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OPTIMIZATION OF COMMERCIAL SILICA GEL REGENERATION VIA RESPONSE SURFACE METHODOLOGY

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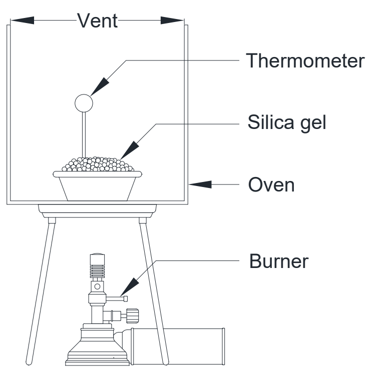
Graphical abstract



Experimental setup

Abstract

Silica gel, a highly porous form of silicon dioxide, is widely used as a desiccant due to its hygroscopic properties and superior characteristics compared to organic materials. Although silica gel can be regenerated and reused, conventional methods often involve prolonged heating and limited ventilation, resulting in energy waste and reduced efficiency. To optimize the regeneration process, this paper presents an experimental approach to regenerate silica gel using response surface methodology (RSM). The study explores the influence of temperature, duration, and natural convection rate, which is regulated by adjusting the vent size of the oven. The results indicate that saturated silica gel at 98-100% equilibrium relative humidity (RH) can be effectively regenerated to nearly its dry weight by applying a temperature of 70°C for a duration of 10 minutes in a cubic oven measuring 15cm on each side, with an approximate vent area of 225cm^2. Moreover, assigning different levels of importance to the influencing factors in the Design Expert software allows calibration of these factors to achieve the desired equilibrium RH.

Keywords: Desiccant, optimization, silica gel, desiccant regeneration, response surface methodology

1.0 INTRODUCTION

Silica gel is the amorphous and porous form of silicon dioxide. It has a high surface area of 700 to 800 square meters per gram [1] which makes it a desiccant with good hygroscopic properties. Moisture indicators such as cobalt(II) chloride and methyl violet may be doped into silica gel as color indicators of its saturation level. In contrast with hygroscopic organic materials such as wood, wool and cotton, silica gel is considered superior by being denser, chemically inert and having higher adsorption capacity. In addition, it retains its stable hard structure even at saturated levels whereas organic materials expand and become softer when saturated [2].

Silica gel has been historically known since the 1640s and in World War I for its usage in the adsorption of vapors and gases in gas mask canister. However, it was not until World War II that it received commercial recognition where it was used in the transport of pharmaceutical and military supplies [3]. In modern times, commercial silica gel is manufactured in grains or beads and is commonly used as a desiccant. Manufactured goods such as food, clothing, and electronics are packaged with silica gel to protect them from early spoilage, mold growth and damage from condensation. In process applications, silica gel is used in chromatography and in drying industrial compressed air systems. It is also widely found inside museum display cases as a humidity buffering agent to prevent mechanical damage to objects caused by fluctuating relative humidity. More recently, silica gel has found an application in air conditioning, particularly in evaporative cooling and dehumidification. It can also be combined with other molecular sieves to form a composite desiccant material which has 20-40% higher dehumidification rates compared to plain silica gel [4].

Due to its high availability and low cost, commercial silica gel is often discarded and seldom recycled after use. Unknown to many, silica gel may be regenerated and reused same as other desiccants. The most effective way of removing adsorbed moisture in silica gel is by heating. Conventional regeneration methods of commercial silica gel are often conducted in industrial ovens where it is left for minutes to hours in an elevated temperature depending on the manufacturer's recommendation [5]. While this ensures the success of regeneration, it wastes immeasurable amount of energy by heating the silica gel for unnecessarily prolonged periods of time and under higher than needed temperatures. Aside from this, ovens are confined spaces which limit air flow. Without adequate ventilation, air saturated with moisture is restricted to escape the oven and drying efficiency is decreased. Considering all these reasons, an optimization of the silica gel regeneration process is of importance. In order to create an efficient and optimal regeneration process of silica gel, all sources of energy consumption must be minimized. In addition, all parameters that mainly influence the regeneration must be investigated.

