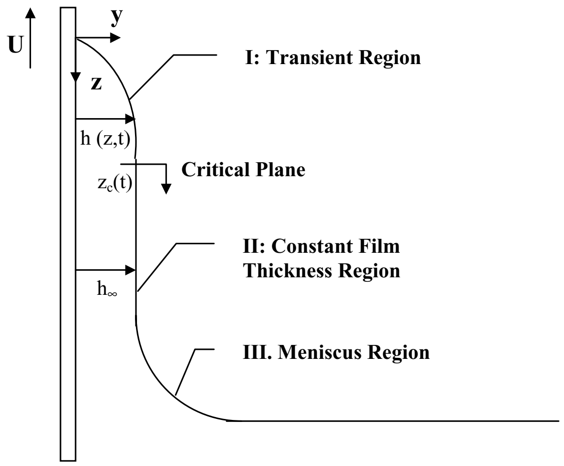


Fig. 2. An instantaneous view of a dip-coating process. Three major regions are labelled. The critical plane separates regions (I) and (II).

to gravity ($\mathrm{m\,s^{-2}}$) and U is the withdrawal speed of the substrate ($\mathrm{m\,s^{-1}}$). It is customary to introduce a scaled film thickness (T) and a capillary number (N_{ca}) as follows:

$$T = h\left(\frac{\rho g}{\eta U}\right)^{1/2} \tag{8}$$

$$N_{\mathrm{ca}} = \frac{\eta U}{\sigma} \tag{9}$$

So that the Landau–Levich expression may be written as (at $h = h_\infty$):

$$T_\infty = 0.994 N_{\mathrm{ca}}^{1/6} \tag{10}$$

This theory is valid for $N_{\mathrm{ca}} \ll 1$. The constant thickness region (II) in Fig. 2, typically constitutes the majority of the film on the substrate, and pertains to Landau–Levich theory. White and Tallmodge patched regions (I) and (II) together to obtain the average film thickness for constant withdrawal speed:[8]

$$T_{\mathrm{ave}} = 0.994 N_{\mathrm{ca}}^{1/6}\left[1 - 0.297\left(\frac{\eta U}{\sigma}\right)^{1/3}\right] \tag{11}$$

or

$$h_{\mathrm{ave}} = 0.994\frac{(\eta U)^{2/3}}{\sigma^{1/6}(\rho g)^{1/2}}\left[1 - 0.297\left(\frac{\eta U}{\sigma}\right)^{1/3}\right] \tag{12}$$

It is believed that a modified model could be used to induce better quality in products, better process control, and consistency. The present study is aimed at achieving a quantitative understanding of the simultaneous effects of the previously mentioned variables, i.e. dipping time and withdrawal speed of the substrate, on the top layer thickness, also at developing a modified model to describe wet membrane formation on the substrate during the dip-coating process.

3. Experimental procedure

3.1. Materials

The characteristics of materials used in this work are given in Table 1 and Fig. 3.

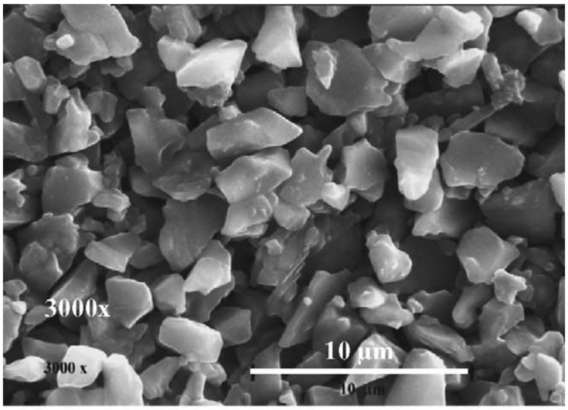
3.2. Preparation of supports

Supports were prepared by the gel-casting process. The flow sheet is illustrated in Fig. 4. Appropriate amounts of monomers (5 wt.%) were dissolved in deionised water to make the pre-mix solutions. Then, ceramic suspensions were prepared by dispersing the required volume fraction of support alumina powder (40 vol.%) to a pre-mix solution

