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FACTORS CONTROLLING MEASUREMENT OF THE CRUSHING STRENGTH OF CALCIUM SULFATE GRANULES EVALUATED BY BALL MILL

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ABSTRACT

Calcium sulfate dihydrate granules were prepared using (10%) aqueous PVP solution. The crushing strength of the prepared granules was evaluated according to the method adopted by Mehta <u>et al</u> using a ball mill. Many controlling factors such as the proportion of the feed, granule size (1875, 1025, 558 μ), speed of rotation (low); I, moderate; II and high; III), type of balls (small porcelain, mixed porcelain and glass balls) and number of balls on the linearity of the equation were studied. The results obtained revealed that, the linearity of the equation was obvious as the proportion of the granules was increased. Granules of small size (1025 and 558 μ) gave under-estimation results. Upon increasing the time of milling, the linearity of the equation was obeyed. This finding was also achieved at low speed and on decreasing the number of balls. On the other hand, the linearity was obtained on decreasing the time of milling using glass and mixed balls as grinding media. In addition the time of milling was decreased at high speed.

INTRODUCTION

Several methods for measuring the breaking strength of granules have been reported[1-5] however most of them are time consuming and can not be applied for fine granules[1-3]. Recently, Mehta et al[6] introduced a simple, less time consuming method and applicable to all granule sizes using a ball mill and applying the following equation:

$$\ln(wa/w + db/da.wb/w) = -kt$$

where w is the weight of granules charged into the cylinder of the ball mill, wa is the weight of granules retained after milling, wb is the weight of granules passed after milling, da is the mean granule diameter, db is the mean granule diameter of the milled granules, wb/w= 1-wa/w and k is a measure of the crushing strength of the granules. Thus, db can be obtained by inserting consecutives values and plotting ln (wa / w + db /dw. wb/w) against time. Under-estimation of db gives curvature toward the abscissa, while overestimation gives curvature away from the abscissa. On the other hand, the real estimation of db gives a straight line. The value of "k" can be obtained by applying the most suitable value for db in Mehta et al equation.

El-Sabbagh et al[7] found that "k" value of kaolin granules can not be since db was difficult to be obtained.

The aim of this investigation is to study some factors affecting the linear relationship of Mehta et al equation such as:

a) The weight of granules charged into the ball mill.

b) The speed of rotation.

c) The types of balls.

d) The number of balls.

e) The granule size.

Calcium sulfate dihydrate was selected as an example of an inexpensive tablet diluent.

EXPERIMENTAL

Materials:

Calcium sulfate dihydrate[1] and PVP K_{25}[2].

Granulation:

Granulation was carried out using 20 ml of 10% w/v aqueous solution of PVP to each 100 gm of calcium sulfate to form a dough mass. The mass was granulated through a sieve No. 8 and dried at 50° for 24 hours.

Crushing strength of the granules:

The crushing strength of the prepared granules was measured according to the method reported by Mehta *et al*[6].

1- Fluka, A.G. Buchs, SG, Switzerland.

2- Supplied by El-Nile Chem. Co., Cairo, A.R.E.

A known weight of the granules of specific size fraction was charged in the cylinder of the ball mill (10 cm i.d.x 9.8 cm long; Labor Muszeripari Muvek, Esztergom, Hungary) using a certain number of balls. The ball mill was rotated at constant rate for certain time. The material was then sieved and the amount retained on the specified screen was weighed. The mean granule diameter of the milled ganules (db) was calculated as exmplified in table (1)* Accordingly the crushing strength of the granules (K) was obtained. The following factors which may affect the crushing strength of the granules were studied:

A) The proportions of the granules:

Three different proportions (15, 30 and 45 gm of granules having a size fraction of -2500 + 1250 μ (1250-2500 μ) were chosen. The material to voids ratio can be expressed as 1:3, 2:3 and 3:3 respectively, 25 small porcelain balls were introduced. The balls have 42 cc volume (diameter of each ball is equal to 1.5 cm), 95.76 gm weight and 2.3 gm/cc density. In addition the bulk volume of 25 small porcelain balls was found to be 115 cc. The speed of rotation adopted was speed II(105 rpm corresponding to 78% of the critical speed).