From a chemistry standpoint, regeneration of silica gel can be considered as a desorption process where trapped water molecules are considered the adsorbate and the surface of the pores is the adsorbent. Adsorption, which is the reverse of desorption, is attributed to multilayer surface adsorption and capillary condensation in the porous network of the desiccant. In a macrostate, adsorption to a solid surface from a gaseous phase is governed mainly by the vapor pressure and temperature [6]. Reversibly, regeneration of silica gel then depends on these parameters. Temperature represents the average kinetic energy of the adsorbed water molecules and influences how much of it escapes the surface. Similarly, vapor pressure indicates the evaporation rate of the water molecules by influencing the water-holding capacity of the surrounding air. Both temperature and vapor pressure can be directly influenced by heating and ventilation. However, the levels of which and how they affect regeneration have to be specified.

While silica gel has a high melting point of 1600°C, it loses its hygroscopic properties if heated above 300°C and newer indicating silica gels have heat-sensitive organic dyes that degrade when heated to about 125-150°C. Thus, the upper limits for regeneration temperature could be set at 120°C and 200°C for indicating silica gel and regular gel, respectively [7]. With efforts toward energy conservation, studies have also looked into lower regeneration temperature limits and the use of renewable heat inputs such as solar and waste heat. Lower regeneration temperatures offer the advantages of lower heating requirement, lower equipment cost and less heat loss. On a study involving a solar heater with an evacuated tube solar collector, silica gel regeneration was demonstrated at temperatures of 54.3-68.3°C with air flow rates ranging from 88-138kg/h [8]. Another study was able to demonstrate regeneration of silica gel at temperatures as low as 40°C with 0.03 kg/s of fresh air flow rate using a solar heater with a parabolic concentrator collector. It was also noted that the regeneration rate is more strongly dependent on the air flow rate rather than the magnitude of heat applied. However, at temperatures lower than 40°C, recondensation occurs [9].

In this study, an experimental method for the optimization of silica gel regeneration is presented. As pointed out earlier, the two main factors affecting regeneration are the temperature and vapor pressure. These are directly and indirectly controlled by heating the silica gel on a vented heating chamber and under different temperatures and durations. While introducing artificial ventilation proved to be beneficial in the regeneration process, this comes with energy usage and additional costs [5,9,10,11]. Unlike the heat source, a way of directly controlling air flow rate through the use of renewable energy may be very difficult and have not yet been successfully demonstrated for this purpose. Thus, natural convection through the vent is instead investigated in this study. In fact, there is a lack of literature about regenerating silica gel with natural convection as the main way of controlling ventilation. The rate of natural convection can be controlled by varying the vent size of the heating chamber. With increasing vent size, the rate of natural convection can also be predicted to increase. However, with increasing vent size and rate of natural convection, heat loss also increases and fresh, colder air coming in have lower water-holding capacity than the hot air leaving. Therefore, finding the optimal vent size that would allow adequate ventilation while minimizing heat loss is one of the goals of this study together with the lowest-possible temperature and duration of the regeneration process.

2.0 METHODOLOGY

A response surface methodology (RSM) specifically, central composite design (CCD) using Design Expert is adopted in this study for the optimization of silica gel regeneration [12]. Three explanatory variables are selected for investigation, namely temperature, duration and vent size. The response variable in this study is determined by the amount of moisture evaporated, which is measured by the weight difference of the silica gel before and after regeneration. The experimental setup for this study is illustrated in Figure 2. Fiberglass, with its high melting point and thermal resistance, is used for the walls of the heating chamber or oven which provides structural integrity during the heating process. Additionally, fiberglass has a relatively higher thermal conductivity which ensures minimal error in the experiments caused by heat loss. The chamber is in the form of a cube with equal sides of 15cm. A WIKA Model A52 bimetallic thermometer was used to accurately gauge the temperature of the heated surface on the oven. The chamber is supported by a cast iron tripod with wire gauze in between for better heat distribution and resistance. A blue indicating silica gel (Figure 3) is used in the experimentation that indicates the saturation level by changing colors from blue to pink.

