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## EXPERIMENTAL INVESTIGATION AND MODELING OF MODIFICATION AGENT ON SURFACE PROPERTY OF AEROGEL PRODUCED VIA AMBIENT PRESSURE DRYING

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### Abstract

A crucial properties for hydrophobic silica aerogels such as surface free energy surface tension contact angle has been determines experimentally and theoretically in the present work, the modification was by hexamethyldisilazane (HMDZ) and trimethylchlorosilane (TMCS) with different concentration from 5% to 15% , two step acid base catalysis followed in preparation, using (TEOS) ethanol and [0.001M] hydrochloride acid with molar ratio was 1:2.8:0.19\*10<sup>-3</sup> respectively , the effect study upon analysis of FTIR spectra and the degree of hydrophobicity was estimated by contact angle measurements. The results refer that the contact angle of aerogel from 130 to 151 by modifying by HMDZ and from 122 to 153 by modifying by TMCS, Neumann's and Young's equation of state depend to determine surface free energy and surface tension of samples, while the specific surface area is measured by using the "Brunauer – Emmett–Teller (BET) method. Here, we have confirmed that the surface free energy of samples can be regulated in varied range from 4.4997 to 0.3115 and 4.1419 to 0.5112 mJ/m<sup>2</sup> by adjusting their surface by TMCS and HMDZ. From results the modification with TMCS is best from HMDZ, theoretical part give a good results to estimate the important information about hydrophobic silica aerogel.

## Keywords

Aerogel, Silylating Agent, Superhydrophobic Silica, Surface Free Energy, Contact Angle

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## 1. Introduction

Silica aerogel is a light ceramic material, it has high surface area, high porosity, low density and low thermal conductivity (Gurav, Jung, Park, Kang, & Nadargi, 2010). The quality properties of this material give it outstanding many applications such as drug delivery, thermal and electrical insulation material, chemical sensors (Gutzov et al., 2014; Hu et al., 2016; Plata et al., 2004). Besides this material used aimed at absorption of Oil-Spill cleanup and self-cleaning windows (Feng, Nguyen, Fan, & Duong, 2015; Lai, Pangilinan, & Advincula, 2018). The qualifications of aerogel depend on factors contributing to the preparation process, the most important parameters adopted in its preparation are pH factor, R-molar ratio, catalysis and precursors (Milea, Bogatu, & Duță, 2011; A. V. Rao, Nilsen, & Einarsrud, 2001; Twej & Al-Sharuee, 2017). Two types of aerogel can be getting according to the surface components, if the surface has more and more of OH group aerogel called hydrophilic, or if its surface is nearly full of H group called hydrophobic aerogel (Du, Liu, Huang, Li, & Zhou, 2016; Wagh & Ingale, 2002; Zong et al., 2015). The most common influence on properties of these two surfaces is the way of drying and the making or not the modification of the surface, the most common influence on these two surface properties is the way of drying and the making or not the modification of the surface, one of the most well-known surface optimizers is chloride-rich compounds that produced during complex chemical processes to extract from the surface and replaced it with chloride (Han et al., 2016; Jemat, Ghazali, Razali, & Otsuka, 2015).

The mutual chemical modification compounds are trimethylchlorosilane (TMCS), phenyltrimethoxysilane (PTMS), hexamethyldisilazane (HMDZ), Trimethylethoxysilane (TMES), and so on which has often been conducted research and experiments (Jiang, Feng, & Feng, 2017; Parale et al., 2013). These are carried out research and experiments in their impact on the structural and surface specifications of the aerogel, where used Ha-Yoon Nah and others Trimethylethoxysilane (TMES), (TMCS) and TMES/TMCS mixture as co-precursor in order to improve the hydrophobicity property and study the effect it on surface property, they found when they mixed TMES and TMCS to gather the surface and hydrophobicity property has been enhanced (Nah et al., 2018). While A. Venkateswara Rao and other make comparison between two organosilane compounds the study give a good results but the optioned aerogels are opaque