B- Granule size:

Three different granule sizes (-2500+1250 μ,-1250+800 μ and -800 + 315 μ) were evaluated at speed of rotation II using 25 small porcelain balls and 45 gm granules.

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* The most perfect correlation was illustrated, table (2).

C- Speed of Rotation:

Three different speeds of rotation , low(I), moderate (II and high III) were evaluated. The speed I was equal to 27 rpm(corresponded to 20% of the critical speed). The speed III was equal to 180 rpm(corresponded to 134% of the critical speed). 25 small porcelain balls and 45 gm granules having a size fraction -2500 + 1250 μ were charged into the mill.

D) Types of balls:

25 glass balls and mixed balls(consisting of 13 large porcelain balls and 25 small porcelain balls) were selected. The bulk volumes of 25 glass balls and the mixed balls were found to be 110 cc. and 310 c.c. respectively. The glass balls have 49 c.c. volume(diameter of each balls is equal to 1.6 c.c.), 118.4 gm weight and 2.4 gm/c.c. density. On the other hand,the large porcelain balls have 120 c.c. volume(diameter of each balls is equal to 2.64 cm), 267.3 gm weight and 2.3 gm/c.c. density . The operation was adopted at speed II using 45 gm granules of size fraction-2500+1250 μ

E) Number of balls:

25, 15 and 5 small porcelain balls were used at speed of rotation II using 45 gm granules of size fraction -2500 + 1250 μ. The bulk volumes of 15 and 5 balls were 70 and 25 respectively. The corresponded voids of charge were 45 and 16 c.c.

RESULTS AND DISCUSSION

A) Proportion of the granules:

Fig. 1(A,B & C) shows the effect of the proportion of calcium sulfate granules on the linearity of Mehta et al equation. It is clear that the linearity is more obvious as the proportion of the granules increased (r= -1 using 45 gm granules). However, the lowest value of k (the granules having the highest crushing strength) was obtained upon charging the highest proportion of the granules (45 gm) into the ball mill cylinder as shown in Fig. 6. This may be attributed to the largest amount of granules present which was subjected to less attribution and impaction[8-10]. Parrot[8-9] reported that, the amount of material to be milled should fill the void and just cover the grinding medium. It is evident that, 45 gm of calcium sulfate granules may be of suitable choice for further study.

B) Granule size:

As regards the effect of granule size on the linearity of the equation, it was found that the granules with mean particle diameter (MED) 1875 μ fulfilled the linearity of the equation at 2,4 and 6 minutes as shown in Fig. 10. On the other hand, granules with MPD 1025 and 558 μ gave underestimation results as in Fig. 2 A&C. Thus, upon increasing the time of milling to 10, 15 and 20 minutes, it was found that the linearity of the equation was obeyed where r= -0.99.

Fig. 2 B&C table 2) and the values of k can be calculated. The granules with large MPD gave the highest k value, while those with small MPD gave the lowest ones (Fig. 6).

C- Speed of Rotation:

It is clear from Fig. 3A that at low speed of rotation (speed I), the results were underestimation. Upon increasing the time of milling to 10, 15 and 20 minutes, the linearity of the equation was obtained as in Fig. 3B (r= -1). On the other hand, the results(Fig. 3C) were overestimation when higher speed (III) was adopted and the linearity was reached when the time was decreased to 1,2 and 3 minutes as Fig. 3D(r= -1). The calculated k values, indicated that, the crushing strength of clacium sulfate granules was higher at low speed than those at moderate and high speed (Fig. 6). This can be attributed to the fact that, at low speed of rotation, the balls simply roll over one another, so little crushing action was obtained. At slightly higher speed, they are projected to short distance across the mill, while at higher speed thay are thrown greater distances[10]. At further higher speed no size reduction occurred, because the balls are hold against the walls and revolves with the mill[8-11]. Thus speed II was selected to measure the crushing strength of the granules.

