Mines Branch Information Circular IC 263

DESULPHURIZING GASES WITH MgO - RECOVERY OF CONCENTRATED SO_2 OR ELEMENTAL SULPHUR

by

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ABSTRACT

Finely divided $MgO-Fe_2O_3$ mixtures were supported on a fiber pad and used at 825°C to remove SO_2 from dilute gas streams. The reaction has an activation energy of about 24 kcal/mole and produces only $MgSO_4$. The efficiency of $MgSO_4$ formation varies with the partial pressure of oxygen in the gas, the ratio of the amount of MgO to fiber, and the linear flow rate of the gases.

$MgSO_4$ may be decomposed to MgO readily by decreasing the partial pressure of oxygen in the gas. When the partial pressure is moderately decreased with added CO, concentrated SO_2 is evolved. When an excess of CO is used, COS is evolved. The COS may be converted readily to sulphur by the addition of SO_2 in a second step. The activity of MgO does not decrease with repeated recycling.

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Direction des mines

Circulaire d'information IC 263

LA DÉSULFURATION DES GAS À L'AIDE DE MgO, AVEC RÉCUPÉRATION DU SO₂ CONCENTRÉ OU DU SOUFRE ÉLÉMENTAIRE

par

P. Marier[1] et T. R. Ingraham[2]

RÉSUMÉ

Pour retirer le SO_2 dilué dans des gaz, les auteurs utilisent des mélanges de MgO et de Fe_2O_3, en poudre très fine déposée sur un tampon en fibre porté à une température de 825°C. La réaction requiert une énergie d'activation d'environ 24 kcal par mole; elle aboutit à la formation de $MgSO_4$ exclusivement. Le rendement en $MgSO_4$ est fonction de la pression partielle de l'oxygène dans le gaz traité, du rapport entre les masses de MgO et du support en fibre, et de la vitesse d'écoulement du gaz traité.

Le $MgSO_4$ peut ensuite être facilement décomposé en MgO; il suffit pour cela de diminuer la pression partielle de l'oxygène dans le gaz. Si on abaisse modérément cette pression partielle tout en ajoutant du CO, il y a dégagement de SO_2 concentré. Si le CO est en excès, c'est du COS qui se dégage. On peut alors, dans une deuxième phase, convertir facilement le COS en soufre élémentaire, en ajoutant du SO_2. La répétition du cycle ne diminue en rien la réactivité du MgO.

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INTRODUCTION

For many years man has relied upon the natural ability of his environment to disperse or to dilute any waste materials he has released into it. The capacity of the environment is finite, and in some instances it has been exceeded. The result is that it has been necessary to move to abandon the assimilative capacity rationale and to institute regulations that will decrease the burden of waste assimilation on the environment.

Our increasing requirements for electrical energy are the principal cause of air pollution. In North America, only a small percentage of our electrical energy comes from water or nuclear power. Most of it is generated from coals or oils that contain significant amounts of sulphur that is converted during combustion to sulphur dioxide. "Sulphur dioxide accounts for most of the damage to materials and much of that to agriculture. SO_2 combines with oxygen and then with moisture to form sulphuric acid. Sometimes this takes place in the lungs of animals, sometimes on the leaves of plants, sometimes in droplets of rainwater and sometimes simply in the atmosphere where the acid persists as a fine, floating mist. . . the gas seems to do its worst in conjunction with particle pollutants that can carry SO_2 deep into the lungs and hold it there against sensitive tissue."[1]

The use of low-sulphur fuels has become the principal means of reducing sulphur dioxide emissions. "Desulphurization of coal and oil is expected to make only a limited contribution to alleviating sulphur oxides . . . The most effective and potentially least expensive method of controlling SO_2 is chemical removal from flue gas."[2] "A breakthrough in curbing sulphur dioxide would not only help the utilities, but also might rescue the steel industry from mounting public criticism. Oxides of sulphur are emitted in large quantities from steel's coking ovens."[1] The same criticism applies to metallurgical industries that use large-scale pyrometallurgical processes on sulphide ores.

Large expenditures have been made in a great variety of projects for removing sulphur dioxide from stack gases. The work can be generally classified into wet gas-washing methods and dry absorption methods. Of the latter, limestone injection has been the most successful to date. Our initial involvement with this method related to a request from Ontario Hydro to develop a method for evaluating the activities of various Ontario limestones.[3] Very substantial differences in activity were noted, and subsequent work[4] established the course of the chemical reactions involved in converting $CaCO_3$ to $CaSO_4$.

Aside from the fact that limestone injection is relatively inefficient, the $CaSO_4$ is essentially a throw-away

product that is part of a chain of converting an air pollution problem to a solids disposal problem.

Ideally, it would be desirable to regenerate the absorbent and to recover a saleable product. When a review was made[4] of the thermodynamic data for most potential oxide-absorbent systems, it was observed, from a computer comparison of equilibrium sulphur dioxide levels over the oxides, that many oxides could, in theory, lower the SO_2 content to acceptable levels. In practice, however, the reaction rates at working temperatures were too slow for most oxides to be of interest. Alternatively, if the temperature were raised to permit a rapid reaction rate, the equilibrium sulphur dioxide levels were unacceptably high. Two oxides were exceptions to these general observations: CaO and MgO. MgO appeared to have a substantial advantage, because it could be regenerated readily to the oxide under reducing conditions. Under similar conditions CaO is converted to the sulphide[5], which is useless for recycling.

In this report, experiments are described in which the chemistry of the reactions for the absorption of SO_2 by MgO is resolved, the regeneration of MgO from $MgSO_4$ is discussed, and the recovery of elemental sulphur from $MgSO_4$ is described. Results are reported on evaluating this method for the desulphurization of synthetic stack gases.

EXPERIMENTAL

Materials and Preparation

A variety of MgO source materials were used in the experiments. In the initial work, a brucite ore and an ore concentrate* prepared from material mined at Wakefield, Quebec, were used. When it became apparent that the chemical reactivity was proportional to the concentration of MgO present, the source of MgO was changed from natural materials to an analytical grade of $MgSO_4$. When iron oxide was required for additions to the magnesium oxide, it was prepared from analytical-grade hydrated ferric sulphate. Cylinder-grade SO_2, O_2 and CO were used without any additional drying or purification. The maximum surface exposure of the $MgO-Fe_2O_3$ was made by evaporating an aqueous solution of $MgSO_4$ and Fe_2SO_4 on Fiberfrax** and then reducing the sulphates with CO. The Fiberfrax was available as rolled battes about 0.5 inch thick and 2 feet wide. It was stable under all the reaction conditions tested and served as an excellent mechanical support for the MgO and also as a catalyst for the SO_2-O_2 reaction to SO_3. There was no appreciable spalling of the MgO from the Fiberfrax. When viewed under the microscope, it was evident that a small droplet of solution had dried around a fiber in such a way as to leave a small porous particle of MgO impaled by the fiber.

