REGENERATIVE CAPACITY OF SILICA GEL FOR GRAIN DRYING

1050 710

by

Gangadhar Vemuganti Rao

B.V.Sc. A. P. Agricultural University, India, 1970

A MASTER'S THESIS

Submitted in partial fulfillment of the

requirements for the degree

MASTER OF SCIENCE

Department of Grain Science and Industry

KANSAS STATE UNIVERSITY
Manhattan, Kansas

1974

Approved by:

Major Professor

LD
2668
T4
1974
R36
C.2
Document

TABLES OF CONTENTS

P	AGE
LIST OF TABLES	i
LIST OF FIGURES	ii
INTRODUCTION	1
LITERATURE REVIEW	3
History	3
Preparation	3
Properties	3
Applications	5
MATERIALS AND METHODS	10
RESULTS AND DISCUSSION	16
APPENDIX	26
CONCLUSION	31
ACKNOWLEDGEMENTS	32
REFERENCES	33

LIST OF TABLES

			PAGE
Table	1.	Representative properties of the three main types of silica gel	5
Tab1e	2.	Silica gel use chart (Davison Chemicals)	7
Table	3.	Regeneration of silica gel (Batch A) using wheat straw as a fuel	20
Table	4.	Regeneration of silica gel (Batch B) using charcoal as fuel	21
Table	5.	Regeneration of silica gel (Batch C) by direct heating using wheat straw as a fuel and steel screen sack as a container	22
Table	6.	Regeneration of silica gel (Batch D) by direct heating using charcoal as fuel and steel screen sack as a container ${\sf var}$	23
Table	7.	Regeneration of silica gel (Batch E) using wheat straw as fuel with a 12 gauge metal sheet underneath the container	24
Table	8.	Regeneration of silica gel (Batch F) using charcoal as fuel with a $\frac{1}{2}$ " sand bed underneath the container	25
Table	9.	Equilibrium moisture content of all batches at $80^{\circ}F$ and 90% relative humidity (a week period)	26
Table	10.	Comparison of particle size with adsorption capacity \dots	27
Table	11.	Temperature readings measured by the Pyrometer with a chromel Alumel Thermocouple	28
Table	12.	Preliminary Lab Tests Showing the Rate of Adsorption (at $85^{\circ}F$ and 90% R. H.) and Rate of Drying ($190^{\circ}F$)	33
Table	13.	Data Sheet Used for Tabulation of Sieving Data and Calculation of Geometric Particle Size or Diameter by Weight	34

LIST OF FIGURES

		PAGE
Figure 1.	Equilibrium moisture for solid desiccants, Chemical Engineer's Handbook	7
Figure 2.	Types of fireplaces and containers	11
Figure 3.	Jacket-type container	12

INTRODUCTION

The moisture content of grain affects processes such as harvesting, storage, feeding, germination and milling of various kinds. Matured grain contains less moisture compared to unripened grain and needs drying to prevent the growth of micro-organisms. The moisture content affects the trading value of the grain,

It is essential to maintain the moisture content of grain at the optimum level during storage. An equilibrium moisture content of above 70 percent relative humidity at room temperature is not suitable for grain storage (7). The equilibrium moisture content is different for each commodity.

The following methods have been used to improve storability and to maintain marketable quality in grains harvested with a high moisture content: 1. Drying to a moisture content safe for storage. 2. Aeration to maintain a low and uniform temperature to prevent moisture migration.

There are several grain drying methods such as sundrying, field drying, mechanically forced heated air, mechanically forced unheated air, etc. Sundrying and field drying depend on climatic conditions. The other artificial drying methods may cause damage to the grain like brittleness, heat damage and reduced nutritive value. Grains are hygroscopic, so by whatever means drying is done they can later gain moisture from high humidity. The Food and Feed Grain Institute at Kansas State University is trying to establish a simple and inexpensive storage unit suitable for humid areas. In their work, silica gel (a drying agent) was placed in the grain mass to dry the grain and to maintain optimum moisture content.

Once the silica gel reaches the maximum adsorbing capacity it needs regeneration for further use. The purpose of the research is to find out how many times the silica gel can be regenerated without losing its adsorbing capacity. Also, to develop a simple and inexpensive regeneration method which does not effect the quality of silica gel.

LITERATURE REVIEW

History:

Silica gel was first known in its hydrogel form. In 1640 Van Helmont reported that amorphous silica becomes liquid in contact with alkali, which later can be precipitated by addition of acid. In 1861 Sir Thomas Graham discovered that the hydrogel may be formed when a hydrosol is destabilized by dialyzing out the electrolyte. He prepared silica gel by dialyzing dilute silica sols obtained by mixing aqueous solutions of sodium silicate and hydrochloric acid (11).

Dr. Walter A. Patrick brought the silica gel into use during the First World War as a possible adsorbent in gas-mask canisters (15). Silica gel production was commercialized in 1920 in the USA.

Preparation:

Silica gel is commercially prepared by mixing sodium silicate solution with a mineral acid. This reaction leads to the formation of hydrosol. Hydrosol is converted into silica gel due to polymerization of silica molecules. Various types of silica gel can be prepared by modifying the reaction conditions such as temperature, concentration and pH. Each variety exhibits specific physical properties (5).