D- Types of balls:

It is demonstrated from Fig. 4 A&C that, both glass balls and mixed balls gave overestimation curves. Thus the time of milling was reduced to 1,2 and 3 minutes, where the linearity of the equation was fulfilled as shown in Fig. 4 B&D. (r= -1). The value of k in cass of glass balls (118.4 gm) was found to be less than that obtained with mixed balls (463.06 gm) as shown in Fig. 6. This indicated that mixed balls grind the granules more efficiently than glass balls. Thus heavy charge of balls produced a fine product in a short time[10], and gave good linearity of the equation. This may be taken as an advantage on calculating the k value of calcium sulfate granules.

E- Number of Balls:

Decreasing the number of balls was found to give underestimation curves as in Fig. 5 A&C. Thus , the time of operation was increased to 10, 15 and 20 minutes, where the linearity was fulfilled as in Fig. 5 B&D (r= -1). The lowest k value was obtained when the least number of balls was used (Fig. 6), where less grinding effect was observed. Parrot[8,9] reported that increasing the total weight of balls of specific size increases the efficiency of milling. This can be obtained by increasing the number of balls[9,10].

In this case, 25 small porcelain balls, weighing 95,79 gm were found to be the most suitable number for measuring the crushing strength of calcium sulfate granules.

In conclusion, for measuring the crushing strength of granules applying the method of Mehta et al the following factors must be considered: a-proportions of feed, b- the mean particle diameter of granules, c- the speed of rotation, d- the type of balls and e- the number of balls.

These factors were found to have a marked effect on crushing strength of calcium sulfate granules. Thus, granules having largest MPD must be selected to just cover the grinding medium. Balls of equal size or mixed balls of different size is preferred. Finally, the machine must be operated at the moderate speed of rotation.

Table 1: The most perfect correlation between time of milling and ln (wa/w + db/da. wb/w) of calcium sulfate granules.

Time min-	Proportion of feed (gm)	average granule size(U)	Speed of rotation	Type of balls	Number of balls	r	Mean granule diameter of the milled granules(U)
A	15	1875	II	P	25	-0.98	800
A	30	1875	II	P	25	-0.99	800
A	45	1875	II	P	25	-1	400
A	45	1875	II	P	25	-1	800
A	35	1025	II	P	25	-0.96	300
B	45	1025	II	P	25	-0.99	300
A	45	558	II	P	25	-0.96	100
B	45	558	II	P	25	-0.98	100
A	45	1875	I	P	25	-0.99	800
B	45	1875	I	P	25	-1	400
B	45	1875	I	P	25	-1	800
A	45	1875	III	P	25	-0.98	800
C	45	1875	III	P	25	-1	800
A	45	1875	II	P	15	-0.99	800
B	45	1875	II	P	15	-1	800
A	45	1875	II	P	5	-0.5	800
B	45	1875	II	P	5	-1	400
B	45	1875	II	P	5	-1	800
A	45	1875	II	G	25	-0.99	800
C	45	1875	II	G	25	-1	800
A	45	1875	II	M	25+13	-0.84	800
C	45	1875	II	M	25+13	-1	800

A: 2,4 and 6 minutes B: 10,15 and 20 minutes
C: 1,2 and 3 minutes P: small porcelain balls
G: Glass balls M: Mixed porcelain balls
I: 27 rpm III: 105 rpm III; 180 rpm.

Table 2: An example of sample calculation*

Time (minutes)	wa/w	wb/w	ln(wa/w + db/da. wl/w) if db is equal to 0U	400 U	800 U
2	0.59	0.41	-0.41	-0.39	-0.27
4	0.45	0.55	-0.80	-0.57	-0.38
6	0.2	0.80	-1.61	-1.00	-0.61
r**	-	-	-0.96	-0.97	-0.98***

* : Average of 3 expirements

** r : Correlation coefficient

*** : The most perfect correlation. In this case we considered 800 U is the mean particle diameter of the milled granules (db). Accordingly the crushing strength of the granules (K) was obtained.