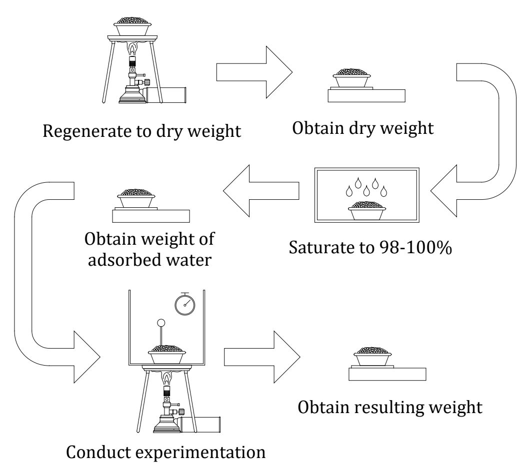


Figure 1 Methodology framework

The experimental procedure (Figure 1) starts with completely regenerating the silica gel to determine its dry weight. Then, it was packed into 5g batches for the experiments. These batches were saturated with moisture and weighed to obtain the amount of adsorbed water. According to the order of the run, each batch of silica gel was employed in the experimentation, with varying factors, namely the temperature, duration, and vent size. Finally, the resulting weight is recorded, and the amount of evaporated water is calculated and documented.

Heat is supplied to the chamber through a Bunsen burner fueled by butane gas. To achieve the desired temperature at the chamber floor, the flame from the burner is adjusted using both the air and fuel valves, while the thermometer monitors the temperature. Once the required temperature is reached, the silica gel is placed inside the oven, and countdown begins. The top side of the oven serves as a vent, and its size can be adjusted by manipulating a lid. For each experimental run, 5g of saturated silica gel is regenerated. Once the duration has been reached, the container holding the silica gel is retrieved and placed onto an AWS Model AWS-100-BLK digital weighing scale. The resulting weight is obtained by subtracting the weight of the container.

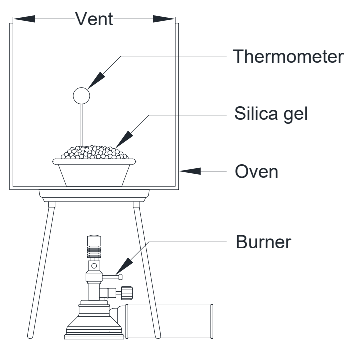


Figure 2 Experimental set-up

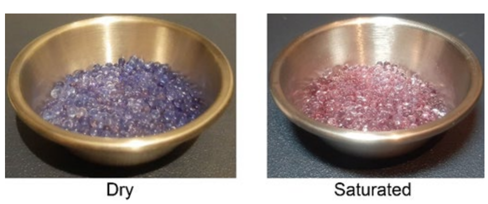


Figure 3 Silica gel saturation colors

In order to achieve accurate and comprehensive results, it is important set a reference point for the moisture content as well as provide a defining property for the silica gel. A parameter used to visualize the performance of a specific type of silica gel is the equilibrium moisture content (EMC). It is defined as the moisture content of an object in equilibrium with a specified relative humidity (RH) and is measured as the weight of water expressed as a percentage of its dry weight. This is expressed in the following equation:

$$EMC = W_e / W_d \cdot 100\% \quad (1)$$

where W_e is the weight of adsorbed water in equilibrium at a specified ambient RH and W_d is the dry weight of the silica gel. The weight of adsorbed water is computed by subtracting the dry weight from the weight of silica gel in equilibrium at a specified ambient RH and temperature. EMC is a dimensionless quantity expressed in percentage and is helpful in rating the adsorption performance of hygroscopic materials. Figure 4 shows the EMC/RH isotherm graph of the silica gel used in this experiment. The EMC values were obtained by allowing the silica gel to reach the desired equilibrium relative humidity at 30°C for over 2 days in an airtight sealed container while expediting it by periodically mixing the silica gel and spreading them in a single layer [1]. From the graph, it can be seen that the adsorption performance of the acquired silica gel for this experiment is significantly lower than a regular density gel which may be due to the addition of the color indicators.

The factors and their high and low treatment levels are listed in Table 1. These are used in determining the parameters of all the runs in the experiments. A temperature low level treatment of 50°C is selected in order to investigate how much regeneration may be done in low temperature. While it has been demonstrated that regeneration may start as low as 40°C [9], it was conducted with artificial ventilation.