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*Concentrate donated by Mr. D.W. Evans, Vice-President, Aluminum Company of Canada, Montreal, Quebec.
**Trade name of fibrous alumina-silica insulating material manufactured by the Carborundum Company, Niagara Falls, New York.

Apparatus and Procedure

The small-scale experiments that were done to establish reaction rates and mechanisms were made with an American Instrument Co. THERMOGRAV balance. The work with brucite ore, ore concentrates and concentrate mixtures with Fe_2O_3 was done with the material suspended in small crucibles. When high-purity MgO, or MgO inoculated with Fe_2O_3, was used, the material was impaled on Fiberfrax fiber cylinders suspended in the balance chamber. $SO_2 - O_2$ mixtures in the ratio of 2:1 were used for sulphation.

The bench-scale experiments that were done to assess the possibility of removing SO_2 from commercial-type waste gases were made in a 2-inch, electrically-heated tube furnace in which strips of Fiberfrax-suspended MgO were placed. The progress of the reaction was assessed by periodically removing and weighing the strips. Sulphur dioxide concentrations as low as 1% were obtained by direct injection of SO_2 into the gas mixture.

The regeneration of MgO from $MgSO_4$ suspended on Fiberfrax was done in a stream of carbon monoxide. Sulphur was recovered from the resulting carbonyl sulphide by a second stage of treatment in which the carbonyl sulphide was reacted with sulphur dioxide over a variety of catalysts. Elemental sulphur was recovered from the gases by programmed cooling and trapping in a plug of pyrex glass wool.

RESULTS

The Formation of MgSO$_4$ from MgO and SO$_3$

In the initial experiments, small amounts of MgO contained in a crucible were exposed at a variety of temperatures to 2:1 mixtures of SO$_2$ and O$_2$ at various concentrations. The sulphation reaction was invariably slow and the MgO was only partly converted to sulphate. When the SO$_2$ and O$_2$ were equilibrated by passage through an asbestos-supported platinum catalyst, the rate of sulphation was rapid at temperatures in excess of 700°C. From these experiments it was evident that MgO is sulphated by SO$_3$ and not by SO$_2$ and O$_2$[6]. The Fe$_2$O$_3$ is a catalyst for converting SO$_2$ and O$_2$ to SO$_3$. The effect of various source materials and of the presence of Fe$_2$O$_3$ on the rate of sulphation is shown in Figure 1, where the percentage sulphation is plotted against time.

The group of curves at the bottom of Figure 1 indicates that MgO from a brucite concentrate may be sulphated more rapidly than MgO prepared by thermal decomposition from either reagent-grade MgCO$_3$ or a freshly precipitated Mg(OH)$_2$. The difference in rates is probably attributable to differences in particle size.

When 5% by weight of Fe$_2$O$_3$ was mechanically mixed with either the MgCO$_3$, the Mg(OH)$_2$ or the brucite concentrate, the sulphation rate was greatly increased. It is of interest to note that the initial stages of the sulphation reaction obey linear kinetics but that the reaction does not go to completion. Even

when the particle size is very fine (<280 mesh), the reaction appears to cease when about 80% of the material has been sulphated.

The foregoing experiments pointed to the necessity of exposing a very large surface area of the MgO if rapid reaction rates were to be achieved. For this purpose, a method was developed for impregnating a fiber support with many extremely fine particles of MgO.

The $MgO-Fe_2O_3$ -Fiberfrax mixture was prepared from an aqueous solution of the mixed sulphates. A hollow cylinder of the Fiberfrax was impregnated with the solution, dried, and then hung vertically in the TGA balance chamber so that the gas mix-tures flowing over it had full access to both sides. The sulphate mixture was activated for use by decomposition at 800°C in a stream of carbon monoxide. Some of the experimental results obtained with $MgO-Fe_2O_3$ mixtures supported on Fiberfrax are shown in Figure 2, in which initial reaction rates are plotted against the percentage of Fe_2O_3. The results show that, at 800°C, the rate of sulphation can be increased substantially by the presence of Fe_2O_3 in amounts up to about 7.5%. The reported rates are entirely relative, because there was no way of knowing, even approximately, the surface area of the MgO on which the reaction was taking place. For this reason the rates of sulphation were calculated from the initial slopes of the rate curves. This implies extrapolation to the initial conditions at which the surface areas would be comparable.

The rate curves for the formation of $MgSO_4$ from MgO supported on Fiberfrax are shown in Figure 3. The rate-time relationship is parabolic as shown by the fact that a linear relationship was obtained when the square of the weight gain in milligrams was plotted against the time in minutes. The curves are essentially linear for the initial 65% of the sulphation reaction. The parabolic form of the curves indicates that as the layer of sulphate thickens it becomes increasingly protective to the oxide. This behaviour reaffirms the necessity of working with very fine particles of MgO to ensure enhanced utilization of the MgO in a minimum of time.

The Arrhenius relationship of Figure 4 shows that the sulphation reaction occurs by a single process having an activation energy of 24.3 kcal/mole, at temperatures up to 850°C (10^3/T = 0.891). The rate of sulphation is linearly related to the rate at which the mixed gases, SO_2 and O_2, pass through the absorption chamber. For example, at 850°C the rate is increased by a factor of 4 when the flow is increased from 50 cc/min to 200 cc/min.

The effect of the partial pressure of SO_3 on the rate of sulphation is shown in Figure 5. In this group of experiments the sulphation reaction was studied only for the initial 10% and then the sample was regenerated for another run. Calculations of P_{SO_3} were made with the data of Evans and Wagman[7] and the correlation equation suggested by Ingraham and Kellogg[8].

The relationship between reaction rate and P_{SO_3} is linear, and the first-power relationship implies that the formation of the sulphate occurs by the addition of a single molecule of SO_3 to the MgO.

In experiments at 750 and 900°C, samples of MgO containing 7% Fe_2O_3 were prepared on a Fiberfrax support and cyclically sulphated and regenerated twenty-five times. Each time, about 80% of the MgO was converted to $MgSO_4$. The initial 60% of the reaction was linear. Comparison of the linear rates showed that there was no change in the activity of the MgO as the runs were repeated. During some of the experiments, carbon dioxide was added to the SO_2-O_2-SO_3 mixture. It served only to dilute the gases and thereby to decrease the reaction rate in the same manner as nitrogen would.