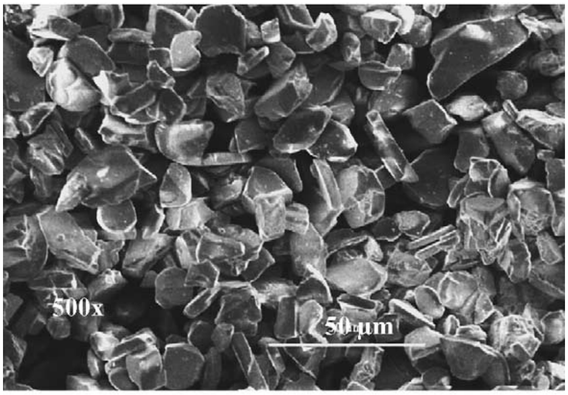
Table 1
Characteristics of materials

Materials	Function	Molecular formula	Characteristics	Supplier
α-Alumina	Ceramic phase (coating)	α-Al_2O_3	See Fig. 3a; average particle size: 4 μm	Good Fellow[a]
α-Alumina	Ceramic phase (supports)	α-Al_2O_3	See Fig. 3b; average particle size: 10 μm	Good Fellow[a]
Polyethylene glycol (PEG)	Polymeric binder	$HO(C_2H_4O)_nH$	Average molecular weight: 35,000; solid white powder	Merck[b]
Acrylamide (AM)	Monofunctional monomer	$C_2H_3CONH_2$	Molecular weight: 71.08; solid white powder	Merck[b]
N,*N*′-Methylene bis-acrylamide (MBAM)	Difunctional monomer (crosslinker)	$(C_2H_3CONH_2)_2CH_2$	Molecular weight: 154.2; solid white powder	Sigma[c]
Ammonium persulfate	Initiator	$(NH_4)\ _2S_2O_8$	Molecular weight: 228.2; solid white powder	Merck[b]
N,*N*,*N*′,*N*′-Tetramethyl ethylene diamide (TEMED)	Accelerator	$C_6H_{16}N_2$	Molecular weight: 116.2; liquid, yellow	Merck[b]
Ammonium poly(methacrylate) (APMA)	Dispersant	$(C_4H_5O_2{}^-NH_4{}^+)_n$	Average molecular weight: 15,000; aqueous solution: 40 wt.%	R.T. Vanderbilt[d]

[a] Cambridge Science Park, Cambridge, UK.
[b] E. Merck, D6100 Darmstadt, Germany.
[c] Sigma Chemical Co., St. Louis, MO, USA.
[d] R.T. Vanderbilt Co., Norwalk, CT, USA.



(a)



(b)

Fig. 3. Powder used in (a) ceramic phase of coating and (b) ceramic phase of supports.

containing 0.3 ml of APMA (ammonium poly(methacrylate)) per 100 g of Al_2O_3.[14] Suspensions were mechanically stirred for at least 2 h and ultrasonicated for 15 min to prevent air trapping. As the gelled parts were demoulded and dried (by a liquid desiccant drying method[15]), they were machined to the desired net-shape, and then sintered at 1400 °C for 5 h.

3.3. Dip-coating of supports

An appropriate amount of polymeric binder, polyethlene glycol (5 wt.% based on dried alumina powder) was dissolved in deionised water, then the required volume fraction of alumina powder was added to the polymer solution and dispersed by ultrasonic mixing and intensive stirring. During the dip-coating process, any sedimentation should be avoided.

A computer-controlled dip-coating apparatus was used for the dip-coating of the porous support systems. They were dipped into the prepared suspensions and withdrawn at speeds ranging from 10 to 40 mm min^{-1} after a dipping time ranging from 8 to 34 s. The coated layers were dried at ambient temperature and then sintered at 1300 °C for 1 h.