Properties:

Silica gel is classified as: 1. Regular density 2. Intermediate density 3. Low density.

Regular density silica gel has extremely fine pores, which allows higher adsorption of water. It is hard and glassy. Intermediate density silica gel is characterized by larger average pore diameter; it lacks fine

pores which reduce adsorption capacity of water. This type of silica gel is more friable. Low density silica gel is characterized by an even larger pore diameter (11). The full description of properties is given in Table 1 (11).

Silica gel has a capacity to adsorb sulfur dioxide, acetone and benzene. Also, silica gel shows specific and selective adsorption for water. It retains its adsorptive capacity even below $100^{\circ}F$ (11, 15, 19). The specific heat of silica gel is .22 B.T.U./lb/ $^{\circ}F$ (2, 15). Figure 1 shows the isotherms of silica gel and other adsorbents.

Representative Properties of the Three Main Types of Silica Gel

Table 1

Properties	Regular density	Intermediate density	Low density
density, g/cc			
apparent (bulk)	0.67-0.75	0.35-0.40	0.12-0.17
particle	1.10-4.20	0.65-0.75	
true	2.20	2.20	2.20
surface area m²/g	750-800	300-350	100-200
pore volume, ml/g	0.37-0.40	0.90-1.10	1.4-2.0
average pore diameter, A ^O	22-26	120-160	180-220

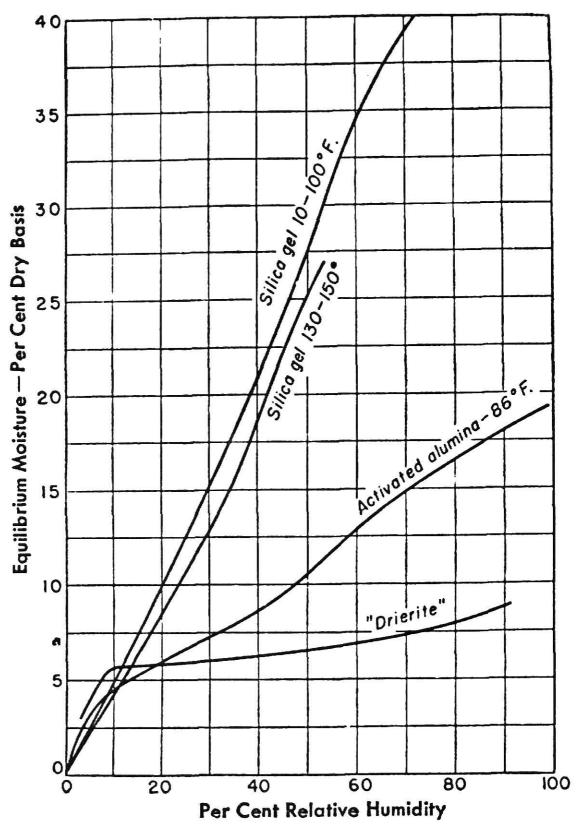


Figure 1. Equilibrium moisture for solid desiccants, Chemical Engineer's Handbook.

Applications:

Silica gel has been used in many industrial and pharmaceutical applications. Table 2 shows its application in industry. The commercial application of silica gel includes: refining of petroleum distillates, desulfurizing motor oils, dehydrating and purifying industrial gases, and serving as catalysts in the preparation of sulfuric acid and hydrogenation of oils (5, 11, 15).

Chemical adsorbents have been used for grain drying for nearly 3 decades (1). Recently, research was conducted by Fleske (1973) on the use of silica gel as a grain drying agent. Several factors were examined in his research. The results indicate that silica gel is a better adsorbent for grain drying than other adsorbents (6).

The ratio of grain to adsorbent required for drying depends inversely on the initial moisture content of grain. The grain to adsorbent ratios for corn with 12%, 15% and 20% initial moisture content were 140:1, 50:1, 35:1 respectively. Furthermore, his data on grain temperature, rate of drying, germination and mold invasion indicated that silica gel can be used for grain drying without adversely affecting the grain quality (6).

Silica gel has other advantages for grain drying. It is chemically inert and in no way reacts with the grain being dried. The drying process with silica gel is relatively slow and hence does not damage the grain (6).

Research is still in progress at Kansas State University to increase the drying rate by aeration while using silica gel. Small bins which can hold 300 lbs. of grain (corn) are used. Silica gel is placed in the grain at different positions. The air movement in the grain mass is brought about by a wind-powered fan placed on top of the bins.

Table 2
Silica Gel Use Chart (Davison Chemicals)

Use Code	Grade	Application
A	01	Dehydration of gases, trace hydrocarbon removal. Usually recommended for special applications where particle size is critical.
В	03	For natural gas and other industrial gases. 1) Bulk dehydration 2) Hydrocarbon recovery
D	05	Liquid and gas dehydrationspecial because of controlled particle size distribution.
E	12,15	Chromatographic gels.
F	13	Drying of flowers.
Н	35	Packagingbecause of size they could be used in selected liquid applications.
I	40	Liquid and gas dehydration.
J	41	Gas dehydrationbecause of particle size it is also recommended for hydrocarbon recovery.
K	42,43,44	Indicatingblue to pink.
N	59	Nondecrepitatingmainly used to top off beds of other gels in order to protect them from entrained liquid water.
P	81	Selective separation.
S	407,408	Bulk liquid dehydration such as refrigerants; airdrying in insulating glass.
T	923,950	Ultrahigh purity chromatographic gels.