The Regeneration of MgO from MgSO₄

1) The effect of changing the partial pressure of CO

When $MgSO_4$ is heated in a neutral or oxidizing atmosphere, it is thermally stable up to a temperature of about 900°C[9]. When the atmosphere is slightly reducing, $MgSO_4$ decomposes readily. For example, in CO the reaction is:

$$MgSO_4 + CO \rightleftharpoons MgO + SO_2 + CO_2 \qquad [1]$$
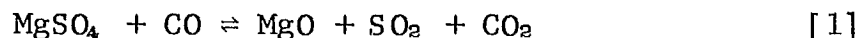
Any atmosphere in which there is a low partial pressure of oxygen will force Reaction 2 to the right and favour the decomposition of $MgSO_4$ by Reaction 3.

$$SO_3 \rightleftharpoons SO_2 + \tfrac{1}{2}O_2 \qquad\qquad [2]$$

$$MgSO_4 \rightleftharpoons MgO + SO_3 \qquad\qquad [3]$$

For example, a Fiberfrax-supported sample of MgO that was sulphated in the sulphur-containing waste gas from a bunsen-burner flame was regenerated at 800°C at a rate of about 10% per minute by merely changing the waste gas composition from oxidizing to reducing. The extreme sensitivity of the sulphation-desulphation reaction to the partial pressure of CO is illustrated in Figure 6, in which the initial rate of weight loss from a cylindrical Fiberfrax-supported $MgSO_4$ sample is plotted against the partial pressure of CO. As previously mentioned, initial rates of reaction were used because virtually the entire reaction curve was linear and because there was no way of knowing the surface area of $MgSO_4$ exposed for reaction. The same material was recycled to make the experiments comparable. In the experiments shown in Figure 6, the CO was diluted with N_2. Another series was done with CO_2 as the diluent. Both series of data were essentially identical. The relationship shown in Figure 6 may be linearized by plotting the logarithm of the partial pressure against the reciprocal of the reaction rate. The regression equation:

$$\text{rate} = 888/(4.688 - \ln P_{CO}) \qquad\qquad [4]$$

gives a fit within about 0.5% for most of the experimental points. It has been used to predict the rates shown in Table 1 for various partial pressures of CO at 800°C. The agreement between the predicted and observed values is good.

TABLE 1

Predicted Rates of Decomposition of $MgSO_4$ at 800°C

In Various Partial Pressures of CO and at a Flow-rate of 50 cc/min

P_{CO} (atm)	Rate (mg/min)	P_{CO} (atm)	Rate (mg/min)
2×10^{-4}	67	7.5×10^{-2}	121
1×10^{-3}	77	1×10^{-1}	127 (127)
2×10^{-3}	81	0.25	146 (145)
4×10^{-3}	86	0.51	165 (168)
1×10^{-2}	115	1.00	189 (189)

(The bracketed figures are the experimentally determined rates.)

The significant feature of Table 1 is the rapid increase in the reaction rate with very small changes in the amount of CO present. When only 5% CO is present, the rate is already over half of that obtained with 100% CO.

2) The effect of changing the flow-rate of CO

The rate of decomposition of $MgSO_4$ to MgO is sensitive to changes in the flow-rate of CO. For example, at 750°C, the rate of reduction was only doubled when the flow-rate of CO was quadrupled. At 850°C, the rate was only increased by 25% when the flow-rate of CO was quadrupled. Under identical conditions of flow, the apparent activation energy for decomposition was 15 ± 2 kcal/mole in the temperature range between 750 and 850°C. At 700°C the reaction is both slow and difficult to initiate.

3) <u>The stability of COS</u>

Sulphur dioxide is thermodynamically unstable in the presence of small amounts of carbon monoxide. The two gases react quickly to produce a third, carbonyl sulphide:

$$SO_2 + 3CO \rightleftharpoons COS + 2CO_2 \qquad [5]$$

Based on the data of Kubaschewski, Evans and Alcock[10], the free energy change for the reaction is given by the equation:

$$\Delta G = -71,230 + 42.89T \qquad [6]$$

The sign and value of the constants in this equation indicate that COS has its greatest stability at relatively low temperatures. Even at 1000°K, the free energy change is almost -30 kcal/mole. The significance of Equations 5 and 6 is that in the presence of more than one mole of CO per mole of $MgSO_4$, the reaction product changes from SO_2 to COS. The balanced equation for complete conversion of the sulphur in $MgSO_4$ to COS is:

$$MgSO_4 + 4CO \rightleftharpoons MgO + COS + 3CO_2 \qquad [7]$$
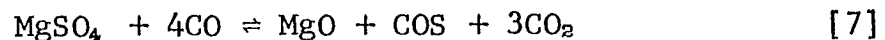
If, downstream from the regenerated MgO, the COS is treated separately with SO_2, elemental sulphur is produced rapidly. The reaction appears to be homogeneous and is best catalyzed by passing the gases over an absorbent having a large surface area. In the region of temperature between 600 and 800°C, the reaction is quantitative when it takes place on the surface of Fiberfrax that is coated with MgO, Fe_2O_3 or a mixture of both oxides. Fiberfrax alone is partly effective as a catalyst.

Contrary to our earlier expectations, the sulphur was not difficult to collect from the hot gas stream. When the gases were passed through a plug of pyrex glass wool, along which a temperature gradient had been established with heating tapes, the sulphur condensed as a bright yellow deposit in the wool. For the purposes of these experiments, the gain in weight of the pyrex wool plug was used to calculate the yield of sulphur from the reaction. The sulphur was invariably clean, of the typical yellow colour, and orthorhombic in structure.

Removal of SO_2 from Flowing Gas Mixtures

In the foregoing parts of this report, the mechanism of $MgSO_4$ formation from MgO and gases containing SO_2 was established. In this section a description will be given of experiments done to determine whether the technique could be extended to the desulphurization of flowing gases containing SO_2.

Either a vertical or a horizontal furnace containing a 2-inch-ID silica tube was used in the experiments. At 825°C the furnace had a zone of about 18 inches in length in which the temperature could be kept reasonably constant (±20°C). Within this zone, Fiberfrax pads of various lengths were used. When fully reacted, the pads could absorb about 25 g of SO_3 to form $MgSO_4$. Dry and moist gases containing various amounts of SO_2 were flowed past the pads. The moisture had no apparent effect on the rate of the reaction.

The results of a series of experiments in which gases containing 4% SO_2, 20% O_2 and 76% N_2 were passed over the absorption pads at 825°C are shown in Figure 7. The time of contact of the flowing gas with the pad was varied from 3 to 12 sec. When the contact time was 12 sec, there was 100% removal of SO_2 from the gas stream up to the time 20% of the MgO was converted to $MgSO_4$. With higher percentages of conversion, the percentage of SO_2 removal decreased sharply to 78% at 80% conversion. When the contact time was 3 seconds, no more than 60% of the SO_2 was removed from the gas stream irrespective of the amount of free MgO present.

The effect of the partial pressure of oxygen in the gas mixture is also shown in the figure by the dotted line. It indicates that when the amount of O_2 is decreased to 4%, the percentage of SO_2 removed from the gas stream does not exceed 50%, irrespective of the amount of unreacted MgO on the fibers. Additional experiments have shown that the percentage of SO_2 removal increases regularly from 4% O_2 to 16% O_2, but that the rate for 20% O_2 is about the same as for 16%.