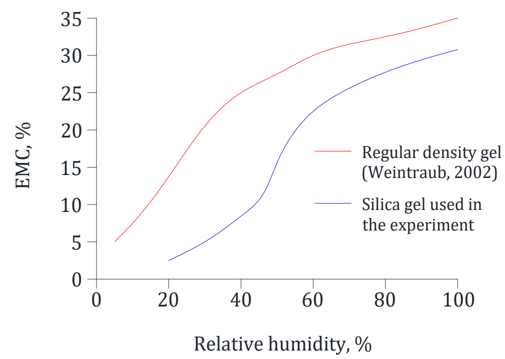


Figure 4 EMC/RH isotherm graph of the silica gel used in this study versus a regular density gel [1]

Thus, a higher temperature is selected for regeneration under natural convection. On the other hand, the high level is set at 80°C to ensure that the temperature of 120°C that would damage the color indicator of silica gel [7] would not be readily reached while also keeping it still relatively low for the purpose of energy savings. For the duration, low and high levels are set at 5 and 10 mins, respectively because of time constraints as well as to minimize the effects of fluctuating ambient temperature and relative humidity throughout the day of experimentation. For the vent size, the high treatment level is set at 225cm² as it is the surface area of the top face of the cubic oven. This high treatment level ensures that maximum convection is achieved according to the oven's geometry. On the other hand, the low treatment level is set at half the high treatment level at 112.5cm² in order not to restrict natural convection too much.

Table 1 Experimental factors and treatment levels

No.	Factors	Units	Low value	High value
1	Temperature	°C	50	80
2	Duration	mins	10	30
3	Vent Size	cm²	112.5	225

The experimentation was carried out during daylight hours, with an average ambient temperature of 31°C and a relative humidity (RH) of 60%, lasting approximately 6 hours. Prior to the experiments, an ample quantity of saturated silica gel at approximately 98-100% RH was prepared and sealed in airtight containers. The factor values and order of runs were assigned by the Design Expert software to adequately explore the factor space and minimize bias. A fixed amount of 5g of silica gel was precisely measured and placed on the sample container (Fig. 2), while ensuring it was evenly spread on a single layer and placed inside the oven. This ensures better heat transfer between the wall of the oven and the silica gel grains. A thermometer probe is to touch the bottom of the container every time temperature is taken and must be held for the manufacturer's recommended response time [13]. Once the desired temperature was attained and maintained on the heated surface of the container, the silica gel was carefully placed inside, and the timer was set for the required duration. Upon completion of the set duration, the silica gel was promptly removed from the oven and weighed to determine the amount of evaporated moisture.

3.0 RESULTS AND DISCUSSION

The experimentation produced the results indicated in Table 2. A quadratic regression model was initially suggested and adopted as its R-squared statistic is above 90%. In the analysis of variance (ANOVA), Backward Elimination method was used to improve and determine the final modified regression model [14]. Following hierarchy, the model term with the largest P-value is excluded from the model with the threshold set at 0.05. This process is repeated until no model term exceeds the threshold. In this process, the model term for the interaction of the duration and vent size was removed as it was implied to be insignificant. The ANOVA table of the final model is shown in Table 3. The final model has an F-value of 88.10 which implies that the model is significant and that there is only a 0.01% chance that this could occur due to noise. Consequently, the Lack of Fit F-value of 0.46 and P-value of 0.81 implies that the Lack of Fit is not significant relative to the pure error. Finally, an adequate precision of 32.10 indicates that the model can be used to navigate the design space.

Table 2 Results of experimental runs

Order	Run	Temp., °C	Time, min	Vent Size, cm^2	Evap. Water, g
1	4	50	5	112.5	0.18
2	15	70	5	112.5	0.47
3	9	50	10	112.5	0.24
4	18	70	10	112.5	0.97
5	10	50	5	225	0.19
6	5	70	5	225	0.70
7	3	50	10	225	0.30
8	8	70	10	225	1.13
9	13	43.18	7.5	168.75	0.13
10	11	76.82	7.5	168.75	1.01
11	7	60	3.3	168.75	0.20
12	16	60	11.7	168.75	0.66
13	14	60	7.5	74.15	0.42
14	2	60	7.5	225	0.41
15	19	60	7.5	168.75	0.37
16	1	60	7.5	168.75	0.39
17	12	60	7.5	168.75	0.28
18	6	60	7.5	168.75	0.32
19	17	60	7.5	168.75	0.44
20	20	60	7.5	168.75	0.34

Model adequacy checks were also conducted to confirm that the model does not violate any assumptions of ANOVA [15]. The first model adequacy check is the normality assumption. This is shown in Figure 5 which can be observed to approximately follow a straight line. Another check is for the residual independence and homogeneity of the variance. From the plots in Figures 6 and 7, it can be seen that the residuals are scattered without any discernable structure or pattern. Thus, the model proves to have not violated any assumptions of ANOVA and can now be interpreted. No outliers were also found that may affect the results negatively.