3.4. Characterisation

3.4.1. Supports

The bulk density, the open porosity, the closed porosity, and the total porosity of the porous support systems were determined using the Archimedes method with a theoretical density of 3.9 g cm^{-3} for α-alumina. The microstructures of the supports were observed by SEM (a Philips XLC scanning electron microscope). The average pore size and pore size distribution of the sintered porous samples were determined by the "bubble point" and "gas (nitrogen) absorption" methods.[4,16–18]

3.4.2. Coated layers

As mentioned, the Archimedes method was used to determine the porosity of the sintered membranes. The thickness of the sintered coated layers was directly determined based on mass balance as follows:

$$h_{\mathrm{ave,s}} = \frac{m_2 - m_1}{\rho_{\mathrm{th}} A(1-\varepsilon_{\mathrm{m}})} \tag{13}$$

where m_1 and m_2 are the weight of the substrate before and after dip-coating and sintering (g), respectively. ρ_{th} is the theoretical density of ceramic (solid) phase of the membrane (g m^{-3}). A is the planar area of the membrane (m^2), approximated to that of the outer surface of tube. SEM was used to investigate the microstructure of the coated layers.

3.5. Statistical design of the experiments

Experimental design is generally applied to determine the dependence of a target variable such as the membrane thickness on the set of the (processing) variables.

To qualify the effects of all of the variables on the membrane thickness, a full four level factorial design of experiment was adopted to predetermine the number of experiments needed to be carried out. The variables are the dipping time and the withdrawal speed of substrate. The number of experiments (N) required for quantifying the effects of all variables is then given by the following equation:

$$N = (\text{levels})^{\text{variables}} = 4^2 = 16 \tag{14}$$

4. Results and discussion

4.1. Quantitative calculation of the membrane thickness

Table 2 shows the porosity, pore size, and pore size distribution of the porous support systems.[4] The viscosity of the suspensions with 10 vol.% of alumina at 25 °C was measured $\eta = 17$ mPa s. It was assumed that the surface tension of the solution was not affected by the change of solute concentration and equaled that of the solvent surface tension,[19] $\sigma = 74 \times 10^{-3}$ N m^{-1}, and the volume fraction of particles in the wet membrane was considered to be 0.5–0.6 (typically, $\phi_{\mathrm{m}} = 0.55$). The permeability of the membrane (K_{m})

