



Regular Article

## Optimization of the Carbon Dioxide Absorption (CDA) and Surface Erosion (SE) of Potassium Superoxide Based Respiratory Air Tablets Using the Taguchi Method

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### ABSTRACT

Potassium superoxide tablets can be used in respiratory air regeneration systems within confined spaces such as spacecraft, submarines, coal mines and individual and collective masks. These tablets react with moisture and carbon dioxide in air and release oxygen. In this study, The effect of five parameters; the pressing pressure (0.5, 2, 4 and 5 bar), humidity (10, 15, 20, 25 %), Catalyst additives ( $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ ,  $(\text{Cu}_2(\text{OH})_3\text{Cl}_2)_2$ ,  $\text{CuO}$ ,  $\text{TiO}_2$ ),  $\text{H}_2\text{O}$  Absorbent additives ( $\text{SiO}_2$ ,  $\text{LiCl}$ ,  $\text{CaO}$ ,  $\text{SiO}_2 \cdot \text{Al}_2\text{O}_3$ ) and  $\text{CO}_2$  Absorbent additives ( $\text{LiOH}$ ,  $\text{NaOH}$ ,  $\text{KOH}$ ,  $\text{Ca}(\text{OH})_2$ ) were investigated in four levels using the Taguchi method. The carbon dioxide absorption and Surface Erosion were selected as criteria for optimizing the performance of Potassium Superoxide tablets based on the analysis of variance and the optimal conditions of each were evaluated separately and simultaneously. The optimal conditions for the higher carbon dioxide absorption and smaller Surface Erosion include the Humidity of 15 %, pressing pressure of 4 bar,  $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$  as the Catalyst,  $\text{SiO}_2$  as the  $\text{H}_2\text{O}$  absorbent and  $\text{Ca}(\text{OH})_2$  as the  $\text{CO}_2$  absorbent. Experiments performed in the performance test show that the optimized tablets in this study show a 28 % and 79 % increase in the carbon dioxide absorption compared to commercial tablets and pure potassium superoxide respectively. The results showed that the catalysts with copper cation had the greatest effect on the performance of the tablets.

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### 1. Introduction

A closed environment is an environment in which human beings cannot be in contact

with a normal atmosphere for any reason. As a result of human respiration in a closed environment, oxygen is consumed and carbon

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dioxide and moisture are produced [1, 2]. Supplying oxygen in a closed environment including spacecrafts, submarines, coal mines and individual masks, is a essential requirement for human respiration [3, 4]. Physical (gas or liquid oxygen capsule) and chemical (chlorate candles, electrochemical, superoxide, etc.) methods are used to supply oxygen in closed environments. Chemical methods have a special advantage over physical methods of oxygen supply in terms of the weight, volume and respiratory efficiency of the system (three effective factors in choosing oxygen supply systems) [3, 5].

One of the indicators defined for oxygen-



In addition, the antibacterial properties of potassium superoxide prevent the growth of harmful microorganisms in closed environments [7]. Other advantages of the potassium superoxide system, which include very simple control, low cost, very high reliability, light and respiratory quotient corresponding to the human respiration rate, no need for cooling chemicals and air filters, can be used simultaneously for several people due to its cost-effectiveness with maximum efficiency [8-11].

According to the research by Mausteller et al., it was found that the breathing air recovery cartridge should have characteristics such as no extra oxygen production and no dust generation. They suggested the use of

generating systems in closed environment is the ability of the system to be a closed Circuit System, which means that there is no need for a normal atmosphere to produce oxygen or release gases such as carbon monoxide or water vapor. Of all the oxygen generating systems, the systems based on alkali metal (usually potassium) superoxides have the capability of being a closed Circuit System. The  $\text{O}_2$  and potassium hydroxide (KOH) are produced from the reaction of  $\text{KO}_2$  and water ( $\text{H}_2\text{O}$ ), typically in the form of water vapor carried in the air.  $\text{CO}_2$  is then absorbed mostly in carbonate and bicarbonate forming reactions with KOH as follows [4, 6]:

tablets and compact discs. These compressed products are less sensitive to the humidity in air but remain sensitive to the reaction with carbon dioxide, which prevents the overproduction of oxygen and the reaction rate remains almost uniform. For this reason, powder compaction forms (granules, slugs, tablets, blocks, etc.), which have easy transportation and proper packaging and achieve the desired respiratory quotient, are used [5, 12]. In addition, it has been shown that in pure  $\text{KO}_2$  compacted tablets, the production of sufficient oxygen begins only a few minutes after the respiration with the equipment, and to produce oxygen simultaneously using the cartridge, decomposition catalysts need to be added to the  $\text{KO}_2$  tablet formulation [13, 14]. The

additive can improve the performance of potassium superoxide tablets. The catalyst can increase the reaction rate of potassium superoxide with water vapor and carbon dioxide. Catalysts that include heavy metal compounds such as copper, cobalt, manganese and etc., by adding water absorber, cause an intermediate phase to be formed where the catalyst can initiate the decomposition of potassium superoxide and adsorb the metabolic carbon dioxide [15].

Porosity is essential to improve the solid-gas ( $\text{KO}_2\text{-CO}_2$  &  $\text{H}_2\text{O}$ ) reaction and improve the release of oxygen in the tablets. In high pressing pressures, the specific surface area and porosity of potassium superoxide pellets are reduced, the gas exchange channels inside the pellets are eliminated and the tablets lose their importance. If the pressing pressure is low, tablets of low strength and durability are obtained. For this reason, the pressure adjustment and porosity are required for the reactivity of potassium superoxide and gases [16, 17].

Designing experiments based on scientific principles is one of the optimization tools in science and engineering. Optimal experimental designs should provide the information needed to perform the analysis and achieve optimal conditions with the least number of experiments [18]. Since the 1980s,

the Taguchi method has been used as an optimization method in the process of engineering experiments. The Taguchi method is an essential tool for the experiments designed based on statistical methods using a set of orthogonal arrays [19]. Due to the many parameters affecting the performance of oxygen generating tablets such as the tablet composition, pressure and conditions of the tablet making environment [20, 21], the Taguchi-based experimental design will be used to optimize these factors to obtain the maximum performance.

In this research, the effect of the operating parameters (the pressing pressure and humidity of the tablet making environment) and composition of tablets (catalyst type, water absorber and carbon dioxide adsorbent) on the Carbon Dioxide Absorption (CDA) and Surface Erosion (SE) of the tablet was investigated. The results were analyzed using the statistical analysis and the optimal conditions of the above parameters were obtained.