The final equation of the regression model in terms of coded factors is:

$$Y = 0.08A^2 + 0.03B^2 + 0.06C^2 + 0.10AB + 0.04AC + 0.28A + 0.14B + 0.05C + 0.35 \quad (2)$$

where Y is the amount of evaporated water, A is the temperature, and C is the vent size. The conversion from the actual factors to coded factors is by [16]

$$x = 2\ (X - X_{average} / X_{range}) \quad (3)$$

Table 3 ANOVA of the final modified model

Model Terms	Sum of Squares	DF	Mean Square	F-Value	P-Value
Model	1.57	8	0.20	88.10	0.0001
A	1.08	1	1.08	484.59	0.0001
B	0.26	1	0.26	115.37	0.0001
C	0.028	1	0.028	12.66	0.0045
A^2	0.094	1	0.094	41.99	0.0001
B^2	0.014	1	0.014	6.16	0.0304
C^2	0.027	1	0.027	12.17	0.0051
AB	0.072	1	0.072	32.40	0.0001
AC	0.013	1	0.013	5.74	0.0354
Residual	0.025	11	2.228×10^{-3}		
Lack of Fit	8.776×10^{-3}	6	1.463×10^{-3}	0.46	0.8109
Pure Error	0.016	5	3.147×10^{-3}		
Corr. Total	1.59	19			

Figures 8-10 show the relationships of each factor to the response variable. In summary, the response variable non-linearly increases as any of the factors increases with temperature having the most significant effect followed by the duration and vent size, accordingly. On the other hand, Figures 11-12 show the interactions that were significant between the factors involved. From the graphs, it is suggested that an increase in temperature causes more evaporated water per increase in the duration. This is also true for the vent size but with lesser effect. This may be due to the fact that since temperature increases the average kinetic energy of the water molecules, it also causes more water molecules to escape the silica gel with increasing regeneration duration and decreasing water-holding capacity of surrounding air due to larger vent sizes that allow more natural convection.