•

Once silica gel reaches 70% of its total adsorbing capacity, it needs to be regenerated for further use. Laughlin et al. (12) and Robert et al. (9) discussed commercial methods of regeneration of silica gel.

The regenerative capacity of silica gel is affected by several factors. High temperatures and impurities affect the physical properties of silica gel like capillary volume, bound water, and surface area (8, 11).

The Encyclopedia of Chemical Technology (11) and Markrides (13) indicate that silica gel loses its "bound water" on exposure to 400°F for 3 hours (11). The results in Markrides experiment (13) show that the surface area of silica gel is reduced upon exposure to 700°F.

Patrick et al. (17) indicated that the adsorption capacity drops to a minimum if silica gel is heated to 1000° C for two hours. This drop is due to loss of capillary space.

Maroya et al. (16) studied the effect of various kinds of inorganic salts on physical properties (water adsorption capacity) of silica gel. The cationic impurities such as Na^+ , K^+ , Ca^{+2} , Pb^{+2} , Zn^{+2} exert a strong effect on the water adsorptive capacity of silica gel.

Mantell (15) describes twenty-nine static laboratory tests conducted by Deschner, Hammerschmid and Capell. The results show that the adsorptive capacity of silica gel declines with continued use and reactivation. The results of this investigation are not in agreement with the statement by the Encyclopedia of Chemical Technology (11) that silica gel can be regenerated 1000 times.

Therefore, research was conducted to investigate a simple, efficient and inexpensive method of regeneration of silica gel on farm conditions.

MATERIALS AND METHODS

The experiments were conducted under a shed in an open area. The purpose of the shed was for protection against wind and rain. The shed has a corrugated steel roof with 8' x 16' floor space. The sides were uncovered to give plenty of ventilation to carry the smoke away from the experiment.

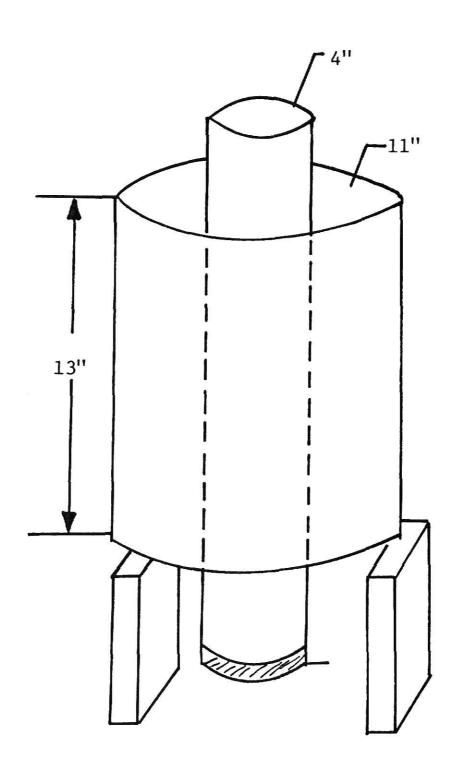
The method for regeneration of silica gel investigated by us was economical and could be operated by unskilled persons. Wheat straw and charcoal were used as fuels. Open and closed fireplaces were designed to burn the fuels. An open fireplace was built with three bricks at 90° angles to hold the containers. Two types of closed fireplaces were studied. One was made out of a metal can (diameter: 11", height: 13") and the other one out of 18 gauge galvanized steel as shown in Figure 2.

A big pan, a small pan and a rotating drum were used to hold the silica gel over the source of heat. The rotating drum was made out of a metal can (diameter: 11", height: 13") as shown in Figure 2. The big pan and the small pan were made out of 18 gauge black steel with dimensions of 18" x 18" x 6"; 12" x 12" x 6" respectively (Figure 2). Later the height of the big pan was raised from 6" to 15" to prevent the smoke from entering the silica gel. A special jacket-type container was also made as shown in Figure 3.

In addition to the above containers, two sacks (8 in. \times 18 in.) were made out of 36 mesh stainless steel bolting cloth, to hold silica gel from direct exposure to heat.

Rotating Drum Closed-Type II Figure 2 Types of Fireplaces and Containers Small Pan Closed-Type I Large Pan Open-Type

Figure 3 Jacket-Type Container



£

The silica gel used in these experiments was a commercial product (TEL-TALE) of The Davison Chemical Division, Baltimore, MD. 21203, USA. It is a regular density silica gel of 42 grade. It contains a colored pigment which will give several reproducible color changes at specific relative humidities. The original color of the silica gel is dark blue and it has a capacity to adsorb 40% of water by weight. As it starts adsorbing moisture it turns from dark blue to light blue; light blue to dark pink; and dark pink to light pink; when it reaches its maximum adsorbing capacity, it turns to light pink. At this stage it needs to be regenerated.