It was of interest to observe that the effectiveness of removing 1% SO_2 from the mixed gas stream was almost the same as that for removing 4% SO_3. This implies that the rate-controlling step of the reaction does not directly involve sulphur dioxide.

In another group of experiments, the effect on reaction rate was studied for various weight ratios of MgO-Fe_2O_3 oxides to fiber. There was no gain in effectiveness when more than 50% mixed oxides was present.

One can gain some feeling for the effectiveness of the oxide mixture on the pads in removing SO_2 from the gas by examining the weight gain of three pads placed in sequence down the gas stream. The weight gain of each is shown in Figure 8. It is of interest to see that the first pad shows an immediate gain in weight. The second pad does not begin to pick up SO_2 until the thirty percent of the MgO on the first fiber pad has been converted to $MgSO_4$. The third pad only begins to pick up SO_2 after the first and second pads have been respectively converted to 65 and 25% of their capacity of $MgSO_4$.

For the three pads in sequence, 80% of the SO_2 is still being removed when 80% of the MgO has been converted to $MgSO_4$.

CONCLUSIONS

Magnesium oxide that contains a small amount of ferric oxide and is supported on a porous alumina-silica fiber base may be used at temperatures from 750 to 850°C to trap the sulphur dioxide from dilute gas streams. The reaction has an activation energy of about 24 kcal/mole. The SO_2 is bound as $MgSO_4$ which may be regenerated to MgO and SO_2 in a slightly

reducing atmosphere with an activation energy of 15 kcal/mole. In the presence of stoichiometric quantitities of CO, the SO_2 is converted to COS, from which it may be converted to elemental sulphur by reaction with SO_2.

By selecting the optimum conditions, 100% of the SO_2 can be stripped from a gas stream. The effectiveness of removal of SO_2 is a function of the amount of MgO, the temperature, the flow rate, and the partial pressure of oxygen. The effectiveness of removal is not a function of the percentage of SO_2 in dilute gas streams.

MgO may be regenerated readily from $MgSO_4$ by the use of CO. Depending on the ratio of $MgSO_4$ to CO, the product may be strong SO_2 or elemental sulphur.

The efficiency of SO_2 removal decreases with rapid flow-rates or low percentages of oxygen.

ACKNOWLEDGEMENTS

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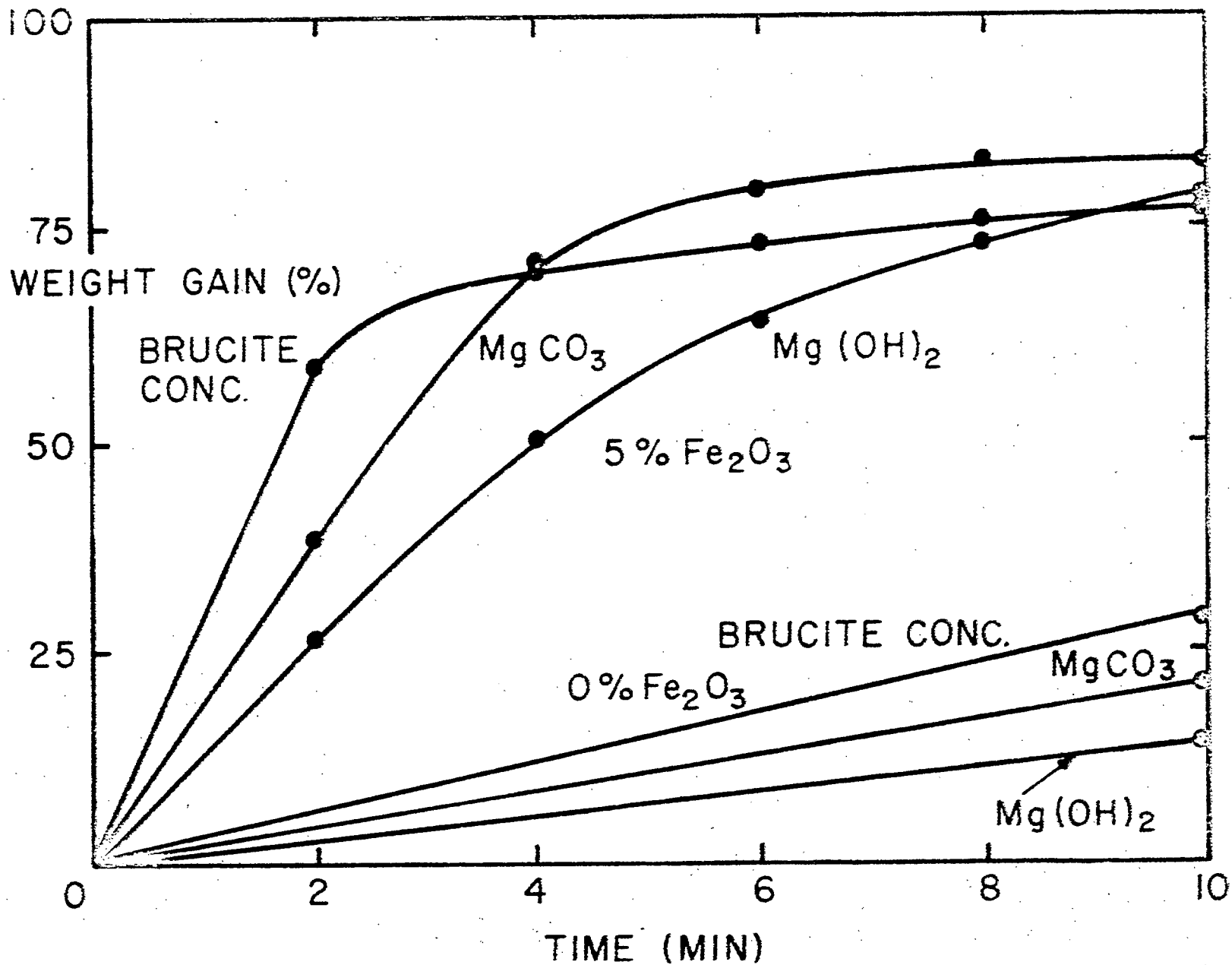
Figure 1.   Sulphation of MgO from different sources
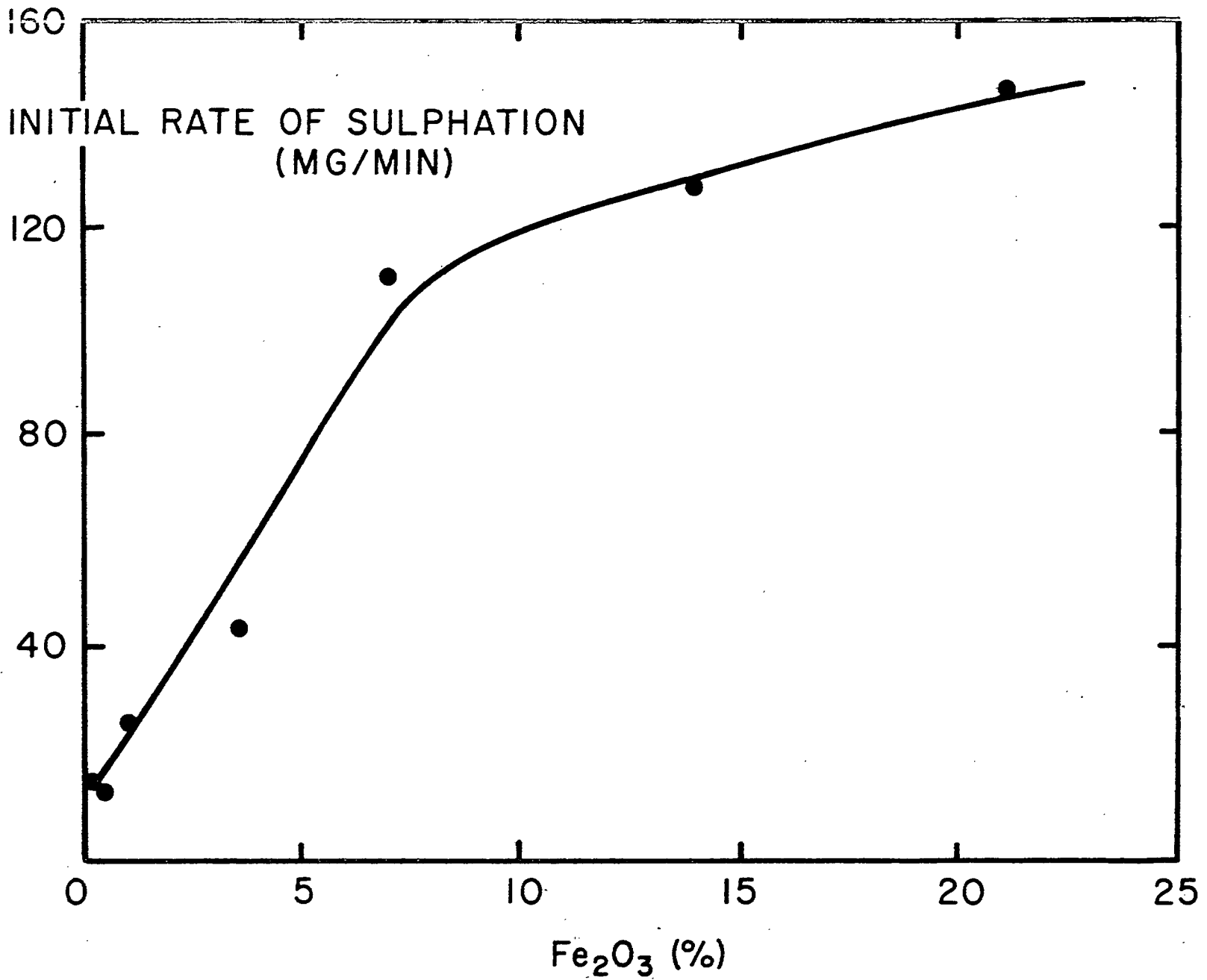(850°C, 2:1 SO_2:O_2 mixtures)