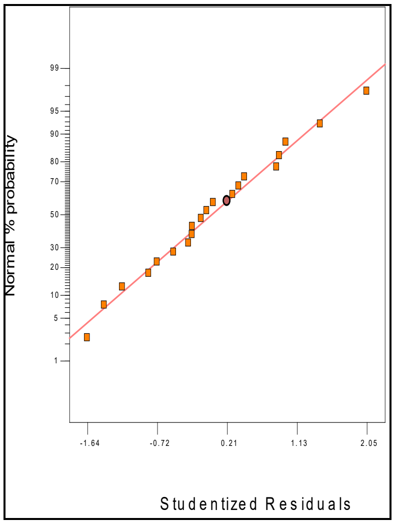


Figure 5 Model adequacy check: normal probability plot of residuals

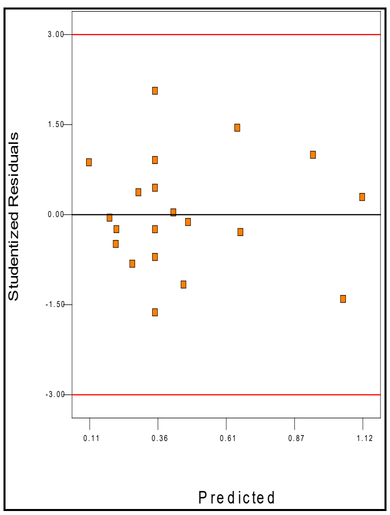


Figure 6 Model adequacy check: residuals vs predicted plot

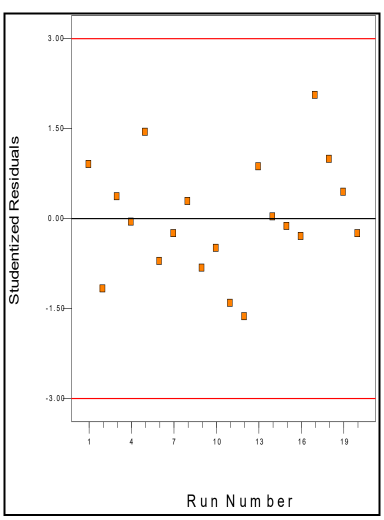


Figure 7 Model adequacy check: residuals vs run plot

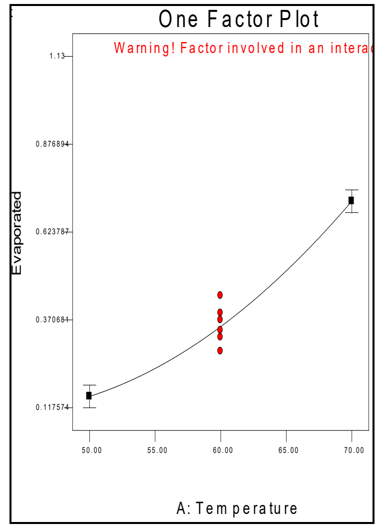


Figure 8 One-factor plot: temperature vs evaporated water

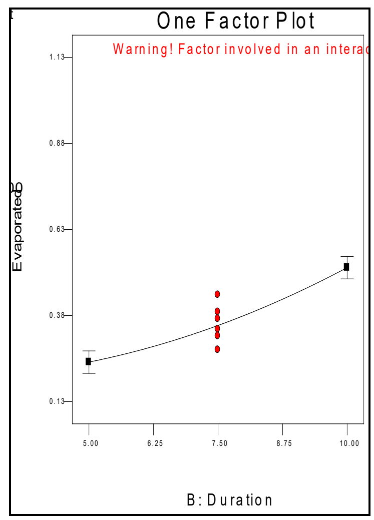


Figure 9 One-factor plot: duration vs evaporated water

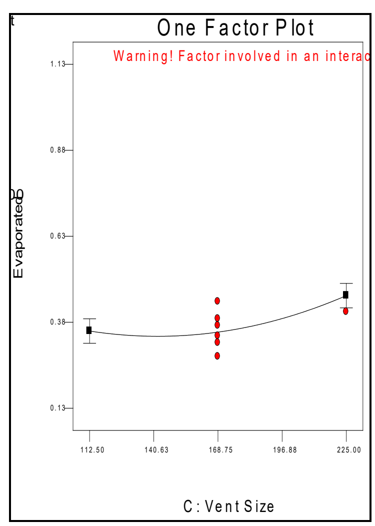


Figure 10 One-factor plot: vent size vs evaporated water

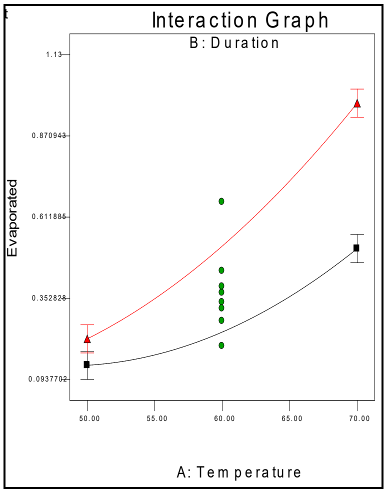


Figure 11 Interaction graph: temperature and duration

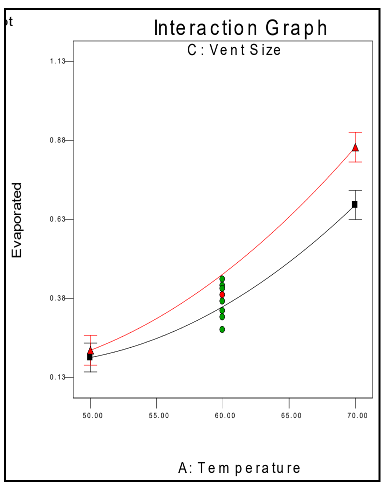


Figure 12 Interaction graph: temperature and vent size

Prior to conducting the experiments, the silica gels were stored in an environment with approximately 98-100% RH and allowed to equilibrate for a period of 2 days [1]. As shown in Figure 4, this equilibration period resulted in an initial EMC of approximately 30% before the regeneration process. In other words, the silica gels were saturated with water, accounting for 30% of their dry weight. Consequently, using Eq. 1, the weight of the saturated silica gel (5g) corresponds to an approximate dry weight of 3.85g. Following the regeneration process, the silica