(A. V. Rao, Pajonk, Bhagat, & Barboux, 2004). Whereas compare A. Parvathy Rao with some researchers between different agents and their influence on physical properties of silica aerogel prepared in ambient pressure (A. P. Rao, Rao, & Pajonk, 2007). Subramaniam Iswar and some researchers are study the effect the aging is studied on the physical and chemical characteristic of silica monolithic, their results refer that the surface area and density reductions with increasing aging time arriving to *Ostwald ripening* processes (Iswar et al., 2017; Voorhees, 1985). Another researchers investigated the influence the changing of another parameters on main properties of silica aerogel prepared via ambient pressure drying, and they improved and controlled on many of these properties (He et al., 2018; Mahadik, Lee, Chavan, Mahadik, & Park, 2016; Yun et al., 2017) . In the present work we investigate the effect of optimization tools on surface properties of silica aerogel under ambient pressure preparation, as well as make theoretical investigation for the effect of modification agent on surface property, and find best equation controls behavior of modified silica aerogel through limited conditions.

## 2. Materials and Experimental Procedure

Two step acid base catalysis used to prepare the alcogel, the source of silica was take from tetraethoxysilane (TEOS) as pressures dissolved in ethanol and hydrochloride acid with [0.001M], the molar ratio of mixture (condensed silica (CS)) was  $1:2.8:0.19 \times 10^{-3}$  respectively. CS was put under magnetic stirrer for 1h and kept for hydrolysis for 24h. After one day added mixture consist of (NH<sub>4</sub>OH+NH<sub>4</sub>F) as base catalysis to the CS, the molar concentration of catalysis was [0.05, 0.002M] respectively, and the molar ratio of TEOS: NH<sub>4</sub>OH: NH<sub>4</sub>F was  $1: 0.05: 0.2 \times 10^{-3}$  respectively. The result sol was saved in a tightly sealed plastic box and left under room temperature until it was transformed into alcogel, finally kept in an oven at 60°C for 30 min in order to support of the silica network, then washed in ethanol many times for the purpose of purification from unwanted plankton, the ethanol replaced with hexane under ambient pressure and room temperature, the made the modification to the surface by silylating agent, TMCS once and again HMDZ at 60°C for 24 for every concentration. The concentration of these silylating was varied 5% to 15% of the solvent. In the last step the modified gel put in oven at 80°C then 40°C then dried at room temperature less than 2 day. The result was light and hydrophobic aerogel with high transparency ready for the necessary tests.

### 3. Methods and Characterization

The hydrophobicity of samples give a good estimation of what the changing in the surface because of depending upon the chemical composition of the surface and surface tension (Wagh, Ingale, & Gupta, 2010). The specific surface area is measured by using the "Brunauer – Emmett–Teller (BET) method" at  $P/P_0 < 0.3$ ". Measuring of contact angle are provided on (a VCA Optima goniometer (AST Products Inc., USA)), where (0.3 $\mu$ l) drops of water controlled by syringe system and distributed drop by drop on tope surface of the aerogel, contact angle meter software used to calculate the contact angle on foundation of the droplet shape in the image. In addition to contact angle measurement can learn about the degree of hydrophobicity as well as surface free energy of the aerogels, where there are no direct ways can help to determine the surface free energy of aerogels. In the present work we determined the surface tension of samples by Neumann's and Young's equation of state, since there are required only one liquid in the calculation of seeming surface free energy but it has limitation that neither the dispersion nor the polar component can be evaluated (Żenkiewicz, 2007).

### 4. Young's and Neumann's Equations

The stationary contact angle is administered by the force equilibrium at the three phase boundary and is specified by Young's equation, there is a relationship between the contact angle  $\theta$  and the interfacial surface tension of liquid vapor interface  $\sigma_{lv}$ , the interfacial surface tension of solid vapor interface  $\sigma_{sl}$  and liquid solid interface  $\sigma_{sv}$  and the surface free energy of the solid as shown in below equation:

$$\sigma_{sv} = \sigma_{sl} + \sigma_{lv} \cos \theta \quad (1)$$

"It is clear that the contact angle is function of the surface tension of solid and liquid".

It is know that the surface tension of solids is less than the surface tension of liquid ( $\sigma_{lv} > \sigma_{sv}$ ), leading to reduce the contact angle with solid surface , furthermore surface tension of liquid is less than of solid then total dampening happens which refer to contact angle is almost equal ( $\theta = 0^\circ$ )(Kwok, Ng, & Neumann, 2000).