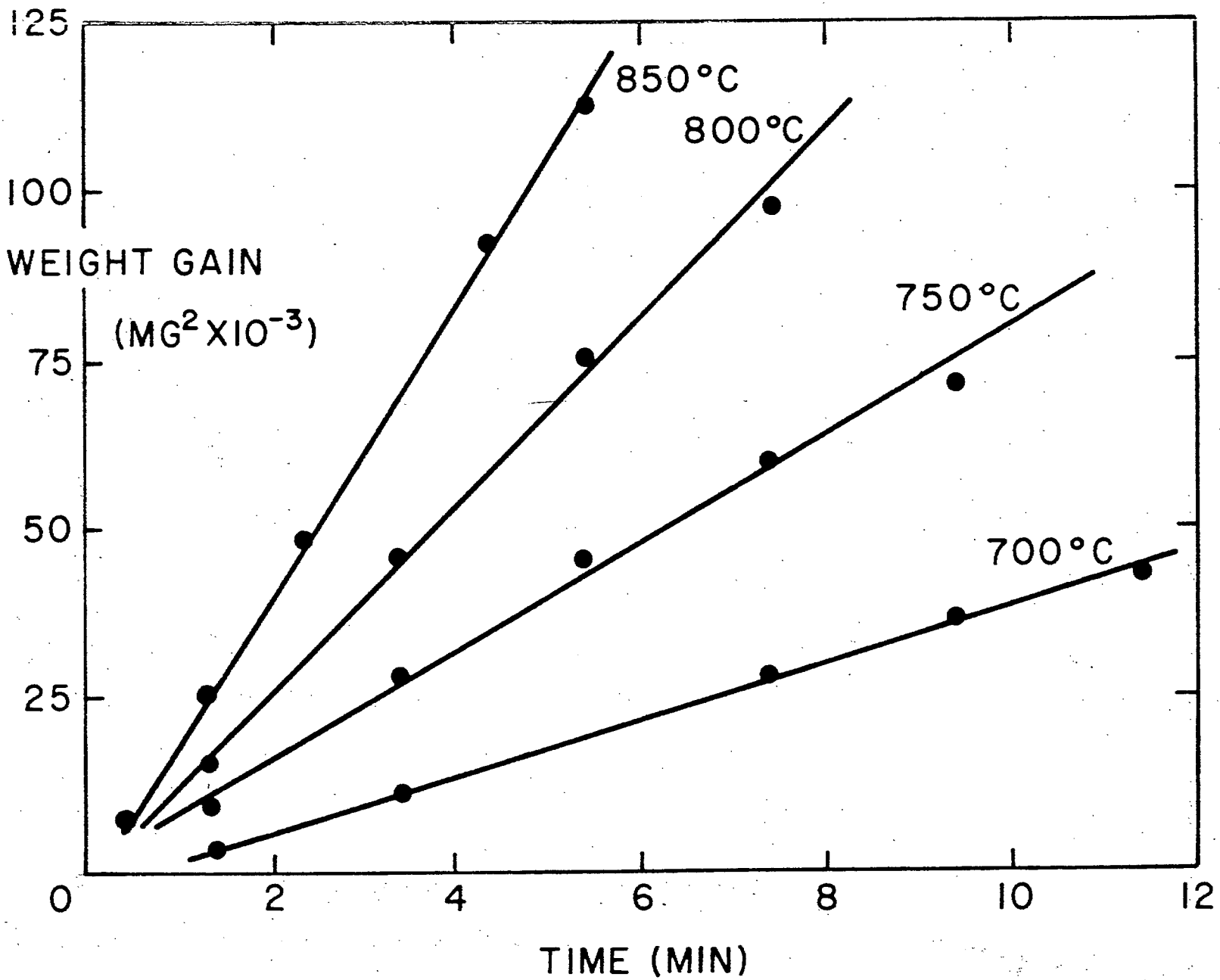Figure 2. Sulphation at 800°C of MgO impregnated pads containing different percentages of Fe_2O_3
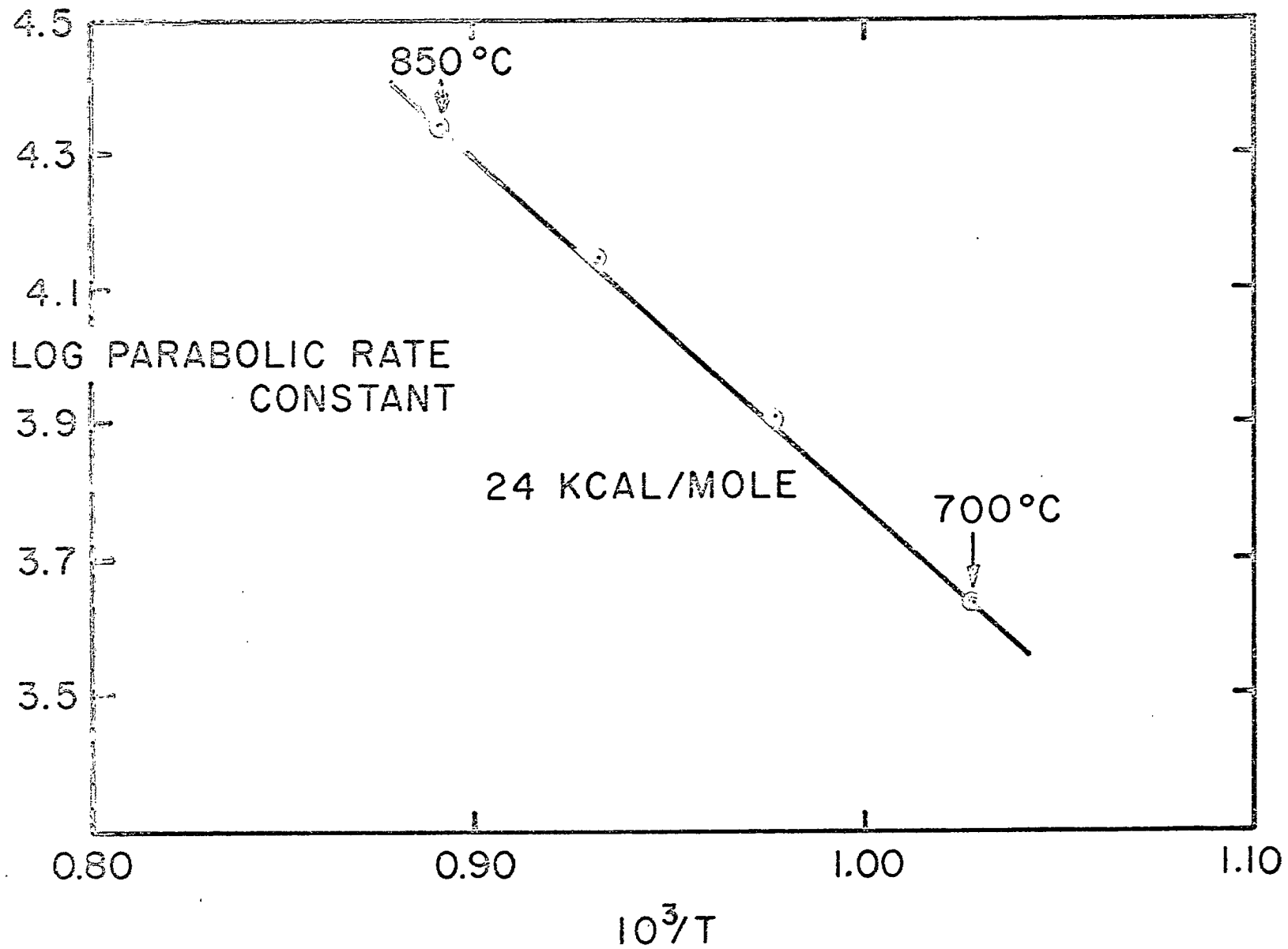
Figure 3. Parabolic sulphation kinetics