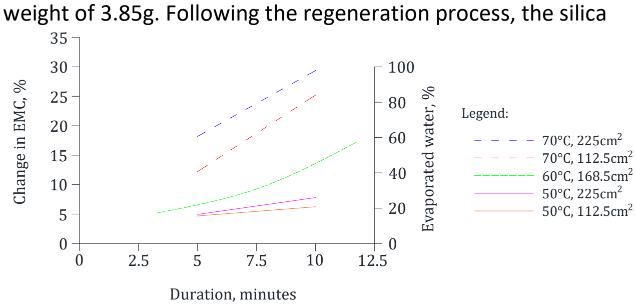


Figure 13 Isotherm graphs of change in EMC vs duration with varying temperatures and vent sizes

gels were weighed again, and the new EMC was determined using Eq. 1. The difference between the EMC values before and after regeneration was then plotted in Figure 13 to illustrate the level of regeneration achieved across various combinations of temperature and vent size. A higher change in EMC indicates more successful regeneration. The graph indicates that at the maximum experimental setting of 70°C temperature and 225cm² vent size, nearly 100% of the adsorbed water has been evaporated from the silica gel.

Employing the numerical optimization feature of Design Expert, several criteria combinations (Table 4) were carried out to find the optimum regeneration factors based on two goals. The range and settings for the factors can be found in Tab. 1. The top results are then presented based on the goal in Table 5 for maximum level of regeneration and Table 6 for maximum energy savings. There are 9 solutions (top 6 listed) found for the goal of maximizing the level of regeneration whereas there are only 3 solutions for energy savings. Since every increase in the factors converts into an increase in the amount of evaporated water, it is obvious that the maximum factors are used in maximizing the level of regeneration. One important thing to note is that when the goal is energy savings, the temperature is not at the minimum of value of 50°C. Instead, 60°C is adopted while the minimum duration value of 5 mins is used to obtain the optimal settings for energy savings. Regardless of both goals, the maximum amount of vent size is used since it does not contribute any energy consumption.

Table 4 Criteria used for optimization

Goal	Temp. (level)	Time (level)	Vent Size (level)	Evaporated Water (level)
Maximum level of regeneration	In range (+++)	In range (+++)	In range (+++)	Maximum (+++++)
Maximum Energy savings	Minimum (+++++)	Minimum (+++++)	In range (+++)	Maximum (+++++)

Table 5 Suggested factor settings for optimization by Design Expert for the goal of maximum level of regeneration

Temp., °C	Time, mins	Vent Size, cm²	Evap. water, g	Resulting EMC, %	Desira-bility
70.00	10.0	224.77	1.12080	1.335038	0.991
70.00	9.97	224.34	1.11584	1.461893	0.986
70.00	10.0	222.18	1.11170	1.567775	0.982
69.83	10.0	225.00	1.11163	1.569565	0.982
70.00	9.99	221.30	1.10784	1.666496	0.978

Table 6 Suggested factor settings for optimization by Design Expert for the goal of maximum energy savings

Temp., °C	Time, mins	Vent Size, cm²	Evap. water, g	Resulting EMC, %	Desira-bility
60.20	5.00	225.00	0.35533	20.91228	0.480
60.12	5.00	224.98	0.35357	20.95729	0.480
61.27	6.03	112.50	0.30914	22.09361	0.396

In practical applications, the level of regeneration is important as it determines the usefulness of a desiccant by the drying potential it can provide, especially in air conditioning applications [17]. The same is true in pharmaceutical industries as well [18]. However, in some cases such as that in museums, silica gel is used as a buffering material that minimizes the fluctuations on ambient RH rather than as a drying agent [19]. This is due to the fact that silica gel adsorbs and also gives off moisture depending on whether the ambient RH is higher or lower than its equilibrium RH. In such applications, regenerating the silica gel back to its dry weight is not necessary and is rather undesirable because it may cause damage to the display object by adsorbing too much moisture from the surrounding air and causing too much RH fluctuation. Therefore, setting a target equilibrium RH at the start is very useful in further optimizing the regeneration of silica gel as it determines the optimum regeneration settings required [20].

As a way of demonstrating this optimization process in this study, consider an end goal of 35% equilibrium RH. Since there is no available data on the corresponding EMC for an equilibrium RH of 35%, it must be approximated from the EMC/RH isotherm graph. The EMC will then be used to solve for the needed amount of evaporated water. For better graph approximation, a MATLAB script was used to graph the EMC/RH isotherm of the silica gel used. Since the graph is non-linear, a cubic spline interpolation is used. Figure 14 shows the interpolated value of the EMC at 35% equilibrium RH.