## 2. Experimental section

### 2.1. Materials

Table 1 summarizes the list of chemicals used in this research along with the supplier, purity, place of using.

**Table 1**

Physicochemical characteristics of chemical generating tablet additives.

Materials	Supplier	Appearance	Purity (wt %)	Application
$\text{KO}_2$	Sigma-Aldrich	Yellow solid	99	Oxygen generator
$\text{LiOH}$	Merck	White solid	98	Carbon dioxide absorber
$\text{NaOH}$	Merck	White solid	98	Carbon dioxide absorber
$\text{KOH}$	Merck	White solid	99	Carbon dioxide absorber
$\text{Ca(OH)}_2$	Sigma-Aldrich	White solid	98	Carbon dioxide absorber

$(\text{Cu}_2(\text{OH})_3\text{Cl})_2$	Commercial	Green crystal	45	Catalyst
$\text{TiO}_2$	Sigma-Aldrich	White solid	99	Catalyst
$\text{CuO}$	Sigma-Aldrich	Black-brown solid	97	Catalyst
$\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$	Sigma-Aldrich	Blue solid	99	Catalyst
$\text{SiO}_2$	Sigma-Aldrich	White solid	96	Humidity absorber
$\text{SiO}_2 \cdot \text{Al}_2\text{O}_3$	Sigma-Aldrich	White solid	98	Humidity absorber
$\text{LiCl}$	Sigma-Aldrich	White solid	99	Humidity absorber
$\text{CaO}$	Sigma-Aldrich	White solid	98	Humidity absorber

## 2.2. Equipment

### 2.2.1. Tablet making in gloveboxes

A Glovebox container was used to make the tablets. The equipment of the pneumatic press was placed inside the Glovebox and the humidity was controlled inside it. Due to the sensitivity of  $\text{KO}_2$  to the moisture in the air, the relative humidity inside the glovebox was

kept constant during tablet making, through dehumidifying by a rotating disc dryer. For the preparation of tablets from the potassium superoxide powder and additive, direct pressing processes are used because they need less equipment and no heat and solvent, have low cost and provide acceptable stability and uniformity of particle size [22].



(a)



(b)

**Figure 1.** (a) Glovebox machine and the hydraulic press system embedded in it to control the environmental conditions of making the tablet, (b) the matrix-punch of the press machine.

### 2.2.2. Carbon dioxide absorption (CDA) measurement chamber

to evaluate CDA of the manufactured tablets, a chamber with the dimensions of  $60 \times 60 \times 60 \text{ cm}^3$  is made of Plexiglass with sensors installed in embedded locations as shown in Figure 2. The humidifier is used to provide the moisture needed to react with potassium

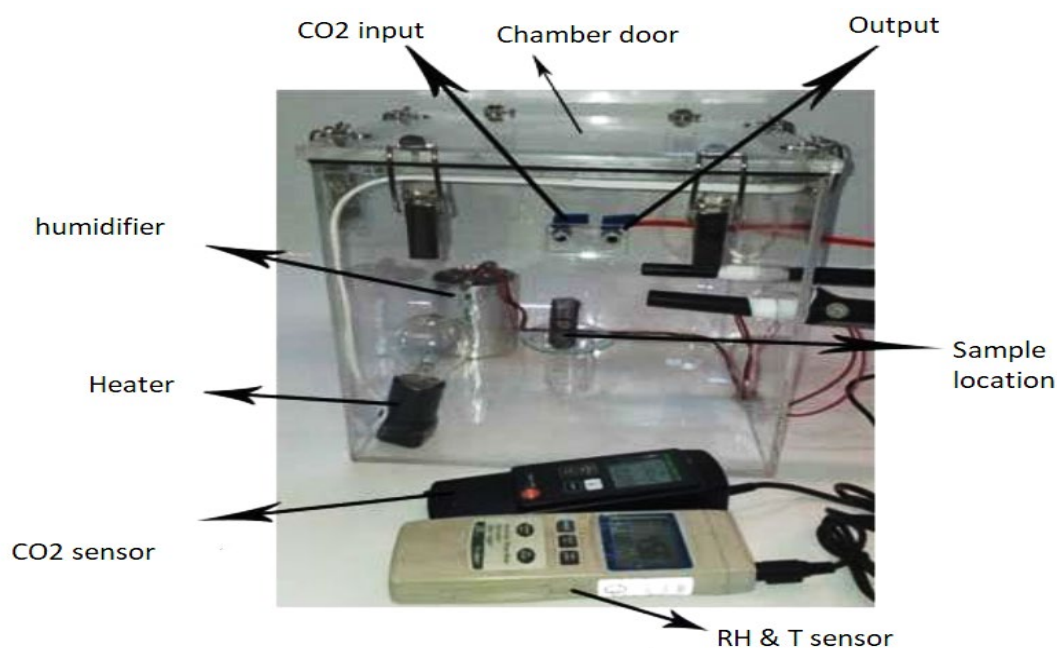
superoxide. Carbon dioxide with 99.99 % concentration is supplied by the capsule and the heater is also used to adjust the temperature. Carbon dioxide by the Testo 535 (Germany, 0 to 9999 ppm,  $\pm 1$  ppm), temperature by the LUTRON YK-90HT (Taiwan, 0 to 50  $^\circ\text{C}$ ,  $\pm 0.1$   $^\circ\text{C}$ ) thermometer, and relative humidity by the LUTRON YK-

90HT (Taiwan, 10 to 95 % ,  $\pm 0.1$  %) were measured at the inside of the chamber.

### 2.3. Friabilator

A device was used to determine the Surface Erosion of tablets as a measure of strength. The abrasive machine is made of a plexiglas chamber that rotates at a speed of 25 rpm and throws the pellets from a distance of 15 cm at a time (Figure 3). To measure the Surface

Erosion, the tablets are first weighed and rotated in the machine for four minutes (100 rounds), then the dust attached to the tablets is removed and tablets are weighed again. If the weight loss is between 0.5-1 %, the tablets are considered suitable for packaging. The optimal erosion percentage (0.5-1 %) can be achieved by adjusting the pressing pressure and humidity of the tablet [23].



**Figure 2.** Chamber to measure the absorption of carbon dioxide by potassium superoxide tablets under different conditions.