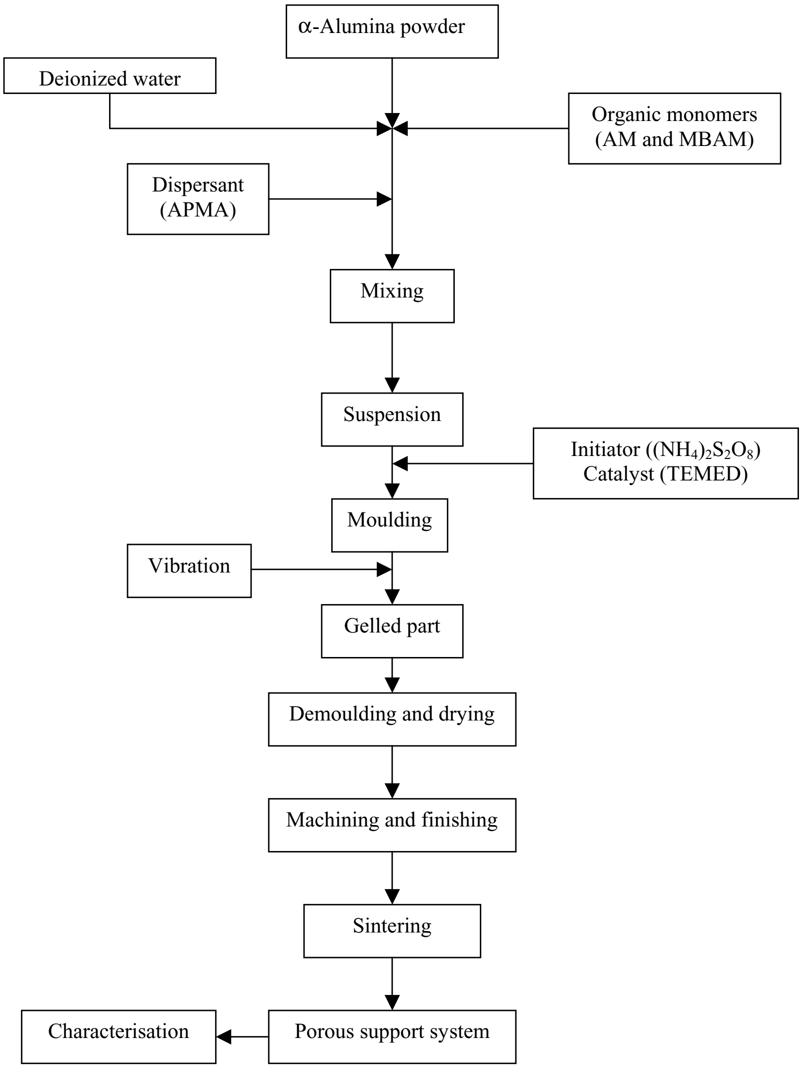


Fig. 4. The preparation process of alumina porous support systems by water-based gelcasting.

was calculated $5.65 \times 10^{-17}\,m^2$ (Eq. (6)). The results for the membrane thickness in the full factorial experimental design are given in Table 3.

4.2. Statistical analysis

A statistical analysis of the results has been carried out to identify the effect of individual variables. The statistical technique popularly known as two-way analysis of variance (ANOVA) was used to determine the significance of each variable.[20] The results are set out in Table 4. It was found that the effects of the variables, i.e. dipping time and withdrawal speed of substrate, on the membrane thickness were highly significant ($\alpha < 0.001$). It was concluded that in the appropriate model for describing the wet membrane formation on the substrate during the dip-coating process, the effects of the above variables must be considered, simultaneously.

4.3. The influence of the dipping time on the membrane thickness

The results given in Fig. 5 show that the membrane thickness increases linearly with the square root of the dipping

Table 2
Porous support system characteristics

	Open porosity volume fraction (%)	Close porosity volume fraction (%)	Pore size distribution (diameter) (μm)	Average pore size (diameter) (μm)
Porous support system	41.3	11.2	0.3–1.16	0.7
Standard deviation (variance)	8.8×10^{-5}	2.6×10^{-5}	–	–

Table 3
The results of four full factorial experimental design

Number	Withdrawal speed (mm min^{-1})	Dipping time (s)	Membrane thickness (μm)
1	11.6	8	12.1
2	11.6	14	21.2
3	11.6	24	31.8
4	11.6	34	40.9
5	21.1	8	24.0
6	21.1	14	33.3
7	21.1	24	45.5
8	21.1	34	54.5
9	30.6	8	37.9
10	30.6	14	45.5
11	30.6	24	60.6
12	30.6	34	68.2
13	40.1	8	48.5
14	40.1	14	58.1
15	40.1	24	69.7
16	40.1	34	80.3