Preliminary laboratory tests were conducted to find the adsorptive capacity and time required to dry the silica gel using a hot air oven. Two kilograms of silica gel were taken in a sieve (diameter: 8", Tyler screen no.: 20). The bed depth was two inches. The sample was placed in a humidifying chamber set at 85°F and 90% relative humidity. Small samples were taken in an aluminum container at 8 hour intervals and dried at 190°C in a hot air oven. The time required to dry the sample to get a uniform weight was 60 minutes. The moisture adsorbed by the sample was 20% by weight after 52 hours. Since it takes a week, or so, to get the silica gel to 30% moisture content, the following method was used for faster wetting of the silica gel.

Hudson's Clipper 6215 Sprayer and a small mixer designed by the Grain Science Department of Kansas State University were used for wetting the silica gel. All the samples were wetted to 30% (75% of its adsorptive capacity) moisture level by adding water with the sprayer while mixing the sample. It is the same as keeping the sample in a humidifying chamber or in the grain mass to be dried.

After wetting, the silica gel was divided into four batches A, B, C, and D. Batch A, weighing 10 lbs., was dried and wetted alternately five times, each time using a different container but the same fuel. The samples were stirred while drying to prevent heat damage to the bottom layer. A big pan was used three times, a rotating drum once and a small pan once. The fuel used was wheat straw. Only open fire-places were used since the closed fireplace did not allow enough air to burn the wheat straw.

Batch B was subjected to the same conditions as Batch A, except that the fuel was charcoal. Charcoal was burned in both open and closed type fire situations. The closed fireplace was made out of a metal can.

Three pounds each of Batch C and D were dried five times in the steel screen sacks by exposure to direct fire. The fuels used for C and D were wheat straw and charcoal respectively.

The data recorded in each experiment included the amount of water added (adsorbed), the amount of water evaporated, the time taken to dry the sample and the amount of fuel used.

Following the five regeneration steps each batch (A, B, C and D) was subjected to the equilibrium moisture test for a week (at 80°F and 90% R.H.), and the particle size analysis. The results were then compared with the original sample.

A method for the equilibrium moisture test was described by Dunstan et al. (4). The particle size analysis was described by Pfost (18).

After observing the physical characteristics of silica gel and the results of the equilibrium moisture test, it was felt that the high temperatures of flame and smoke were destroying the adsorbing capacity

of silica gel. Temperatures were measured by a pyrometer (Eimer and Amend NYC No. 30-38) which has a chromel alumel thermocouple and can read temperatures up to $2200^{\circ}F$.

Two more experiments were conducted (Batch E and F) as before. The weights of Sample E and F were 4 lbs. 6 ozs. and 6 lbs. respectively. Both of them were dried five times. This time the side walls of the big pan were raised to 15" to reduce the smoke contamination. Sample E was dried on an open fireplace. Wheat straw was used as fuel. A 12 gauge metal sheet was placed under the pan to reduce the temperature. Sample F was dried in the small pan on the closed fireplace (the one which was made out of galvanized steel). Charcoal was used as a fuel. The temperature was reduced by placing a ½" sand bed on the 14 gauge steel in between the fireplace and the pan. In other words, the pan was in contact with the sand but not with the fire.

Again, the equilibrium moisture test and the particle size analysis were run on the samples from batches E and F. The results were compared with the original sample and with the batches A to D.

RESULTS AND DISCUSSION

The main objective of the project was to investigate the regenerative capacity of silica gel using simple, efficient and inexpensive methods.

Tables 3 to 8 show the type of containers used (small pan, large pan and rotating drum), the time required to dry the sample and the amount of the fuel used. Also, they show the percentage of water adsorbed (added) and evaporated. Under all drying conditions, the amount of water that evaporated was similar with certain minor variations.

The amount of fuel used depended on the drying time and the drying time depended upon weather conditions (temperature and wind velocity).

Drying a material involves a) vaporization of moisture contained in the material at or beneath the surface and then diffusion of the vapor into the air, and b) the movement of water and/or vapor from the interior to the surface of the material before it can diffuse into the air (3).

The time required for the first phase of drying was about 2/3 of total time required for drying and at this stage silica gel becomes loose and dry without much change in the color. The second phase was about 1/3 of total time required for drying. The second phase is very important because the color changes are very fast. Since all the water is evaporated at this stage the bottom layer of silica gel is more prone to heat damage if left unstirred.

Table 3 shows the results of Batch A, silica gel dried five times on an open fireplace, using wheat straw as a fuel. Table 4 indicates the results of Batch B, silica gel dried five times on open and closed fireplaces, using charcoal as fuel. Tables 5 and 6 show the results of Batches C and D silica gels dried in steel screen sacks by direct exposure to fire.