**Figure 3.** Friabilator device used to measure the SE (Surface Erosion) of tablets.

### 2.4. Taguchi experimental design

In this study, all tablets contained 88 wt % of

potassium superoxide and 12 wt % of additives. The additive consisted of 5 wt % of

the carbon dioxide adsorbent, 5 wt % of the moisture absorbent and 2 wt % of catalyst. The pressing pressure and humidity of the tablet making environment were considered as physical variables and different types of adsorbents were considered as chemical

variables on the tablet performance. The number of variables is considered as 5 and for each parameter there are 4 levels. Table 1 presents the different levels of variables affecting the performance of the oxygen generating tablet.

**Table 2**

Parameters and their levels for use in the experimental design.

Level parameter	1	2	3	4
Press (bar)	0.5	2	4	5
Tablet humidity (%)	10	15	20	25
Catalyst	(Cu <sub>2</sub> (OH) <sub>3</sub> Cl <sub>2</sub> ) <sub>2</sub>	CuO	TiO <sub>2</sub>	CuSO <sub>4</sub> .5H <sub>2</sub> O
H <sub>2</sub> O absorbent	SiO <sub>2</sub>	CaO	SiO <sub>2</sub> .Al <sub>2</sub> O <sub>3</sub>	LiCl
CO <sub>2</sub> absorbent	LiOH	Ca(OH) <sub>2</sub>	NaOH	KOH

The Taguchi method and Qualitek-4 software were used to design the experiment. The number of experiments suggested by the Qualitek-4 software is L<sub>16</sub> array. Table 3

presents the conditions of various experiments designed based on the Taguchi method to optimize the performance of oxygen generating tablets.

**Table 3**

Different conditions of experiments designed by the Taguchi method in L<sub>16</sub> array and the Surface Erosion, carbon dioxide absorption and OEC responses value.

Expr. No.	Press (bar)	Humidity (%)	Catalyst	H <sub>2</sub> O absorbent	CO <sub>2</sub> absorbent	SE (%)	CDA (%)	OEC
1	0.5	10	(Cu <sub>2</sub> (OH) <sub>3</sub> Cl <sub>2</sub> ) <sub>2</sub>	SiO <sub>2</sub>	LiOH	2.32	24.6	53.4
2	0.5	15	CuO	CaO	Ca(OH) <sub>2</sub>	4.65	26.4	35.2
3	0.5	20	TiO <sub>2</sub>	SiO <sub>2</sub> .Al <sub>2</sub> O <sub>3</sub>	NaOH	2.82	21.4	39.7
4	0.5	25	CuSO <sub>4</sub> .5H <sub>2</sub> O	LiCl	KOH	4.34	28.9	45.3
5	2	10	CuO	SiO <sub>2</sub> .Al <sub>2</sub> O <sub>3</sub>	KOH	1.63	22.3	54.1
6	2	15	(Cu <sub>2</sub> (OH) <sub>3</sub> Cl <sub>2</sub> ) <sub>2</sub>	LiCl	NaOH	1.68	28.1	69.6
7	2	20	CuSO <sub>4</sub> .5H <sub>2</sub> O	SiO <sub>2</sub>	Ca(OH) <sub>2</sub>	1.73	31.1	77.4
8	2	25	TiO <sub>2</sub>	CaO	LiOH	1.14	22.4	59.0
9	4	10	TiO <sub>2</sub>	LiCl	Ca(OH) <sub>2</sub>	0.57	21.8	63.2
10	4	15	CuSO <sub>4</sub> .5H <sub>2</sub> O	SiO <sub>2</sub> .Al <sub>2</sub> O <sub>3</sub>	LiOH	0.59	24.5	70.5
11	4	20	(Cu <sub>2</sub> (OH) <sub>3</sub> Cl <sub>2</sub> ) <sub>2</sub>	CaO	KOH	0.55	25.6	73.8

12	4	25	CuO	SiO <sub>2</sub>	NaOH	0.00	24.1	75.3
13	5	10	CuSO <sub>4</sub> .5H <sub>2</sub> O	CaO	NaOH	0.00	19.5	62.6
14	5	15	TiO <sub>2</sub>	SiO <sub>2</sub>	KOH	0.56	21.1	61.3
15	5	20	CuO	LiCl	LiOH	0.00	18.8	60.7
16	5	25	(Cu <sub>2</sub> (OH) <sub>3</sub> Cl <sub>2</sub> ) <sub>2</sub>	SiO <sub>2</sub> .Al <sub>2</sub> O <sub>3</sub>	Ca(OH) <sub>2</sub>	0.00	21.3	67.5

For optimization purposes, less TE and higher CDA were considered as responses separately. To simultaneously optimize TE and CDA, the Overall Evaluation Criteria (OEC) of Qualitek-4 software was used. The OEC is a parameter that is obtained from the merged responses with a certain weight as a response and varies in the range of 0 to 100 [24]. In this study, the weight of the TE and CDA responses were 50 % in OEC values. The calculated OEC values are shown in Table 3.

### 2.5. Procedure

It should be noted that the total chemicals used to produce each tablet is about 0.3 grams, of which 88 % is potassium superoxide and 5 wt % is the carbon dioxide

adsorbent, 5 % is the moisture absorbing and 2 % is catalyst. To make the tablets, the chemical materials were placed inside the matrix according to the experimental design conditions, and after adjusting the humidity and pressing pressure conditions, the tablet was made. The tablets are made with a diameter of 9.04 mm and a height of 4.54 mm. Once made, the tablets are placed in a glass container under the Globbox environment and then secured with paraffin glue. The number of tablets required for each sample is 10. Of these, there are 5 tablets for the CDA testing, and 5 tablets for the Surface Erosion test. Figure 4 shows 16 different types of tablets made according to the design of the Taguchi experiment, which are placed in sealed glass containers.



**Figure 4.** 16 Different types of pellets made according to the Taguchi experimental design.

To determine the Surface Erosion (SE) of tablets, first we weighed the 5 tablets and placed them inside the Friabilator device where the tablets rotated at 25 rpm for 4

minutes. After 4 minutes, the tablets are weighed and the TE is calculated by the following formula.

$$SE, \% = \frac{W_i - W_f}{W_i} \times 100$$

where  $W_i$  and  $W_f$  are the initial weight and the weight after the rotation of the tablet respectively.