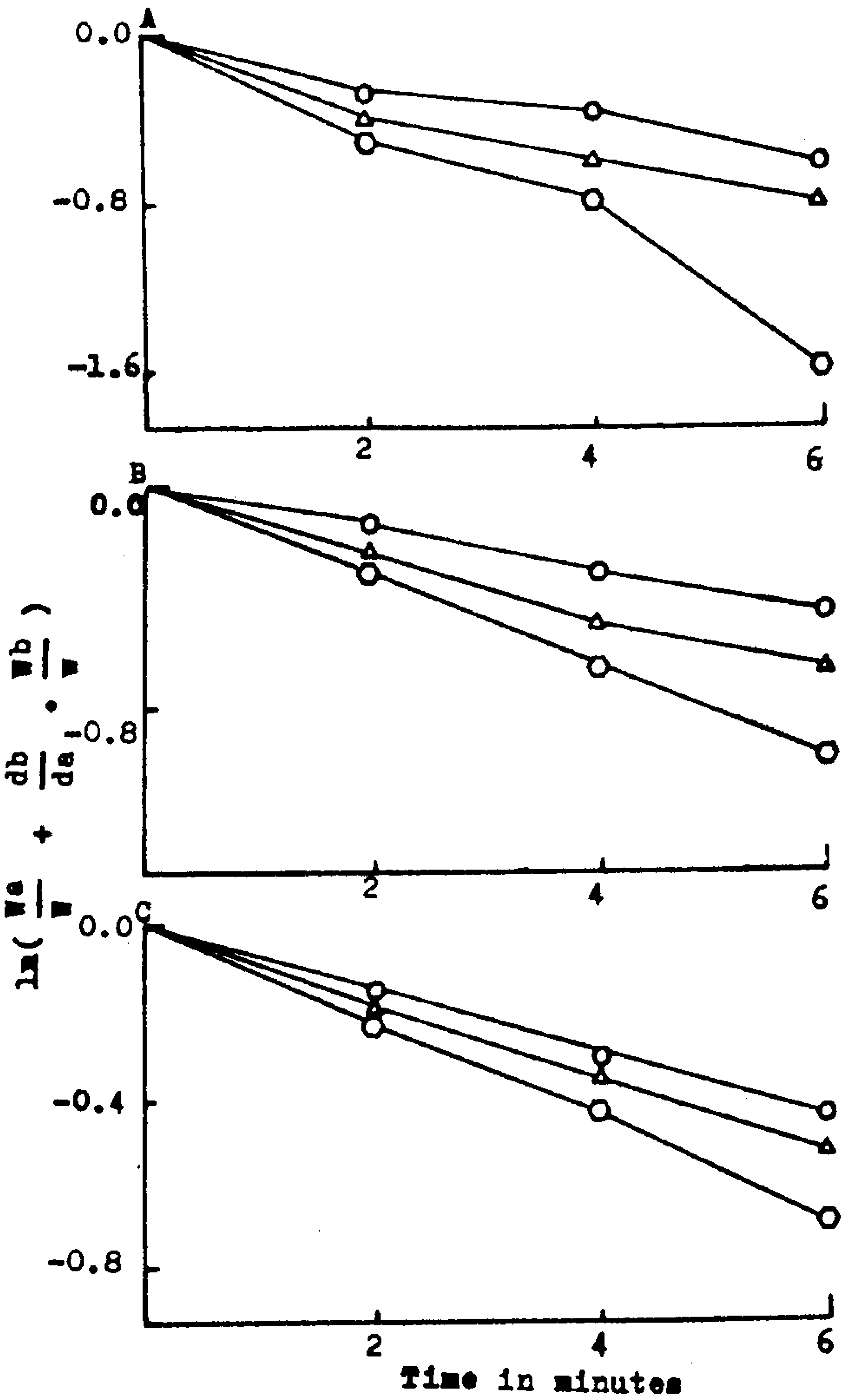

Figure 1.Effect of the Proportion of granules on the linearity of Mehta et al equation.