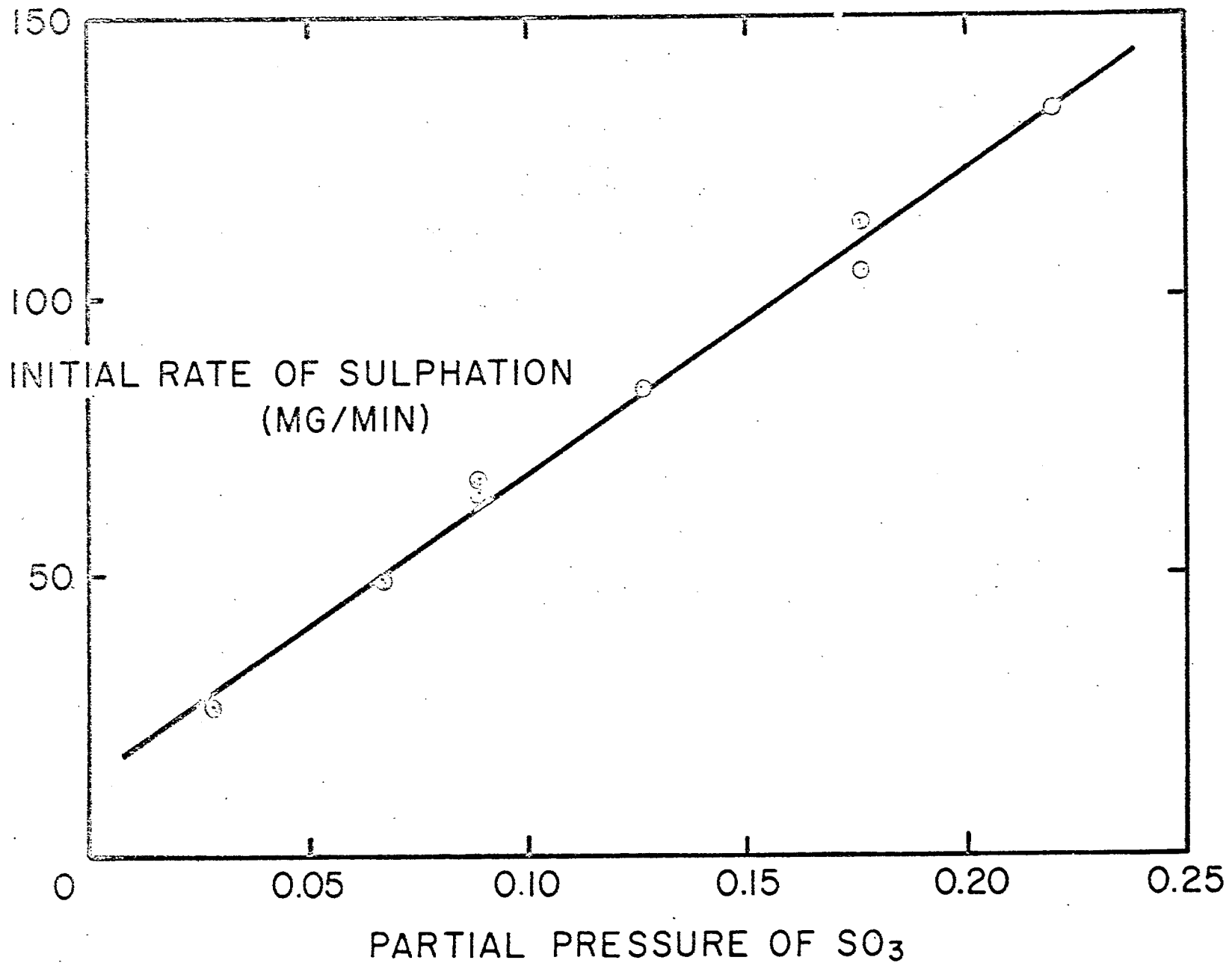Figure 4. Arrhenius plot for the sulphation of MgO