Table 3

Regeneration of silica gel (Batch A) using wheat straw as a fuel

Amount	fuel	1 bale	3/4 bale	½ bale	½ bale	½ bale
Time required to evaporate a lb.	of water	48.33 min.	37.6 min.	26.69 min.	17.58 min.	18.51 min.
Water porated	%	30	25	28.12	29.71	28.67
Water evaporated	weight	3 lbs	2 lbs 8 ozs	2 lbs 13 ozs	2 lbs 9 ozs	2 lbs 7 ozs
dsorbed ed)	%	30	30	28.12	30.40	29.59
Water adsorbed (added)	weight	3 1bs	3 1bs	2 lbs 13 ozs	2 1bs 10 ozs	2 lbs 8 ozs
Final weight	of gel	7 lbs	7 1bs 8 ozs	7 1bs 3 ozs	6 1bs 1 oz	6 1bs 1 oz
Initial weight	of gel	10 lbs	10 lbs	10 lbs	8 lbs 10 ozs	8 1bs 8 ozs
	Container	small pan (3" bed depth)	rotating drum	big pan (1½'bed depth)	big pan	big pan
	Fireplace	open type	open type	open type	open type	closed type
Test	No.	П	2	е	4	5

Table 4

Regeneration of silica gel (Batch B) using charcoal as fuel

Amount	of	fuel		10 lbs	10 lbs	10 lbs	8 1bs	8 lbs
Time required to	evaporate a lb.	of water		36.36 min.	47.27 min.	33.33 min.	21.53	43.77
er .	rated	%	2	27.5	27.5	30.37	29.30	29.20
Water	evaporated	weight		2 lbs 12 ozs	2 1bs 12 ozs	3 lbs	2 1bs 14 ozs	2 lbs 13 ozs
sorbed		%	0	30	30	30.37	29.94	29.87
Water adsorbed	(added)	weight	Q.	3 lbs	3 Ibs	3 1bs	2 lbs 15 ozs	2 lbs 14 ozs
Final	weight	of gel		7 1bs 4 ozs	7 lbs 4 ozs	6 lbs 14 ozs	6 1bs 15 ozs	6 lbs 13 ozs
Initia1	weight	of gel		10 lbs	10 lbs	9 lbs 14 ozs	9 lbs 13 ozs	9 lbs 10 ozs
1) A 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1		Container		big pan	small pan	small pan	big pan	rotating drum
		Fireplace		open type	open type	closed type	closed type	closed type
E	Test	No.	ē	Н	7	m	7	5

Table 5

Regeneration of silica gel (Batch C) by direct heating using wheat straw as a fuel and steel screen sack as a container

Amount	fue1	2/3 bale	1/2 bale	1/4 bale	1/4 bale	1/4 bale
Time required to evaporate a lb.	of water	30. min.	50.67 min.	46.67 min.	69.81 min.	75. min.
Water evaporated	%	33.3	1	30.0	23.28	18.3
Water ev	weight	1 1b	T.	12 ozs	8½ ozs	szo ^z _t 9
tained	%	1	1	ı	23.28	18.3
Water retained	weight	ı	A	ŀ	szo %8	6½ ozs
adsorbed ded)	%	33.3	27.27	25.00	32.87	36.61
Water adso (added)	weight	1 15	12 ozs	10 ozs	12 ozs	13 ozs
Final	of gel	2 lbs	1 1b 14 ozs	1 1b 12 ozs	1 1b 12 ozs	1 1b 12 ozs
Initial	of gel	3 lbs	2 1bs 12 ozs	2 1bs 8 ozs	2 1bs 4½ ozs	2 lbs 3½ ozs
	Fireplace	open type	open type	open type	open type	open type
Test	No.	н	2	т	7	S

Table 6

Regeneration of silica gel (Batch D) by direct heating using charcoal as fuel and steel screen sack as a container

Amount	fuel	2 lbs	2 lbs	1½1bs	1½1bs	1½1bs
Time required to evaporate a lb.	of water	37.04 min.	64. min.	38.27 min.	26.88 min.	33.33 min.
aporated	%	27.00	21.62	28.12	31.00	27.9
Water evaporated	weight	13 ozs	8 028	13½ ozs	15 ozs	12 ozs
tained	%	Ĭ	t	t	j	23.25
Water retained	weight	1	ı	Î	1	10 ozs
dded	%	27.0	29.73	27.0	28.12	34.8
Water a	weight	13 ozs	11 ozs	13 ozs	13½ozs	15 ozs
Final weight	of gel	2 1bs 3 ozs	2 lbs 6 ozs	2 lbs 2½lbs	2 1bs 1 oz	1 1b 15 ozs
Initial weight	of gel	3 1bs	2 1bs 14 ozs	3 1bs	3 1bs	2 lbs 11 ozs
	Fireplace	open type	open type	open type	closed type	closed type
Test	No.	1	7	n	7	. 5

The results in Tables 3 to 6 indicate that a 1b. of water from silica gel (30% water) can be evaporated in 17 to 75 minutes.

But the other important factor to be considered is the adsorbing capacity of silica gel after five successive regenerations.

Table 9 shows the results of the equilibrium moisture test of batches A, B, C, D, E and F indicating the adsorbing capacity of silica gel after five successive regenerations, in comparison to the original silica gel. The loss in adsorption capacity of silica gel dried on wheat straw, charcoal (indirect fire), wheat straw, charcoal (direct fire) is 2.56%, 4.06%, 7.53%, 4.30% respectively.

It was felt that this loss might be due to high temperature and/or smoke contamination. Table 11 shows the temperature readings of wheat straw and charcoal at different bed depths.