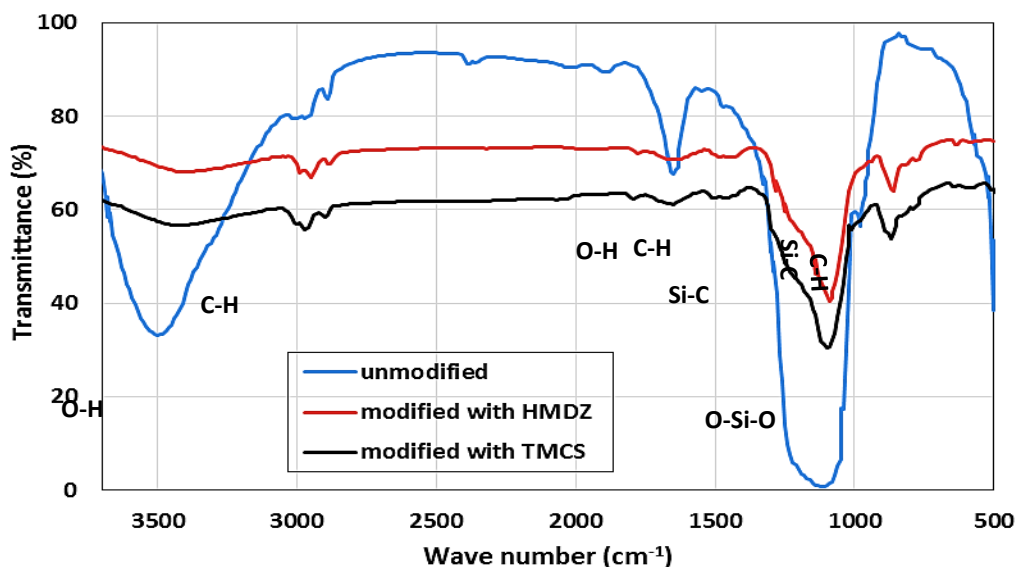
In order to be able to calculate the surface free energy from the contact angle, the second unknown variable  $\sigma_{sl}$  must be determined. Neumann has proposed the following equation for calculating the interfacial tension (Tavana, Jehnichen, Grundke, Hair, & Neumann, 2007):

$$\sigma_{sl} = \sigma_l + \sigma_s - 2\sqrt{\sigma_l \cdot \sigma_s} e^{-\beta(\sigma_l - \sigma_s)^2} \quad (2)$$

the constant  $\beta$  has been determined empirically and take a value  $0.000125 \text{ (mJ/m}^2\text{)}^{-2}$ . Fourier transform infrared spectroscopy (FTIR-8400S Fourier Infrared spectrophotometer SHIMADZU) spectra has been used to detect the change in the chemical bonding of the silica aerogel with variation of modification agent. Fitting curves for parameters study were taken by the program "Table curve 2D Version 5.01".

## 5. Results and Discussion

Figure (1) illustrated the FTIR spectra of silica aerogel samples where (blue line) refer to unmodified sample, (red line) modified with HMDZ and (black line) modified with TMCS. For all samples the peak lies at around  $(1150 \text{ cm}^{-1})$  refer to asymmetric bending mode of silicon dioxide, while the effect of modification in HMDZ and TMCS agents is very clear in FTIR spectra in region at  $(3400 \text{ and } 1560 \text{ cm}^{-1})$  which attributed to  $(-\text{OH})$  bond, these bonds reduced compared with unmodified samples means the enhanced of the surface aerogels, in addition to, it has been note the peaks lie at  $(840 \text{ and } 1250 \text{ cm}^{-1})$  are attributed to  $(\text{Si}-\text{C})$  bond. The absorption peaks observed at  $(1400 \text{ and } 2950 \text{ cm}^{-1})$  due to bending and stretching of  $(\text{C}-\text{H})$  bond (A. V. Rao, Kulkarni, & Bhagat, 2005). From FTIR spectrum it can be say that the enhancement of surface by HMDZ and TMCS silylating agents was successful by replacing the polar bond  $(-\text{OH})$  by nonpolar bond such as  $(\text{Si}-\text{C}$  and  $\text{C}-\text{H})$  leading to hydrophobic surface of aerogel.