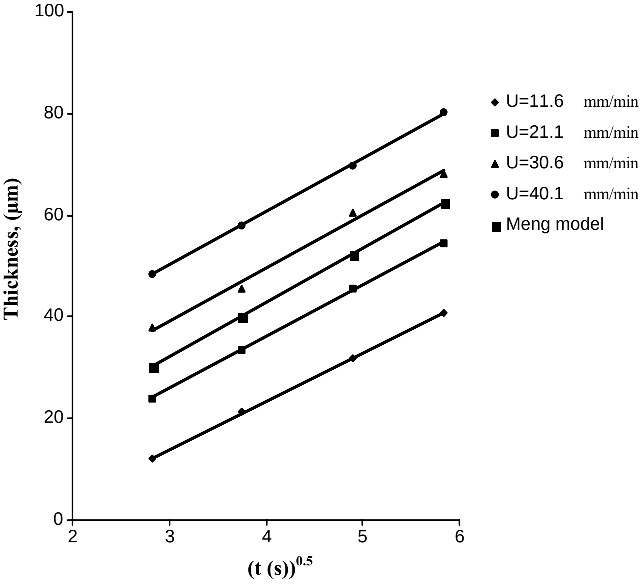


Fig. 5. The membrane thickness vs. the square root of the dipping time.

time ($t^{0.5}$). This relation is in agreement with the model derived by Meng et al. (Eq. (4)).

It should be noted that all lines in Fig. 5 are nearly parallel. This also fits the Meng model. The intercepts of the lines, however, increase as the withdrawal speed of the substrate increases (Table 5). Fig. 6 illustrates the linear correlation between the line intercepts and the withdrawal speed with an order of 2/3, on the basis of Landau–Levich theory. Thus, the final regression equation obtained may be written as follows:

$$h_{\text{ave}} = 10.7t^{1/2} + 5.1U^{2/3} - 42.0 \tag{15}$$

Table 5
Curve fitting results on the basis of Meng model

Withdrawal speed (mm min^{-1})	Solid line slope (μm s^{-1})	Solid line intercept (μm)	R^2
11.6	9.50	−14.7	0.9998
21.1	10.20	−4.8	0.9997
30.6	10.48	7.7	0.9911
40.1	10.53	18.6	0.9994
Meng model	10.69	0.0	1.0000

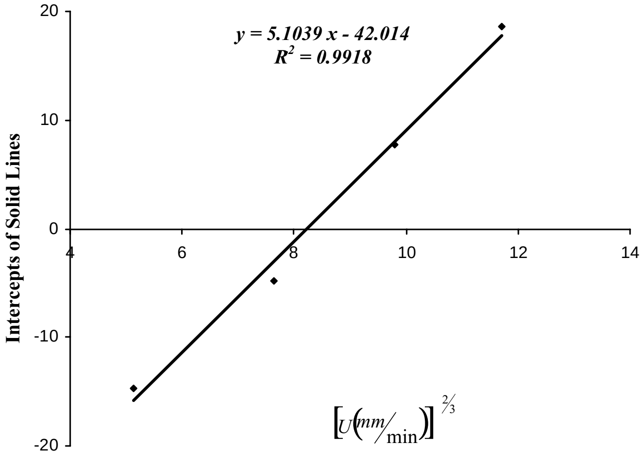


Fig. 6. The linear correlation between the line intercepts and the withdrawal speed with an order of 2/3, on the basis of Landau–Levich theory.

Table 6
Curve fitting results on the basis of White model

Dipping time (s)	Solid line slope (μm (mm min^{-1})$^{-2/3}$)	Solid line intercept (μm)	R^2
8	5.6	−17.3	0.996
14	5.6	−8.2	0.995
24	5.9	1.5	0.996
34	6.0	9.5	0.998
White model	6.0	1.3	1.000

Table 4
Two-way analysis of variance[20]

Source of variation	Sum of squares	Degrees of freedom	Mean square	Computed f	Significant α
Withdrawal speed	3204.50	3	1068.0	123.3	<0.001
Dipping time	2030.30	3	676.7	34.7	<0.001
Error	77.67	9	8.66	–	–
Total	5312.47	15	–	–	–