Table 7 shows the results of Batch E silica gel dried on an open fireplace using wheat straw as fuel. Table 8 shows the results of Batch F, silica gel dried on a closed fireplace using charcoal as fuel. In these experiments (E and F) the temperature was reduced from 1300° - 1600° F to 200° - 300° F by placing a sand bed below the container.

The results of Table 7 and 8 indicate that the time required to dry the sample of silica gel (Batches E and F) are not much different from Batches A, B, C and D. The equilibrium moisture test indicates that adsorption capacity is similar to the original sample of silica gel.

There is a change in the color of silica gel (blue with a black tinge) when compared to the original sample (dark blue). But it does not affect either the adsorption capacity or the identification of color change at different humidities.

Table 7

Regeneration of silica gel (Batch E) using wheat straw as fuel with a 12 gauge metal sheet underneath the container

Amount	fuel	1/2 bale	1/2 bale	1/3 bale	1/3 bale	1/4 bale
Time required to evaporate a lb.	of water	42.37 min.	49.51 min.	34.27 min.	32.73 min.	45.70 min.
Water evaporated	%	27.5	30.0	30.0	31.42	30.4
Water ev	weight	19 ozs	21 ozs	21 ozs	22 ozs	21 ozs
dded	89	29.4	30.0	30.0	30.0	30.4
Water added	weight	20 ozs	21 ozs	21 028	21 ozs	21 ozs
Final weight	of gel	3 1bs 1 oz	3 1bs 1 oz	3 1bs 1 oz	3 lbs	3 1bs
Initial weight	of gel	4 1bs 4 ozs	4 obs	4 1bs 6 ozs	4 1bs 6 ozs	4 lbs 5 ozs
	Container	Big pan	Big pan	Big pan	Big pan	Big pan
	Fireplace	open type	open type	open type	open type	open type
Test	No.	н	2	ю	4	5

Table 8

Regeneration of silica gel (Batch F) using charcoal as fuel with a $^{1}\!2"$ sand bed underneath the container

to Amount b. of	fuel	5 1bs	5 1bs	4 lbs	4 1bs	4 1bs
Time required to evaporate a lb.	of water	22.86 min.	42.86 min.	32.00 min.	33.15 min.	22.86 min.
Water evaporated	%	29.16	29.16	31.25	30.20	29.47
Water ev	weight	28 ozs	28 ozs	30 ozs	29 ozs	28 ozs
lsorbed	%	29.16	29.16	29.16	29.16	29.47
Water adsorbed (added)	weight	28 ozs	28 028	28 ozs	28 ozs	28 ozs
Final weight	of gel	4 1bs 4 ozs	4 1bs 4 ozs	4 1bs 2 ozs	4 1bs 3 ozs	4 1bs 3 ozs
Initial weight	of gel	6 lbs	6 1bs	6 1bs	6 1bs	5 lbs 15 ozs
	Container	small pan				
	Fireplace	closed type				
Test	No.	н	2	m	4	5

Equilibrium moisture content of all batches

at 80°F and 90% relative humidity (a week period)

Table 9

Initial wt. of all samples was 5 grams

	-	Net weight		Average	Average moisture	Decrease in	
No.	Sample	1	2	wt.	content	adsorption	
1	original	6.75	6.70	6.72	25.56	0	
2	A	6.60	6.40	6.50	23.00	2.56	
3	В	6.40	6.35	6.37	21.50	4.06	
4	c ₂	6.15	6.20	6.17	18.96	6.60	
5	C 5	6.10	6.10	6.10	18.03	7.53	
6	D ₂	6.45	6.45	6.45	22.48	3.08	
7	D 5	6.40	6.30	6.35	21.26	4.30	
8	E	6.70	6.70	6.70	25.37	0.19	
9	F	6.70	6.70	6.70	25.37	0.19	
			6 4				

 C_2 : Sample taken from batch C after 2nd regeneration

 C_5 : Sample taken from batch C after 5th regeneration

 D_2 : Sample taken from batch D after 2nd regeneration

 $[\]mathbf{D}_{\mathbf{5}}$: Sample taken from batch D after 5th regeneration

THE FOLLOWING PAGE IS BLURRY DUE TO DOUBLE PRINTING IN THE TEXT.

THIS IS AS RECEIVED FROM THE CUSTOMER.

Table 10

Comparison of particle size with adsorption capacity

		Geometric	Geometric		No. of	
		mean	mean	Surface	particles	
No.	Ç	particle	standard	area	per	Moisture
No.	Sample	size	deviation	sq cms/gram	gram	adsorbed
ì	A	1070.27	1.307	24.292	182.84	21.87
2	В	1188.97	1.513	41.000	1186.09	21.25
3	С	1112.75	1.368	35.574	621.38	18.03
4	D	1074.50	1.439	38.672	885.22	20.63
5	E E	917.82	1.435	40.002	972.99	25.37
6	- F	1047.65	1.379	46.203	1382.63	25.37
7	c: original	1708.49	1.417	40.850	1009.93	25.56
		(B4) B \$1 \$24 8 8				

The purpose was to show whether the particle size affects the adsorption capacity because in the process of drying, the silica gel particles become smaller compared to the original. The results indicate that the particle size within the limits of these tests does not have any effect on adsorption capacity.