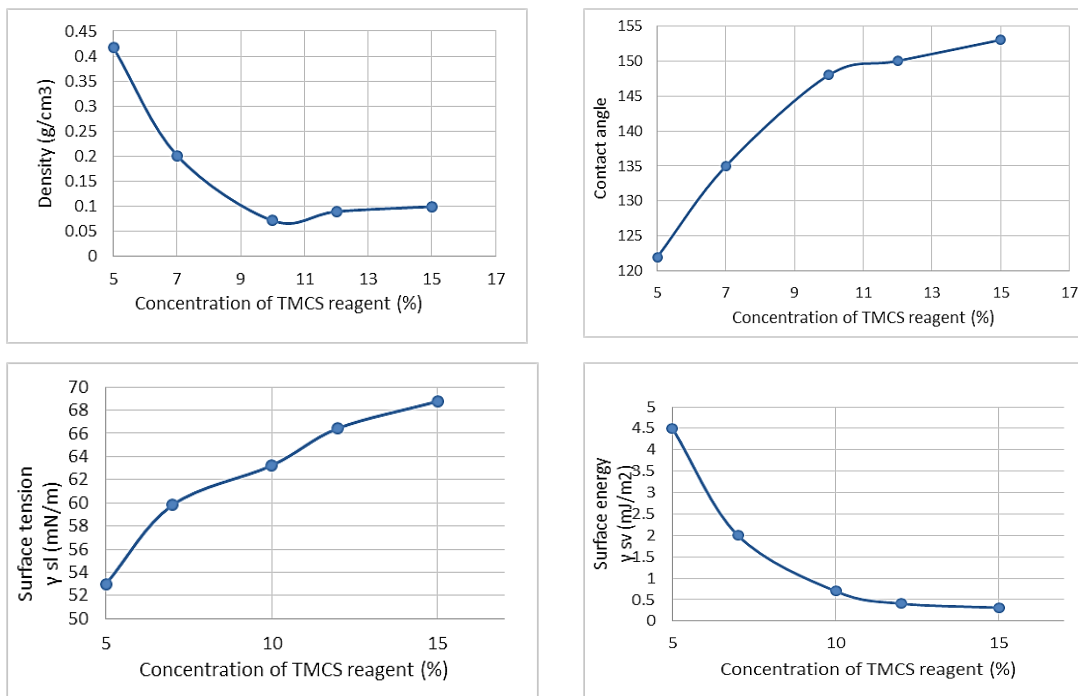
**Figure 1:** FTIR spectra of silica aerogel samples for unmodified sample, modified with HMDZ and modified with TMCS

Table (1) represent to the surface specifications of aerogel represented by contact angle, surface tension, density and surface energy and the effect of concentration of agents on these parameters.