Table 7
The experimental and calculated values of membrane thickness

Number	Membrane thickness (μm)			
	Suspension with 10 vol.%, $\eta = 17$ mPa s		Suspension with 15 vol.%, $\eta = 19$ mPa s	
	Calculated	Experimental	Calculated	Experimental
1	10.5	12.1	18.3	21.2
2	18.6	21.2	28.2	31.8
3	28.8	31.8	40.8	44.0
4	37.1	40.9	51.0	59.1
5	23.5	24.0	32.3	38.5
6	31.6	33.3	42.2	46.7
7	41.9	45.5	54.8	59.5
8	50.1	54.5	65.0	69.5
9	34.5	37.9	44.0	49.6
10	42.6	45.5	54.0	62.0
11	52.9	60.6	66.6	69.4
12	61.1	68.2	76.8	94.3
13	44.3	48.5	54.6	61.5
14	52.4	58.1	64.5	79.5
15	62.7	69.7	77.2	87.8
16	71.0	80.3	87.3	99.7
	RMSD = 0.0126		RMSD = 0.0166	

4.4. *The influence of the withdrawal speed on the membrane thickness*

Fig. 7 presents corresponding data for membrane thickness as a function of the withdrawal speed of the substrate with an order of 2/3, at different dipping times. The linear relations, indicate a good agreement between the experimental data and the Landau–Levich model (Eq. (12)). However, the fitting lines have different intercepts for different dipping times (Table 6). The linear relationship between the line intercepts and the square root of the dipping time is shown in Fig. 8. Thus, the final regression equation obtained may be written as follows:

$$h_{\text{ave}} = 8.9t^{1/2} + 6.0U^{2/3} - 42.0 \qquad (16)$$

4.5. *Modified model proposed for forming membrane*

It was found that in the model proposed for wet membrane formation during the dip-coating process, the effects of both variables; dipping time and withdrawal speed of substrate must be considered. It is believed for this purpose, that a modified model, i.e. the superposition of both previous mentioned models, can be proposed based on the experimental results obtained from Eqs. (15) and (16).

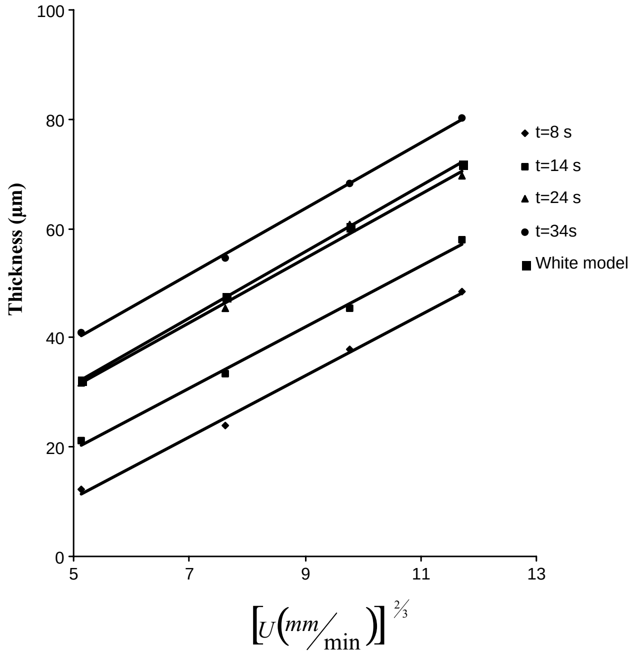


Fig. 7. The membrane thickness vs. the withdrawal speed with an order of 2/3.

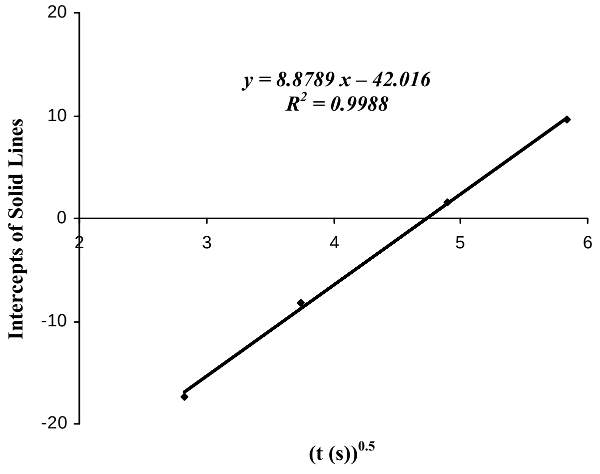


Fig. 8. The linear correlation between the line intercepts and the square root of the dipping time, on the basis of Meng theory.