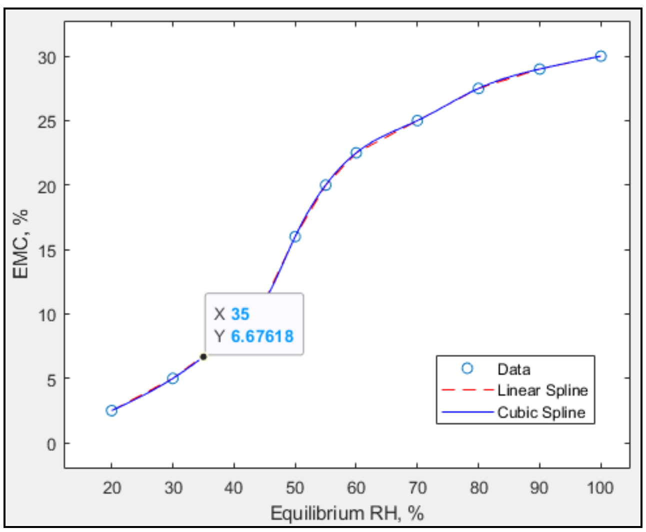


Figure 14 Interpolated values of EMC plotted as a cubic spline generated by a MATLAB script

Finally, Eq. 4 below is used for determining the needed amount of evaporated water from the silica gel after regeneration. The equation was derived from the difference in the EMC being the initial EMC minus the final EMC with the initial EMC at 30% and the dry weight of silica gel at approximately 3.85g.

$$We = 3.85\,(30 - EMC_{desired}) / 100 \qquad (4)$$

Substituting the desired EMC of 6.67618% yields the needed amount of evaporated water of 0.89707g. Then, using the numerical optimization feature of the Design Expert, the settings for the regeneration may now be obtained. The top 3 solutions indicated in Table 7 summarizes the required temperature, duration and vent size settings for the regeneration process. The criteria are for temperature and duration to be in range with 3 importance level, vent size to be maximum with 3 importance level, and the amount of evaporated water is in target with 5 importance level. Any of the listed solutions can generate the goal.

Table 7 Optimum regeneration process solution for a target of 35% equilibrium RH with energy savings as primary goal

Temp., °C	Time, mins	Vent Size, cm^2	Evaporated Water, g	Desirability
69.11	8.36	225.00	0.897069	1.000
68.00	8.93	225.00	0.897069	1.000
67.18	9.34	225.00	0.897072	1.000

4.0 CONCLUSION

In this study, optimization of silica gel regeneration without artificial air flow is presented by using three factors, namely the temperature, duration and vent size via response surface methodology (RSM). The experimentation results suggest that all factors have significant relationships with the response variable (Figs. 8-10). The amount of evaporated water from the silica gel increases for increase in any of the factors with temperature having the most significant effect. In addition, there are significant interactions between temperature vs duration and temperature vs vent size (Figs. 11-12). The trend of the interactions was that for every increase in temperature, both the increase in evaporated water per change in duration and vent size also increases. The interaction is more significant in the regeneration duration.

In the optimization of the regeneration, it was found that 5g of silica gel at approximately 98-100% saturation level may be regenerated back to nearly its dry weight (~1% EMC) at a regeneration temperature of 70°C, duration of 10 mins and vent size of 225cm^2 with only natural convection as its way of ventilation. On the other hand, optimal values considering energy savings is found at a regeneration temperature of around 60°C, duration of 5 mins and vent size of 225cm^2 (~21% EMC). Regeneration optimization was achieved by maximizing evaporated water, while energy-saving optimization involved minimizing both temperature and duration. To put into perspective, the settings used to maximize the amount of evaporated water which maximized the evaporated water while keeping all factors within range of their low and high levels can reach up to nearly 100% resulting evaporated water. while it is around 30% for the settings used in energy savings (Fig. 13).

In this study, the amount of silica gel (5g) and the size of the oven were held constant for the experimentation. Future studies may look into the effects of varying the size of the oven and increasing the amount of silica gel to the rate of regeneration. Furthermore, studies may also examine how the ratio of the vent area concerning the oven size relates to the regeneration rate. Finally, the current study conducted experimentation under approximately the same ambient conditions. Thus, the effects of changing ambient temperature and RH to the regeneration rate may also be investigated.

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