INITIAL RATE OF SULPHATION
(MG/MIN)

PARTIAL PRESSURE OF SO_3

Figure 5.   Initial rates of sulphation of MgO at 800°C in various
partial pressures of SO_3
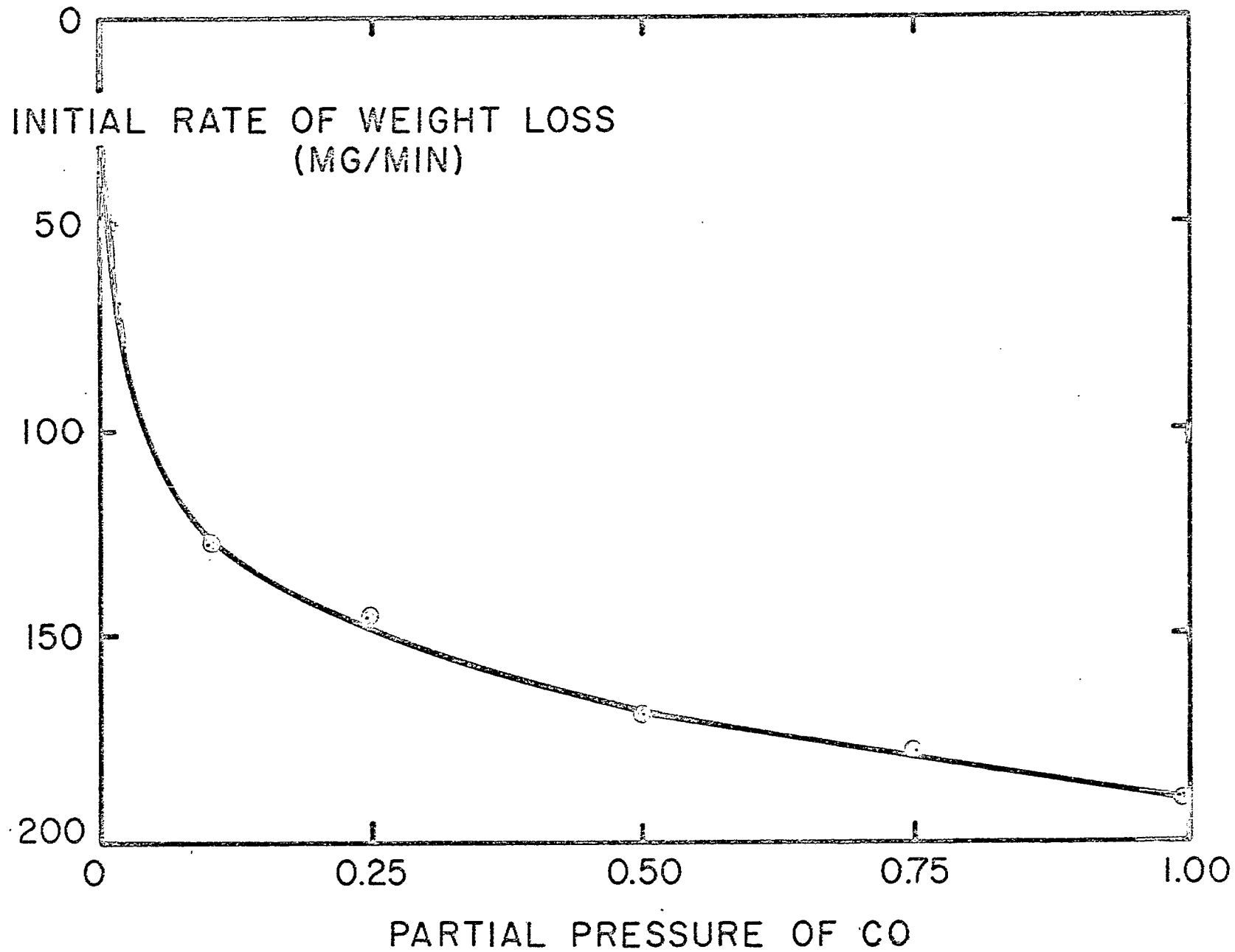
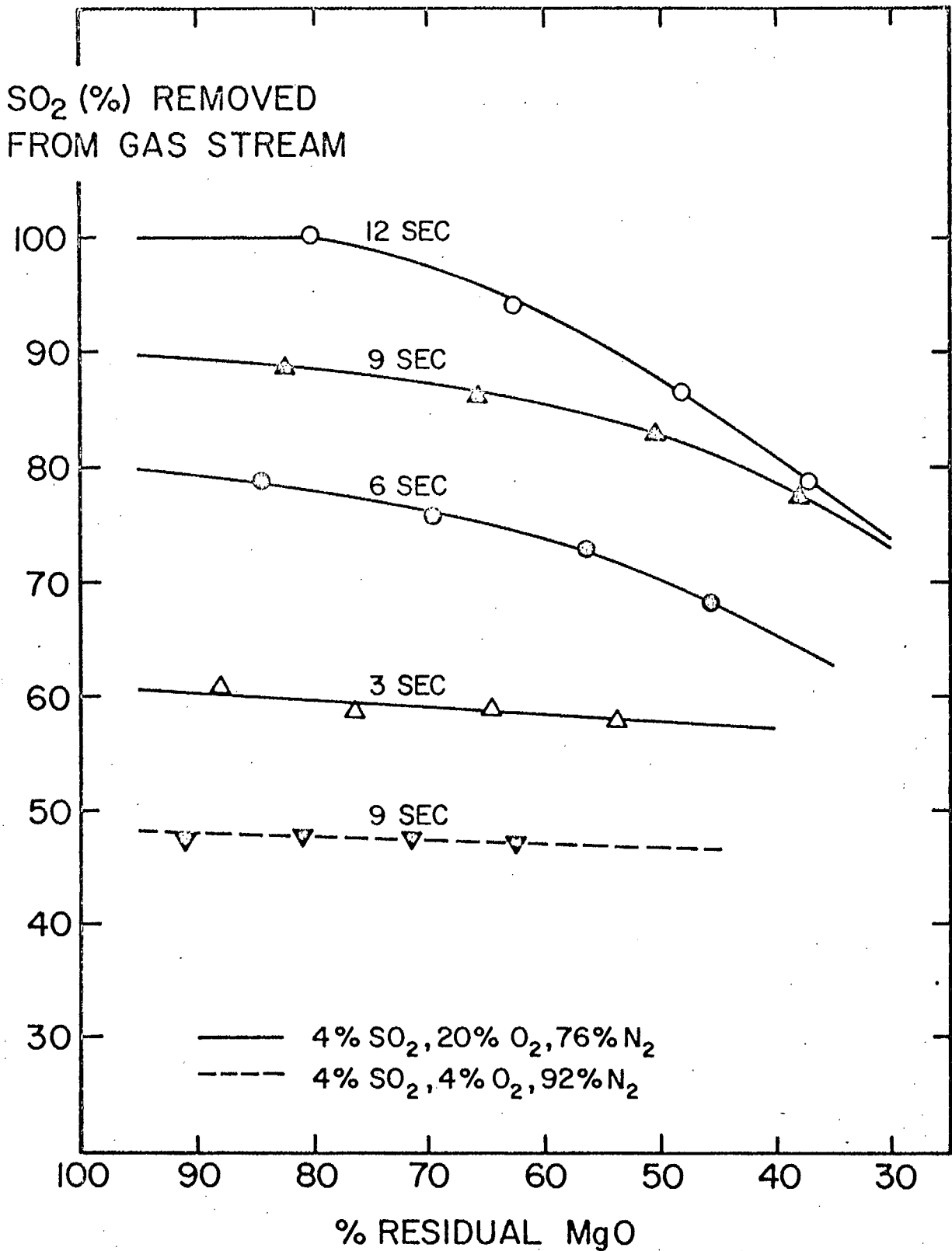Figure 6. The regeneration of MgO from MgSO₄ at 800°C in CO-N₂ mixtures