Tables show the sieving data used for particle size analysis (Table 12) and the drying and adsorption rate of silica gel (Table 13).

Table 11

Temperature readings measured by the Pyrometer with a chromel Alumel Thermocouple

Wheat straw flame temp. = $1400^{\circ} - 1600^{\circ}$ F

Charcoal flame temp. = 1600° - 1800° F

When silica gel was wet the temperature of the whole mass was = 100° - 200° F. As soon as it gets dried the temperature readings are as follows:

Fuel: charcoal: silica gel (2" bed depth) temperature in a 18 gauge container:

Bottom layer = 600° - 800° F

Top layer = 220°F

By placing a 14 gauge metal sheet below the container:

Bottom layer = 400° - 600° F

Top layer = $200^{\circ} - 220^{\circ}$ F

By placing a $\frac{1}{2}$ " sand bed in between 14 gauge metal sheet and 18 gauge metal container:

Bottom layer = 200° - 300° F

Fuel: wheat straw: silica gel (3/4" bed depth) temperature after placing a 12 gauge metal sheet below the 18 gauge metal container

 $= 200^{\circ} - 300^{\circ} F$

Table 12

Data Sheet Used for Tabulation of Sieving Data and Calculation of Geometric Particle Size or Diameter by Weight

Tyler	Diameter	Weight in gms							
Screen No.	in micron	Original	A	В	С	D	Е	F	
3	6730	-	-	=	-	-	=	-	
4	4760	-		-	: - :	-	-	-	
6	3360	-	_	-	7 <u></u> 8	=	=	\$1 <u></u> 8*	
8	2380	10.5	0.5	0.5	0.5	0.5	_	0.3	
10	1680	45.8	11.8	11.0	10.5	8.0	1.8	7.0	
14	1190	42.0	32.5	41.3	36.0	34.0	10.3	32.0	
20	841	6.3	28.5	34.0	33.5	33.5	41.0	38.5	
28	595	0.5	17.8	11.5	17.0	13.8	30.0	21.0	
35	420	3 5	7.5	1.5	3.5	5.0	8.0	5.0	
48	297	1 1	1.2	=	0.5	0.5	0.5	0.5	
65	210	· -	0.3	_	0.3	-	_	_	
100	149	-	19 <u>—</u> 19	-	==	=	-	<u>1400)</u> 70,67	
150	105	-	-	-	-	-	-	=	
200	74	-	_	1200	<u></u>	× =	=	4 <u>12.5</u> 274.1	
270	53	-	-	-	-	-	_	_	
PAN	-	-	_	_	-	_	_	_	

ĕ

Sample	Adsorption	Drying	Total Wt	Container	Sample Wt	Moisture by
No.	Time	Time	in gms	Wt in gms	in gms	Wt in gms
254		7201 No. 121 No.			200 (004.2)	
1	½ hr	Initial	27.905	14.065	13.840	
(Top		30 min	27.760		13.695	1.048
layer)		45 min	27.700		13.635	1,480
		1 hr	27.680		13.615	1.620
2	8 hrs	Initial	30.385	15.350	15.035	: -
-	o ma	30 min	29.323	13.330	13.973	7.064
		45 min	29.250		13.900	7.549
		1 hr			13.898	7.562
9		1 111	29.240		13.898	7.502
3	18 hrs	Initial	36.666	15.292	21.374	2 7
		30 min	33.968		18.676	12.623
		45 min	33.845		18.553	13.198
		1 hr	33.805		18.513	13.385
4	24 hrs	Initial	38.872	15.362	23.510	_
		30 min	35.545		20.183	14.151
		45 min	35.415		20.053	14.700
		1 hr	35.400		20.038	14.768
5	47 hrs	Initial	37.790	15.345	22.445	r <u>-</u>
	27	30 min	33.500		18.155	19.113
		45 min	33.465		18.120	19.269
		1 hr	33.460		18.115	19.292
6	47 hrs	Initial	39.327	15.500	23.8 27	
(Top		30 min	34.500		19.000	20.025
layer)		45 min	34.385		18.885	20.674
		1 hr	34.370		18.870	20.800
7	52 hrs	Initial	39.265	15.495	23.770	s z
,	JZ IIIS	30 min	34.753	13.493	19.259	18.982
		45 min	34.672		19.177	19.323
		1 hr	34.650		19.155	19.415
8	52 hrs	Initial	1883	470	1413	: = .:
		3½ hrs	1619		1149	18.680
		5½ hrs	1589		1119	20.800
		₩.				

CONCLUSION

From the results of the investigation it was concluded that the silica gel can be regenerated by a simple and inexpensive method. In this method a farmer can use either wheat straw or charcoal as a heat source to dry the silica gel after using it for grain drying. To avoid the smoke contamination and high temperatures he should follow the following procedure.

The container needs to be placed on a half-inch sand bed on a 14-gauge metal, instead of placing it on direct fire. The wall of the container should be high enough (24") to avoid smoke contamination.

The batch needs to be stirred every 10 minutes while drying the sample. The color changes observed while drying silica gel are pink to light blue; light blue to dark blue; dark blue indicates that it is completely dried.