To determine the carbon dioxide absorption (CDA), we put 5 tablets in the test chamber apparatus for 120 minute and measured the carbon dioxide concentration. The initial concentration of the carbon dioxide gas inside the functional testing machine is approximately 9000 ppm [25]. The CDA is calculated by the following formula.

$$CDA, \% = \frac{C_i - C_f}{C_i} \times 100 \quad (5)$$

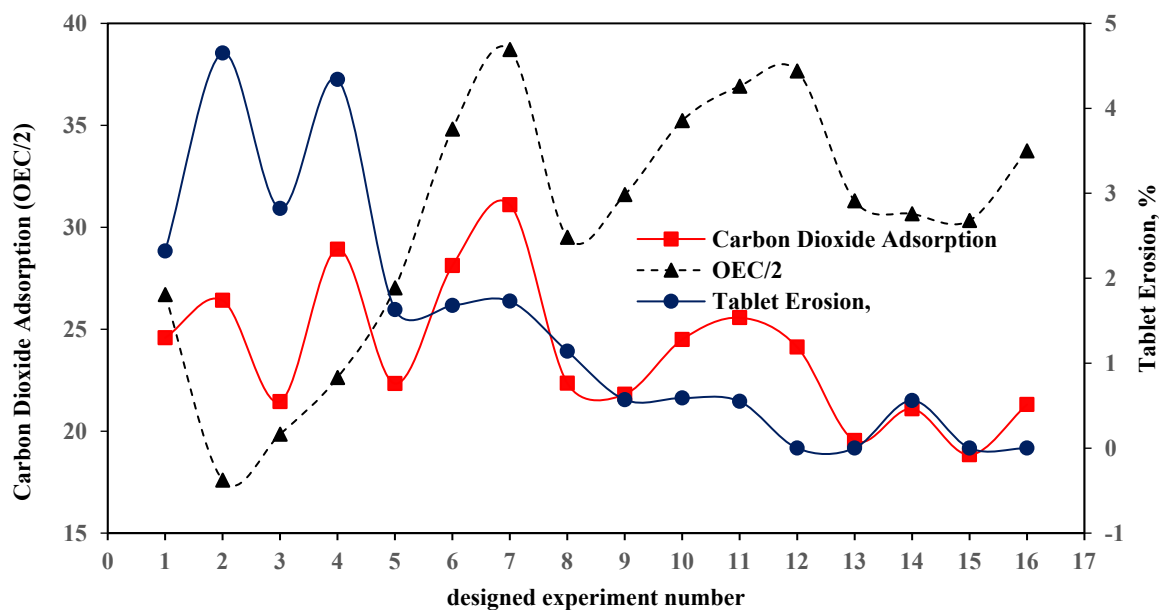
where  $C_i$  and  $C_f$  are the initial concentration of  $CO_2$  and the concentration of  $CO_2$  after 120 minutes in test chamber respectively.

### 3. Results and discussion

Many criteria such as the oxygen released yield, carbon dioxide absorption, chemical activity, stability and toxicity have been studied to evaluate the chemical composition

of respiratory air recovery systems. The CDA is one of the important parameters, which has been selected as a criterion in this study. On the other hand, due to the highly toxic and corrosive properties of potassium superoxide, it can be dangerous, if its "dust" enters the human body through the nasal passages and lungs.  $KO_2$  dust in the air is caused by the SE of the compressed forms of  $KO_2$ . Therefore, controlling the SE can be effective in reducing the generation of dust [4].

The values of the CDA and SE of different tablets were calculated according to the procedure, of which the results are presented in Table 3. Figure 5 shows the results of the CDA and SE of tablets versus the design experimental number. The Overall Evaluation Criteria (OEC) of Qualitek-4 software was used to investigate the effect of selected parameters on the CDA and SE simultaneously. The OEC values of tablets are also presented in Table 2 and the variations of OEC values according to the experiment number are presented in the Figure 5.



**Figure 5.** Variation of the CDA (Carbon Dioxide Adsorption), SE (Surface Erosion) and OEC/2 results of tablets versus the designed experiment number.



The results of Figure 5 shows that the trend of CDA has changed sinusoidally between 18.8 and 31.1 %, with the highest and lowest results being related to experiments 7 and 15 respectively. Also, a decreasing trend of SE is observed by increasing the number of experiment after number 4. In order to be able to draw OEC changes in the same vertical column, the OEC results divided by two are presented in the diagram. The results show that the trend of changes for OEC after Experiment 4 has increased significantly.

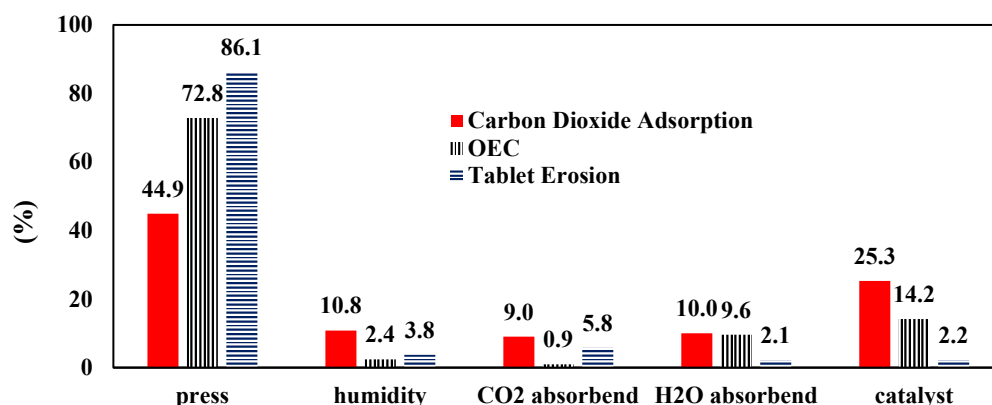
The CDA, SE and OEC results of the tablets were analysed using Qualitek-4 statistical software based on the analysis of the variance

(ANOVA) (Table 4) and the effect of each parameter was evaluated (Figure 6). The results showed that, as expected, the pressing pressure had the greatest effect on SE (86.1 %) and CDA (44.9 %), which correlated with previous findings [15]. The effect of other parameters on SE is very small. In the case of CDA, the effect of other parameters is considerable, and for example the catalyst has about 25.3 % effects. Regarding the effect of different parameters on the results of OEC, it is observed that the CO<sub>2</sub> adsorbent parameter has a very small effect and can be ignored, but the other parameters have significant effects on OEC.

**Table 4**

Results of the analysis of the variance (ANOVA) of CDA, SE and OEC parameters of tablets.