SO$_2$ (%) REMOVED
FROM GAS STREAM

Figure 7. Percentage of SO$_2$ removal as a function of contact
time and residual MgO

WEIGHT GAIN (%)

85% $MgSO_4$
92% REMOVAL OF
4800 CC SO_2 INJECTED
70% SO_2 REMOVAL
EFFICIENCY

PREVAILING CONDITIONS
AT THIS INSTANT

8 SECONDS CONTACT TIME
PER PAD

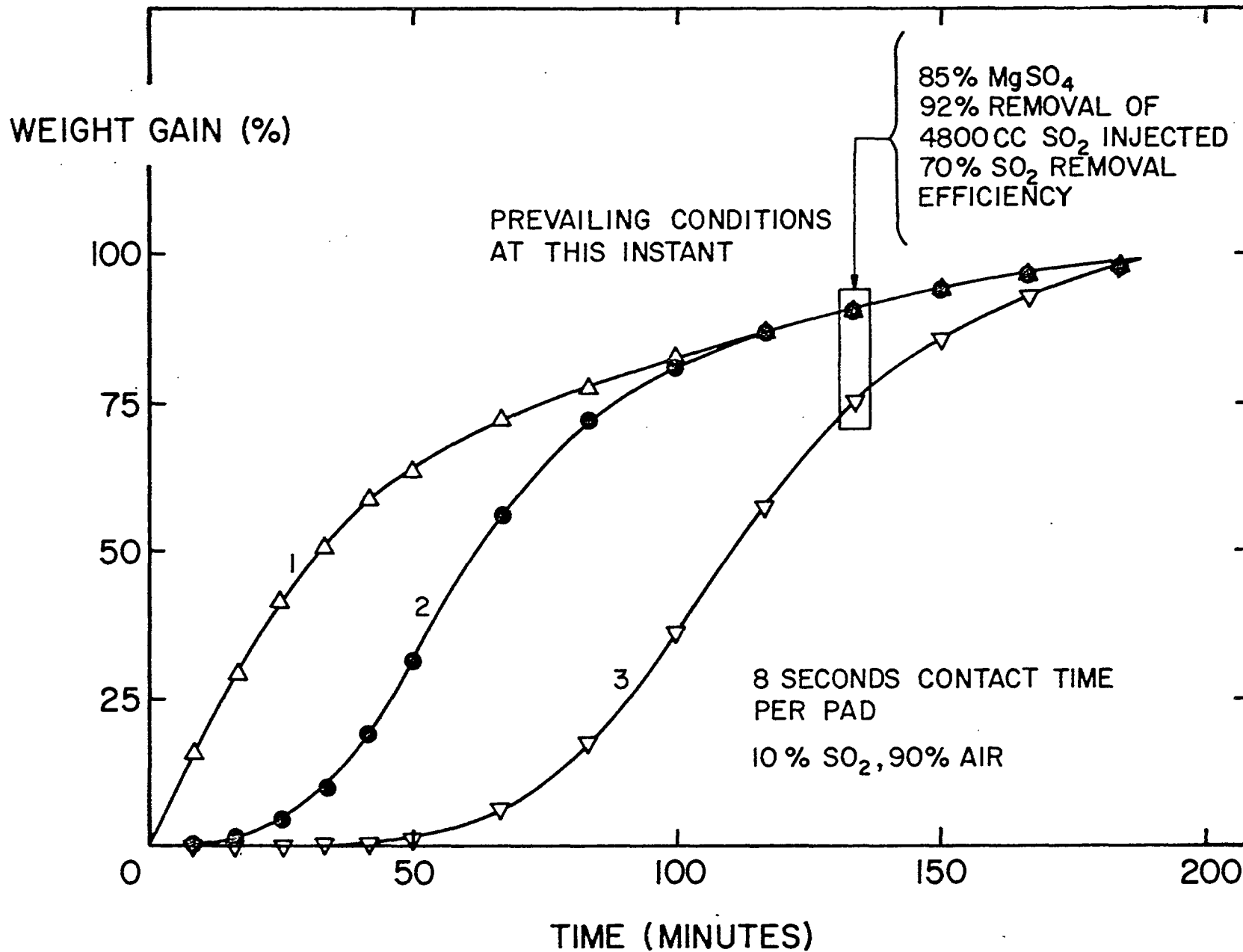10 % SO_2, 90% AIR

TIME (MINUTES)

Figure 8. The sequential sulphation at 825°C of three MgO
impregnated pads