We hope this regeneration method of silica gel for grain drying will help to solve the problems of grain drying in developing countries and the energy crisis in developed countries.

ACKNOWLEDGEMENTS

The writer expresses his gratitude to Dr. Harry B. Pfost, major professor, for his guidance and valuable suggestions during studies, research and preparation of this manuscript.

The author wishes to thank Dr. Do Sup Chung and Dr. T. O. Hodges, the members of his graduate committee, for their support and guidance in the completion of his work.

The author wishes to recognize The Food and Feed Grain Institute,
Kansas State University, for financial assistance provided for this
project.

The writer is also grateful to his parents and friends for their encouragement throughout the studies.

REFERENCES

- Anderson, J. A. and A. W. Alcock. 1954. Storage of Cereal Grains and their Products. American Association of Cereal Chemists. St. Paul, Minnesota.
- 2. Chemical Engineer's Handbook. 1950. McGraw Hill Book Company, Inc. New York.
- Chin, S. C. and W. H. Johnson. 1969. Kinetics of Moisture Movement in Hygroscopic Material. I. Theoretical Consideration of Drying Phenomena. Trans. of the ASAE, 12(1):109.
- 4. Dunstan, E. R., D. S. Chung and T. O. Hodges. 1974. Adsorption and Desorption Characteristics of Grain Sorghum. Trans. of the ASAE, 16(4):667-670.
- Debussy, J. H. 1968. Materials and Technology I. Air and Water Inorganic Chemical Nucleonics. Long Mans Green and Co. Ltd. London, Harlow, Amesterdam. First ed.
- Fleske, L. 1973. Application of Drying Agents for Small Scale On-farm Drying and Storage in Humid Regions of Developing Countries. M. S. Thesis, Kansas State University.
- 7. Hall, D. W. 1970. Handling and Storage of Food Grains in Tropical and Sub Tropical Areas. FAO. Paper No. 90:51,68,141.
- 8. Hauser, E. A. 1955. Silicic Science. D. Van Nostrand Company, Inc. Princeton, New Jersey.
- Irving, R. 1951. Regeneration of an Adsorptive Bed Containing Constituents with a High Vapor Pressure. Chemical Abstracts: 45.
- Johnson, A. I. and Auth, G. H. 1951. Fuels and Combustion Hand Book. McGraw-Hill Book Company, Inc. New York. First ed.
- Kirk, R. E. and D. F. Othmer. 1954. Encyclopedia of Chemical Technology. The Interscience Encyclopedia, Inc. New York. 12: 345-360.
- Laughlin, C. D. and H. M. Gwyn, Jr. 1948. Regenerating Adsorbents. Chemical Abstracts. 42:2410f.
- Markrides, A. C. 1959. Thermal Aging of Silica Gels. Journal of Physical Chemistry. 63:1789-1792.
- 14. Max, N. Y and Imre Zwiebel. 1971. Adsorption Technology. Alch E Symposium Series. No. 117, 67:103-104.
- 15. Mantell, C. L. 1945. Adsorption. McGraw Hill Book Company, Inc. New York and London. First ed. 167-178 and 270.

- 16. Muroya, Masaki, Konda and Scilchi. 1971. Effect of Impurities upon Physical Properties of Silica Gel by Heat Treatment. Chemical Abstracts: 3505.
- 17. Patrick, W. A., J. C. W. Frazer and R. I. Rush. 1927. A Study of the Structural Changes in Amorphous Materials. Silica Gel. Journal of Physical Chemistry. 31:1511-1520.
- 18. Pfost, H. B. and Swinehart, C. E. 1970. Feed Manufacturing Technology. American Feed Manufacturers Association.
- 19. Vail, J. G. 1952. Soluble Silicates Their Properties and Uses. Rein Hold Publishing Corporation. New York 36.

REGENERATION CAPACITY OF SILICA GEL FOR GRAIN DRYING

Ъу

Gangadhar Vemuganti Rao

B.V. Sc. A. P. Agricultural University, India, 1970

AN ABSTRACT OF A MASTER'S THESIS

Submitted in partial fulfillment of the

requirements for the degree

MASTER OF SCIENCE

Department of Grain Science and Industry

KANSAS STATE UNIVERSITY Manhattan, Kansas

We have investigated the regenerative properties of silica gel after successive use and regeneration steps on farm conditions. The research was aimed at developing a simple and inexpensive regeneration method for silica gel used in grain drying.

A series of experiments were conducted. Silica gel was wetted to 30% moisture level by adding water with a sprayer while mixing. It was then divided into four batches. Each batch was dried five times using different fireplaces (closed, open), containers (big pan, small pan, rotating drum) and fuels (wheat straw, charcoal). Following five successive regenerations as stated above, each batch was subjected to an equilibrium moisture test and compared with the original sample. A decrease in adsorption due to high temperatures and smoke was noted.

The damage to silica gel during drying was minimized by using a thick (14 gauge metal sheet) with a ½" sand bed underneath the containers. Due to reduction in temperature and smoke contamination, the equilibrium moisture content of these samples were the same after five successive regeneration steps when compared to the original sample.