Parameter	DOF	Sum of Squars			Variance			Percent (%)		
		SE	CDA	OEC	SE	CDA	OEC	SE	CDA	OEC
Press	3	28.405	80.521	6157.006	9.468	26.84	2052.355	86.109	44.895	71.165
Humidity	3	1.239	19.352	667.631	0.413	6.45	222.543	3.756	10.79	7.716
Catalyst	3	0.735	45.368	773.975	0.245	15.122	257.991	2.228	25.296	8.945
H <sub>2</sub> O absorbent	3	0.703	17.93	288.802	0.234	5.976	96.267	2.132	9.997	3.338
CO <sub>2</sub> absorbent	3	1.904	16.177	764.265	0.634	5.392	254.755	5.772	9.02	8.833
Other/Error	0	-	-	-	-	-	-	-	-	-
Total	15	32.987	179.351	8651.742	-	-	-	100	100	100



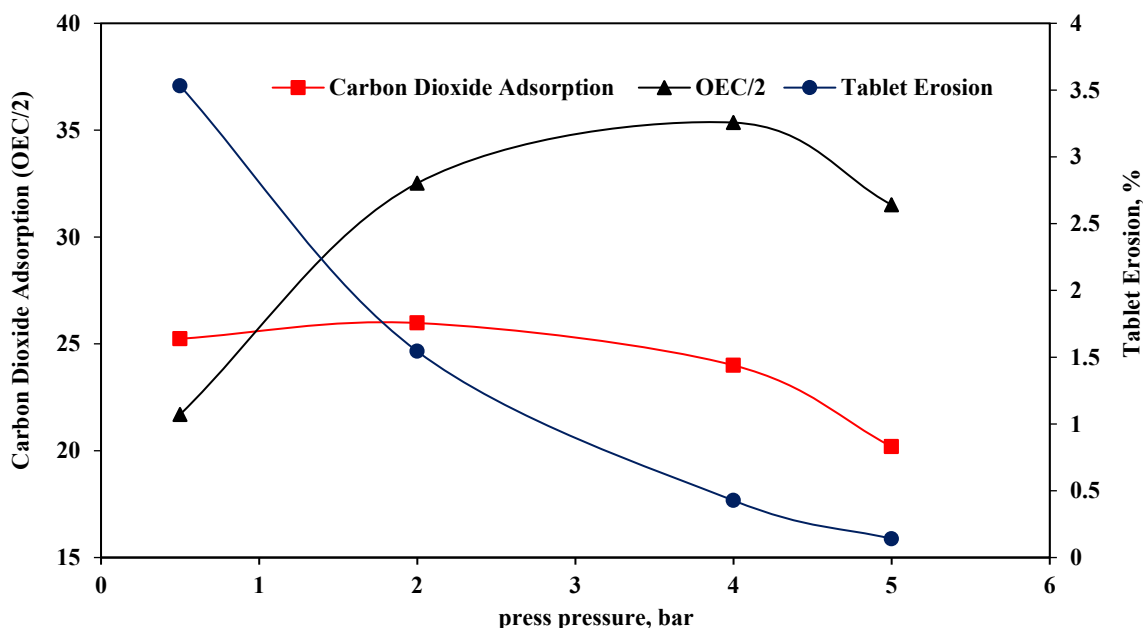
**Figure 6.** Percentage effect of different parameters on the CDA (Carbon Dioxide Adsorption), SE (Surface Erosion) and OEC (Overall Evaluation Criteria) of tablets based on the analysis of the variance in experiments designed by the Taguchi method.

In the following, the variability of each parameter in CDA, TE and OEC/2 tablets alone will be examined based on the analysis of the variance.

### 3.1. Effect of the pressing pressure

Figure 7 shows the effect of different pressing pressures on the CDA, SE and OEC/2 of tablets. The results show that by increasing the pressing pressure in the range of 1 to 4

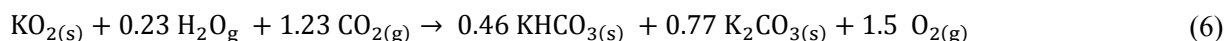
bar, no significant change is observed in CDA, but a further increase to 5 bar causes a decrease in CDA. On the contrary, the trend of changes in TE indicates that a noticeable decrease in SE is observed by increasing the pressing pressure. The trend of changes in OEC/2 is increasing to 4 bar and then decreasing due to the effect of merging the two parameters of CDA and TE in OEC.



**Figure 7.** Variation of the CDA (Carbon Dioxide Absorption), SE(Surface Erosion) and OEC/2 of tablets versus the pressing pressure of tablet making based on the analysis of variance.

The pressing pressure of the tablet has an adverse effect on the porosity of the tablets, and the porosity of the tablets also allows reactions between potassium superoxide and other reaction products with CO<sub>2</sub> and gaseous H<sub>2</sub>O according to the following reaction. More porosity provides more and larger

channels that increase the reaction of potassium superoxide with moisture and carbon dioxide [25, 26]. Therefore, increasing the pressing pressure can reduce the absorption of CO<sub>2</sub> due to the reduction of porosity:



A study by Wang reports that, increasing the pressing pressure causes the particles that make up the tablet to become more compact and engage with more pressure, which in turn

reduces the Surface Erosion of the tablet [27]. It is found that the pressing pressure has a double-edged effect on the performance of the tablets. On the one hand, increasing the

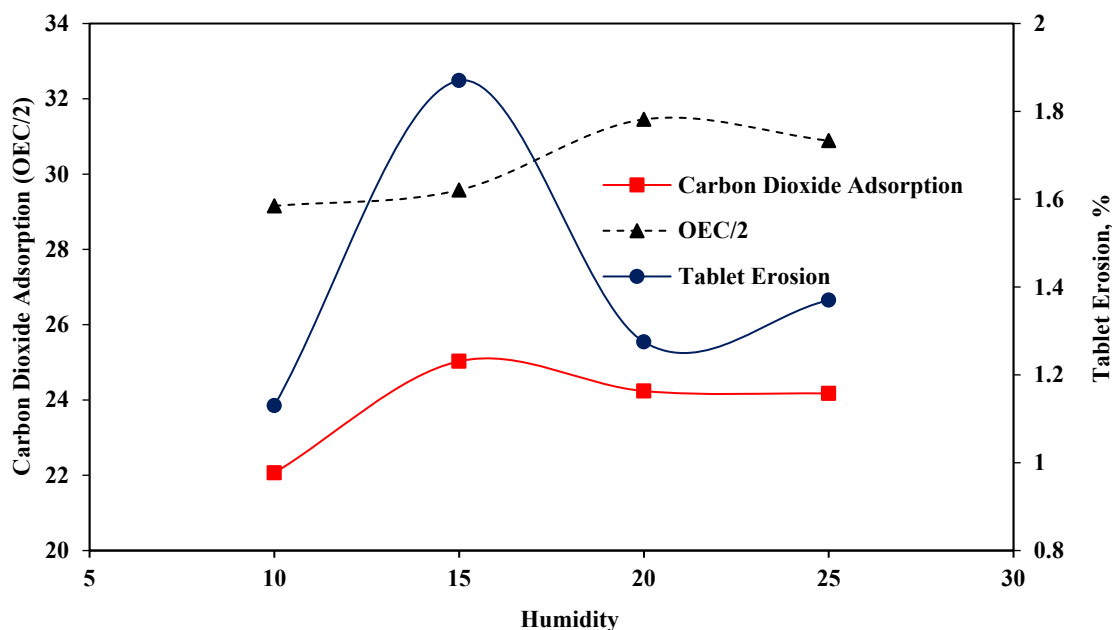
pressing pressure reduces the absorption of carbon dioxide and on the other hand increases the strength of the tablets [16, 25]. These contrasting effects of the pressing pressure make it important to optimize the presses applied to create tablets with optimal performance. For this reason, the pressing pressure of 4 bar is selected as the optimal because on the one hand it has less Surface Erosion and a suitable carbon dioxide absorption.