$$h_{\text{ave}} = \alpha_1 \left\{ 2\beta \left(\frac{\varepsilon_s \sigma K_m}{\eta \alpha R} t \right)^{1/2} \right\} + \alpha_2 \left\{ 0.994 \frac{\beta (\eta U)^{2/3}}{\sigma^{1/6} (\rho g)^{1/2}} \times \left(1 - 0.297 \left(\frac{\eta U}{\sigma} \right)^{1/3} \right) \right\} + C \tag{17}$$

where α_1 and α_2 are the correction factors in the Meng and Landau–Levich theories, respectively. These correction factors can be calculated by dividing the fitting coefficients of Eqs. (15) and (16), respectively to the theoretical coefficients:

$$\alpha_1 = \frac{8.9}{2(\varepsilon_s \sigma K_m / \eta \alpha R)^{1/2}} = \frac{8.9}{10.7} = 0.83 \tag{18}$$

and

$$\alpha_2 = \frac{5.1}{0.994 \eta^{2/3} (\sigma^{1/6} (\rho g)^{1/2})} = \frac{5.1}{6.0} = 0.85 \tag{19}$$

Therefore, the final model obtained may be written as follows:

$$h_{\text{ave}} = 0.83 \left\{ 2\beta \left(\frac{\varepsilon_s \sigma K_m}{\eta \alpha R} t \right)^{1/2} \right\} + 0.85 \left\{ 0.994 \frac{\beta (\eta U)^{2/3}}{\sigma^{1/6} (\rho g)^{1/2}} \times \left(1 - 0.297 \left(\frac{\eta U}{\sigma} \right)^{1/3} \right) \right\} - 42 \tag{20}$$

After obtaining the modified model, a set of adequacy tests were performed to see how it fitted with the actual experimental data. Table 7 shows the experimental and calculated values of membrane thickness. The root mean square deviation (RMSD) of the experimental and calculated results has been defined as:

$$\text{RMSD} = \frac{1}{M} \left[\sum_{i=1}^{M} \left(\frac{W_i^{\text{e}} - W_i^{\text{c}}}{W_i^{\text{e}} + W_i^{\text{c}}} \right)^2 \right]^{1/2} \tag{21}$$

in which M is the number of data and W^{e} and W^{c} are the experimental and caculated results, respectively. The RMSD of membrane thickness results has been calculated 0.0126 and 0.0166 for the suspensions with 10 and 15 vol.% of alumina, respectively. The RMSD magnitudes represent a desirable agreement between the experimental and calculated results obtained from the modified model.

5. Conclusions

A statistical analysis indicated that the effects of two critical variables in the dip-coating process, i.e. dipping time and withdrawal speed of substrate, are highly significant ($\alpha < 0.001$) for the top layer thickness.

A modified model could be proposed by superposition of Meng and Landau–Levich models, based on this hypothesis. The model shows that when the suspension and substrate are fixed, the membrane thickness increases linearly with the square root of the dipping time ($t^{0.5}$) for a constant withdrawal speed of substrate. This linear relation fits with the predictions made by Meng model. Also, the membrane thickness has a linear dependency on the withdrawal speed of substrate with an order of 2/3, for a constant dipping time. The linear relations show a suitable coupling between this modified model and Landau–Levich theory.

The experimental data corresponding to the membranes formed from suspensions of 10 and 15 vol.% of α-Al_2O_3 powder, showed very good agreement with the modified model proposed.

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