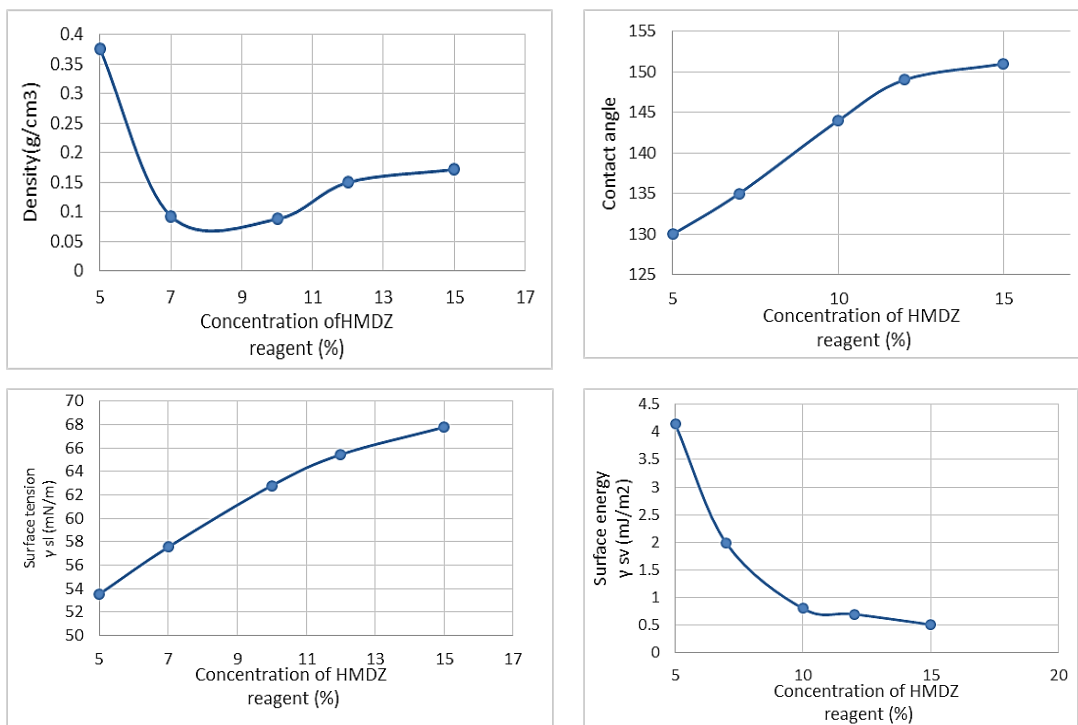
**Table 1:** Effect the Concentration of (HMDZ and TMCS) on Surface Specifications of Aerogel

Modification in HMDZ Silylating Reagents				
Concentration (%)	Density (g/cm <sup>3</sup> )	Contact angle (θ)	Surface tension σ <sub>sl</sub> (mN/m)	Surface energy σ <sub>sv</sub> (mJ/m <sup>2</sup> )
5	0.376	130	53.54	4.1419
7	0.093	135	57.55	1.9813
10	0.088	144	62.8	0.8018
12	0.15	149	65.44	0.7001
15	0.172	151	67.78	0.5112
Modification in TMCS silylating reagents				
5	0.417	122	52.98	4.4997
7	0.201	135	59.8	2.0036
10	0.071	148	63.23	0.6989
12	0.089	150	66.44	0.4091
15	0.099	153	68.77	0.3115

As presented in figure (2) and table (1), when modified aerogel samples in HMDZ silylating and with increase the concentration of silylating reagent contact angle (θ) and surface tension (σ<sub>sl</sub>) to be increased from 130 to 151, and 53.54 to 67.78 mN/m respectively, while this results was reverse in case of density and surface energy, it's found the density reduced from (0.376 to 0.172 g/cm<sup>3</sup>) and surface energy from (4.1419 to 0.5112 mJ/m<sup>2</sup>). These results are same in case of modification in TMCS but with more equivalents, as with increase of its concentration the contact angle (θ) and surface tension (σ<sub>sl</sub>) to be increased from 122 to 153, and 52.98 to 68.77 mN/m respectively, similarity the reducing in density and surface energy was from (0.417 to 0.099 g/cm<sup>3</sup>) in case of density and from (4.4997 to 0.3115 mJ/m<sup>2</sup>) for surface tension. The effect of changed concentration on these parameters represent in below figures



(a) Modification in TMCS Silylating Reagents



(b) Modification in HMDS Silylating Reagents

**Figure 2:** Effect of Concentration of (a) TMCS (b) HMDZ on Density, Contact Angle, Surface Tension and Surface Energy Values of modified Silica Aerogel

For experimental curves, firstly it has been used fitting curve by liner function for all parameters with changing the concentration of HMDZ and TMCS silylating reagent. Figure (3) show the fitting curve of modification in four different concentration of HMDZ and TMCS for silica aerogel samples, the using of linear function give the best estimation for sample's behavior and silylating reagents with limited condition which has been used, where there are many parameters and limitation have important rules to give aerogel low density, high surface area and so on, such as pH, catalysis and precursors (Milea et al., 2011; Twej & Al-Sharuee, 2017). Table (2) illustrated the theoretical relation variation of concentration between HMDZ, TMCS and density, contact angle, surface tension, and surface energy.