### 3.2. Effect of humidity

Due to the reactive nature of potassium superoxide when submitted to moisture, the initial suggestion was to make the tablet in an environment with the relative humidity of as

low as possible. In this study, a disc dryer was used that had the ability to dry up to 10 %. Based on this, the humidity of the tablet making environment is selected in the range of 10 to 40 %.

Figure 8 shows the effect of different amounts of tablet-making humidity on the CDA, SE and OEC/2 of tablets. The trend of changes shows a maximum value for CDA and STE in the amount of 15 % of moisture. The Surface Erosion of the tablets, in addition to the inherent properties of the participating materials and the pressing pressure, also depends on the amount of moisture in them. Very dry powders that contain a small amount of moisture usually give more abrasive tablets than powders that have more moisture.



**Figure 8.** Variation of the CDA (Carbon Dioxide Absorption), SE (Surface Erosion) and OEC/2 of tablets versus the humidity of the tablet making chamber based on the analysis of the variance.

The Surface Erosion of the tablets depends on the amount of moisture in them. Very dry tablets that contain a small amount of moisture usually get more eroded than moist tablets. In addition to, According to the Zao results, when the content of moisture in the tablet increases, the hardness of the tablet

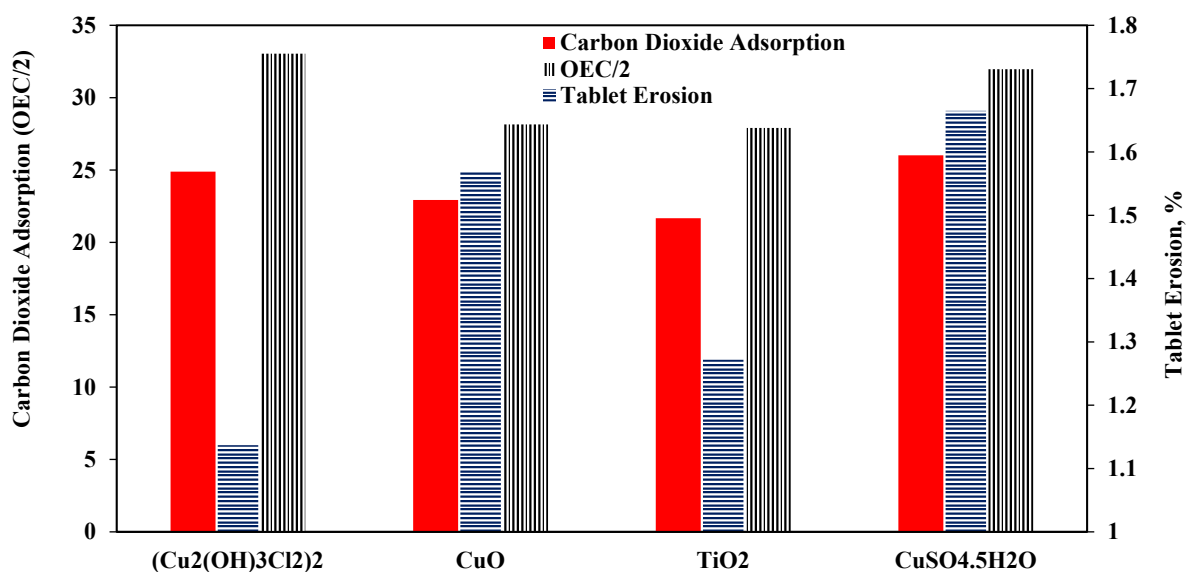
increases and the porosity of the tablet decreases, which decreases the exchange of CO<sub>2</sub> and H<sub>2</sub>O gases with potassium superoxide. Therefore, the optimum content of moisture in the tablet should be chosen. In general, as the humidity of the tablet is increased, the Surface Erosion of the tablets

increases and the amount of the carbon dioxide absorption decreases due to the reaction of potassium superoxide with moisture. However, at the humidity levels above 15 %, potassium superoxide reacts with moisture, and at less than that level, it causes more Surface Erosion of the tablets [22, 28].

### 3.3. Effect of catalyst

A study by Rainer et al. in 1987 found that

pure superoxide tablets could not produce enough oxygen for human respiration. Catalysts are mainly added to potassium superoxide formulations to speed up the reaction of potassium superoxide with carbon dioxide and moisture. Heavy metal oxides, mainly copper oxides, are used as catalysts [15, 29, 30]. Figure 9 shows the effect of the catalyst on the CDA, SE and OEC/2 of the tablets.



**Figure 9.** Variation of the CDA (Carbon Dioxide Adsorption), SE(Surface Erosion) and OEC/2 of tablets versus the Catalyst type of tablets based on the analysis of the variance.