**Table 2:** Relations of Modeling for Variables and Coefficient of Determination

relation	Fitting equation	r <sup>2</sup>	
		HMDZ	TMCS
Concentration vs. Density	$y^{0.5} = a + be^{-x}$	0.855	0.925
Concentration vs. Contact angle	$y^2 = a + b \frac{\ln x}{x}$	0.984	0.973
Concentration vs. Surface tension	$y^2 = a + b \ln x$	0.996	0.987
Concentration vs. Surface energy	$\ln y = a + b \frac{\ln x}{x}$	0.996	0.998

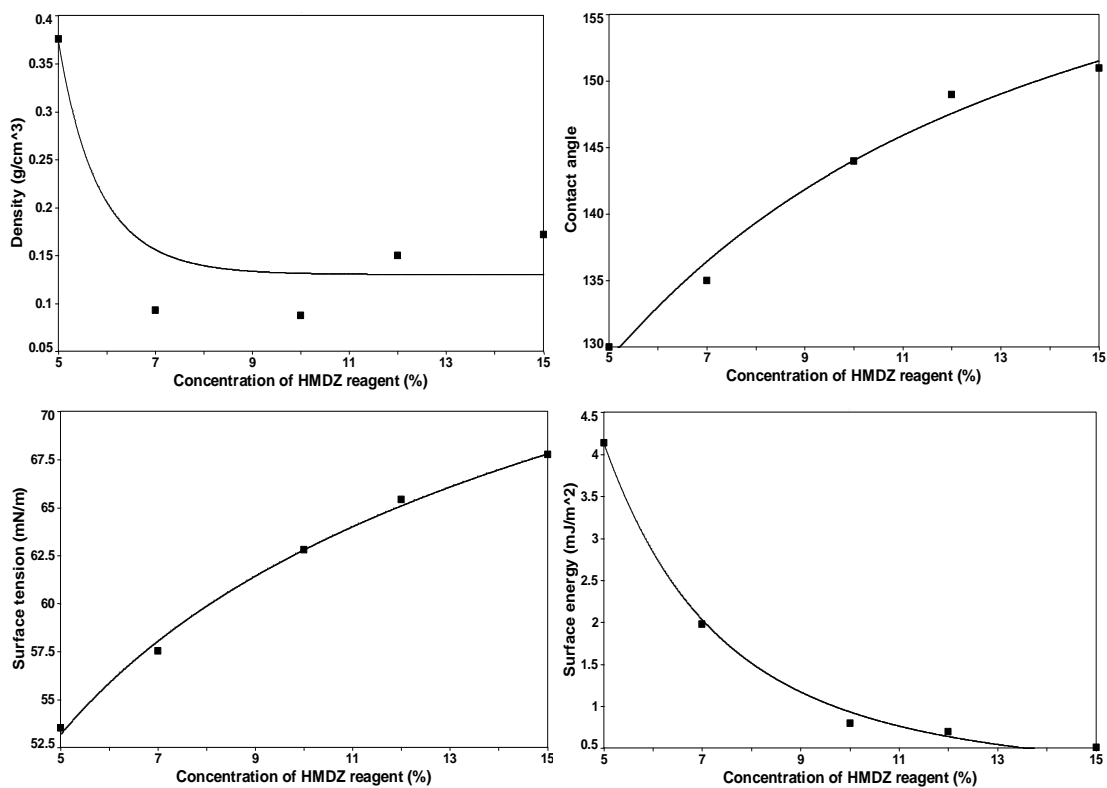
In addition to the advantage of used this function, is it give a nearest matching between experimental and theoretical results as show in (r<sup>2</sup>) value , furthermore there are two coefficients only represented in ( a and b) value as shown in table(3). As well as there are rapprochement comparatively between the HMDZ and TMCS silylating reagent in their influenced on density, surface tension, contact angle and surface energy.