The results of Figure 9 show that CuSO<sub>4</sub>.5H<sub>2</sub>O has the most and TiO<sub>2</sub> has the least effect on the carbon dioxide absorption of the tablets. As expected, the use of copper-based catalysts has a greater effect than titanium-based catalysts have on the adsorption of carbon dioxide whereas CuSO<sub>4</sub>.5H<sub>2</sub>O has the greatest effect on it. In addition; The results of Figure 9 show that the values obtained from the Surface Erosion of the tablets in the four types of catalysts are close to each other and practically with very small changes, and this shows that the catalyst does not affect the Surface Erosion of the tablets. One reason for this is that the Surface

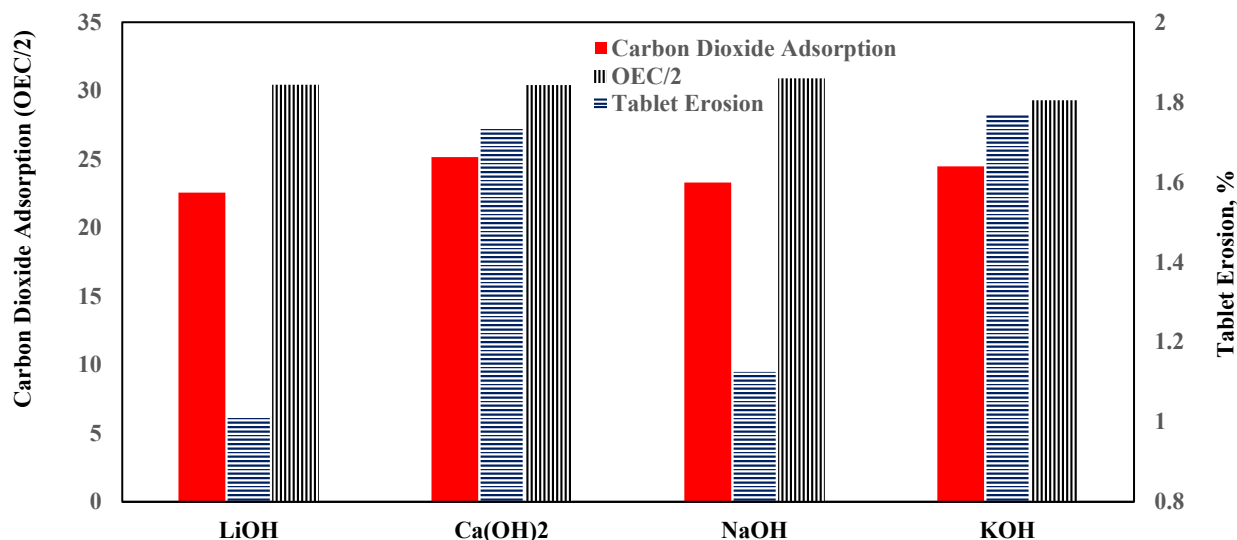
Erosion of the tablets is a physical phenomenon in which the tablet breaks into its components [31].

### 3.4. Effect of the carbon dioxide adsorbent

Figure 10 shows the effect of different types of carbon dioxide adsorbents on the CDA, SE and OEC/2 of the tablets. It is clear that calcium hydroxide has the most and lithium hydroxide the least effect. The results show that potassium, sodium and lithium hydroxides have almost the same yields, but calcium hydroxide has a higher yield capacity. This higher capacity of calcium hydroxide is due to the presence of two

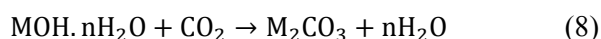
groups of hydroxides that have already had the same adsorbent group. A study by Dedman and Owen on the absorption of carbon dioxide by alkaline and alkaline

hydroxides found that calcium hydroxide and magnesium hydroxide had a higher carbon dioxide absorption capacity than alkaline hydroxides [32-34].



**Figure 10.** Variation of the CDA (Carbon Dioxide Adsorption), SE (Surface Erosion) and OEC/2 of tablets versus the carbon dioxide adsorbent type of tablets based on the analysis of the variance.

It can be said that carbon monoxide absorbers, in addition to absorbing carbon dioxide, are also moisture absorbers. In order to absorb carbon dioxide from the air, carbon dioxide adsorbents first form a moisture-soluble phase and then absorb carbon dioxide. The mechanism of these reactions is as follows:



Among the hydrated forms of lithium hydroxide, lithium monohydrate is essential for the adsorption of carbon dioxide due to the low reaction. However, lithium hydroxide decomposes lithium monohydrate due to its high heat output (14-22 kcal/mol) in the carbon dioxide absorption process. Therefore, lithium hydroxide has a lower capacity to absorb moisture and carbon dioxide.



In this study, lithium hydroxide has a lower carbon dioxide absorption capacity.

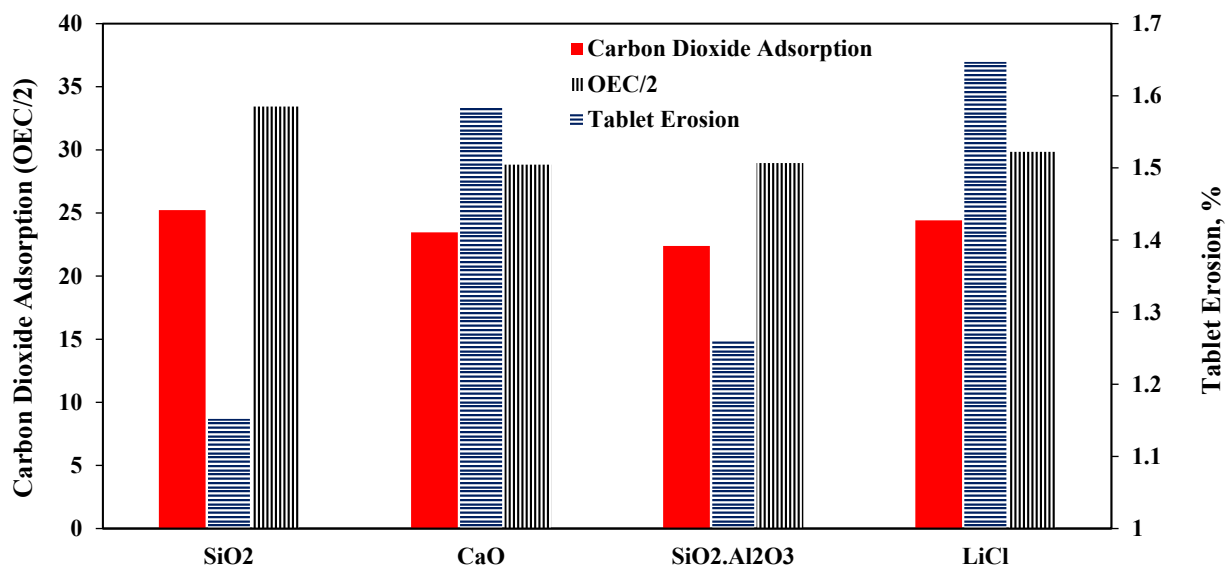
### 3.5. Effect of the H<sub>2</sub>O adsorbent

Figure 11 shows the effect of different types of H<sub>2</sub>O adsorbents on the CDA, SE and OEC/2 of the tablets. The results show that different species of water adsorbents had the same effect on the absorption of dioxide carbon, but the order of the effect of these adsorbents on the Surface Erosion of the tablet was LiCl > CaO > SiO<sub>2</sub>.Al<sub>2</sub>O<sub>3</sub> > SiO<sub>2</sub>.