Figure (3) and (4) represents each of the fitting curves for experimental results (theoretical behavior) and experimental results of modified aerogels with different concentration and different modification reagent, and their effect on density, contact angle, surface tension and surface free energy:

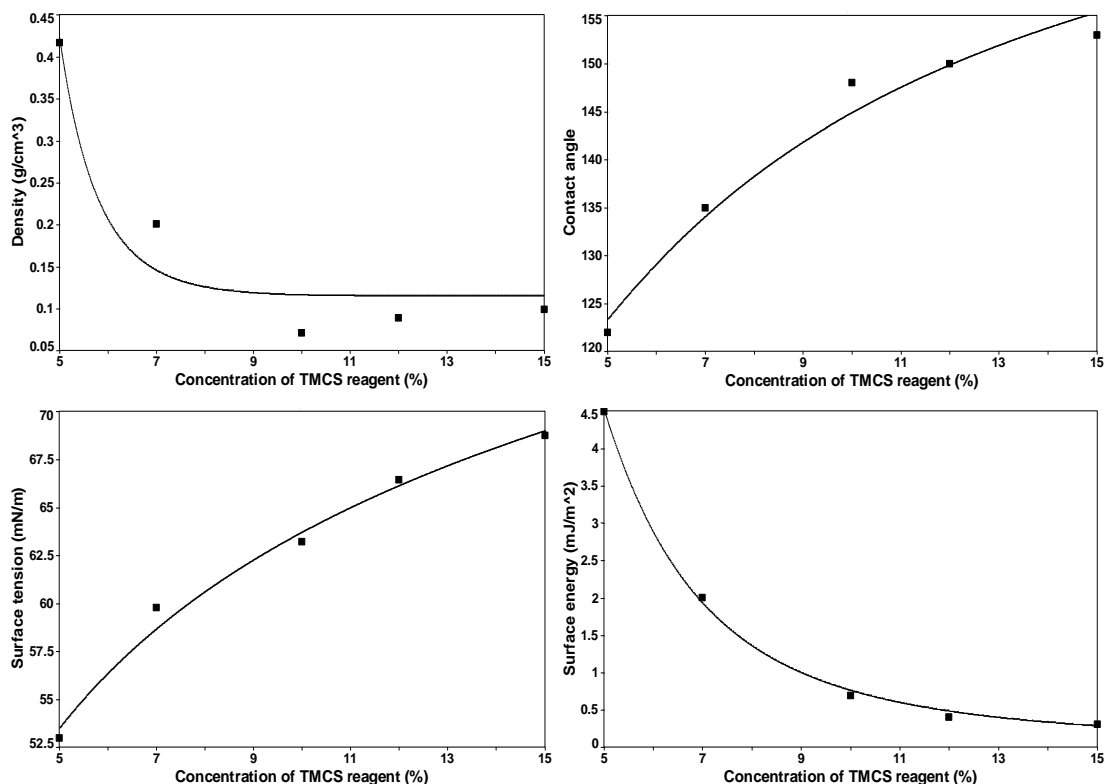


**Table 3: The Value of Equation's Coefficients**

Fitting equation	<i>a</i> - coefficient		<i>b</i> - coefficient	
	HMDZ	TMCS	HMDZ	TMCS
$y^{0.5} = a + be^{-x}$	0.36	0.34	36.9	46
$y^2 = a + b \frac{\ln x}{x}$	30988	35531	-44427	-63071
$y^2 = a + b \ln x$	245	83	1607	1728
$\ln y = a + b \frac{\ln x}{x}$	-3.8	-4.7	16.2	19.3



**Figure 3: Fitting Curve of Modification in Different Concentration of HMDZ for Silica Aerogel Samples**



**Figure 4:** Fitting Curve of Modification in Different Concentration of TMCS for Silica Aerogel Samples

For two cases of modification observed that the fitting curve of density is far away from experimental results, this is because that the reducing of aerogel density affected by nature of silica network, where the broken of bonds from or aggregation in particles cause reduced in density in the same conditions of preparation and with withdraw these causes, density may be change.

While noticed there are near of surface tension, contact angle and surface energy between experimental and theoretical result in limited range of concentration from 5% to 15%, so it can be utilize this fitting curve to determine this parameters without preparation of samples.

## 6. Conclusions

The modification was by hexamethyldisilazane (HMDZ) and trimethylchlorosilane (TMCS) with different concentration enhanced the crucial properties of hydrophobic silica aerogel, by increasing the contact angle and surface tension, also the experimental and theoretical results closed together range of concentration from 5% to 15%, so it can be utilize this fitting curve to determine this parameters without preparation of samples. In addition to the

use of fitting curve by liner function give a best closer results between experimental and theoretical results.

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