The reason that lithium chloride and calcium oxide have more Surface Erosion than others is the opening properties of these two substances. Openers are materials or mixtures of materials that are added to the granulation of a tablet in order to separate and break down its compacted components when placed in a humid environment. While making tablets, these materials absorb the moisture in the atmosphere of making tablets

and during the packaging, transportation and performing functional, physical tests, etc. This causes the tablets to be wasted before reaching the application stage, because most of the tablets are crushed due to the opening property, and as a result, more Surface

Erosion of the tablets. As it is obvious, silica has the lowest rate of Surface Erosion and the highest amount of yield and OEC and is selected as the optimal moisture absorber [35].



**Figure 11.** Variation of the CDA (Carbon Dioxide Adsorption), SE (Surface Erosion) and OEC/2 of tablets versus the H<sub>2</sub>O adsorbent type of tablets based on the analysis of the variance.

#### 4. Taguchi's proposed conditions for optimal conditions

The values of the CDA, TE and OEC of tablets were analysed based on the Taguchi method using Qualitek-4 statistical software

for the preparation of tablets with the smaller SE and/or higher CDA. The results of optimal conditions for the preparation of potassium superoxide tablets with the smaller SE and/or higher CDA are presented in Table 5.

**Table 5**

Optimal conditions of SE, CDA and OEC with predicted and experimental results.

Parameter	Optimum Conditions			Results					
	SE	CDA	OEC	SE		CDA		OEC	
				Prid	Expr.	Prid	Expr.	Prid	Expr.
Press	5	2	2						
Humidity	10	15	15						
Catalyst	CuCl <sub>2</sub> .CuO	CuSO <sub>4</sub> .5H <sub>2</sub> O	CuSO <sub>4</sub> .5H <sub>2</sub> O	0.89	0.89	31.892	31.52	26.94	27.01
H <sub>2</sub> O absorbent	SiO <sub>2</sub>	SiO <sub>2</sub>	Caoline				6	9	2
CO <sub>2</sub> absorbent	LiOH	Ca(OH) <sub>2</sub>	KOH						

#### 4.1. Separate optimization of the CDA or TE of tablets

Although the pressing pressure has a high effect on the Surface Erosion of the prepared tablets, considering that the effect of other parameters is not less than 2 %, the effect of any of the parameters was not ignored. The optimal conditions predicted based on the statistical analysis for the preparation of tablets with the least abrasion include the humidity of 10 %, the press of 5 bar,  $(\text{Cu}_2(\text{OH})_3\text{Cl}_2)_2$  as catalyst, LiCl as the  $\text{H}_2\text{O}$  absorbent and  $\text{Ca}(\text{OH})_2$  as the  $\text{CO}_2$  absorbent with a SE of 1.36 %. The predicted conditions were not according to any of the designed experiments, so a tablet was made according to the predicted conditions for the minimal Surface Erosion. This tablet has 1.41 % of erosion, which was very close to the predicted amount.

Similarly, all parameters were considered to obtain the optimal values for the preparation of tablets with the maximum CDA. The optimal conditions predicted based on the statistical analysis include the humidity of 15 %, the press of 2 bar,  $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$  as catalyst,  $\text{SiO}_2$  as the  $\text{H}_2\text{O}$  absorbent and  $\text{Ca}(\text{OH})_2$  as the  $\text{CO}_2$  absorbent with a CDA of 31.86. The predicted conditions were not designed according to any of the experiments, so a tablet was made according to the predicted conditions for the maximum carbon dioxide absorption. The tablet has an absorption of 31.88 % of carbon dioxide, which was very close to the predicted amount.

#### 4.2. Simultaneous optimization of the TE and CDA of tablets

The OEC value was used to optimize simultaneously smaller SE and higher CDA of tablet preparation parameters by the Taguchi method. The optimal conditions that

the software proposes based on OEC values include the humidity of 15 %, the pressing pressure of 4 bar,  $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$  as Catalyst,  $\text{SiO}_2$  as the  $\text{H}_2\text{O}$  absorbent and  $\text{Ca}(\text{OH})_2$  as the  $\text{CO}_2$  absorbent. The OEC value of tablets under the optimum condition equals 85.6. These conditions were not consistent with any of the experiment, so a separate experiment was performed under the above conditions set at an OEC value of 85.4. The small difference between the OEC value suggested by the software and the OEC value of the experiment confirms the accuracy of the test.

#### 5. Conclusions

The oxygen supply by Potassium superoxide tablets is one of the chemical methods of supplying human respiratory oxygen. Pure potassium superoxide tablets are not applied to respiratory air recovery cartridges due to their incompatibility with Human Respiratory Factor. In order to use these tablets, the influence of various factors such as the pressing pressure, catalyst, moisture absorber and so on must be investigated. With the control of the tableting conditions, it is possible to quickly achieve a uniform diffusion of oxygen, proportional to the respiratory system coefficient with the human respiratory rate. The better performance of tablets can be achieved by the experiment design and data analysis. With the control of the tableting conditions, the rate of the uniform diffusion of oxygen, the proportion of the respiratory system coefficient with that of the human respiratory system, can be quickly achieved. In this study, four levels were used for each of the parameters of the pressing pressure, tableting humidity, catalyst, moisture absorber and carbon dioxide adsorbent using the Taguchi method. The optimal conditions proposed by the

software Qualitek-4 are the pressing pressure (4 bar), humidity (15 %), catalyst (CuSO<sub>4</sub>.5H<sub>2</sub>O), H<sub>2</sub>O adsorbent (SiO<sub>2</sub>) and carbon dioxide adsorbent calcium hydroxide. Experiments performed in the performance test show that the optimized tablets in this study show a 28 % and 79 % increases in the carbon dioxide absorption compared to commercial tablets and pure potassium superoxide respectively.

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### Nomenclature

CDA	Carbon Dioxide Absorption.
KO <sub>2</sub>	Potassium Superoxide.
OEC	Overall Evaluation Criteria.
SE	Surface Erosion.
TE	Tablet Erosion.
W	Wight of Tablets.

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