A=15 gm , B=30 gm , C=45 gm

O db=0.0 u , ▲db= 400 u , O db = 800 u

Factors controlling measurement of the crushing strength of calcium sulfate granules evaluated by ball mill

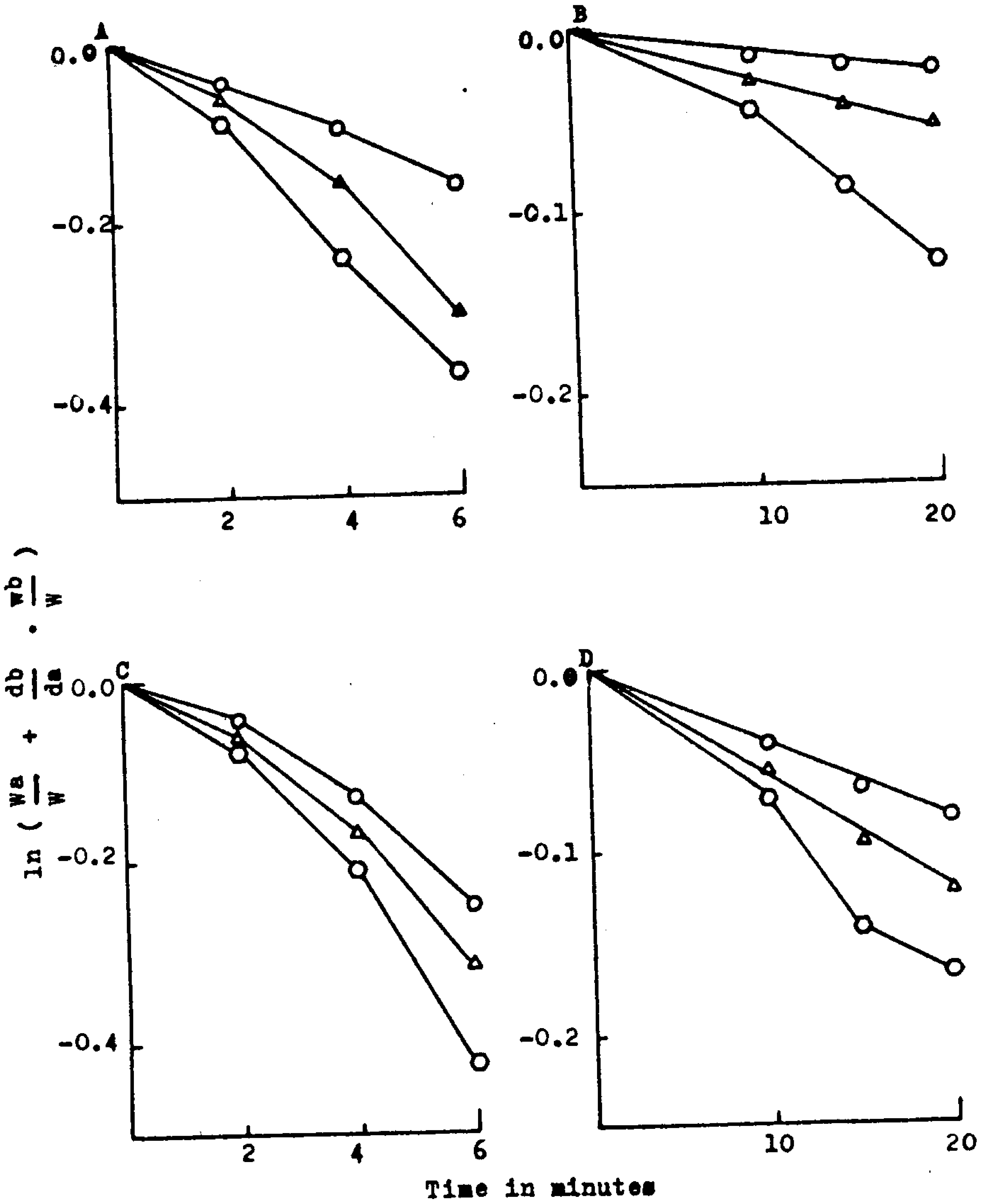

Figure 2. Effect of granule size on the linearity of the equation.
A and B = 1025 u , where, O db= 0.0, △ db= 300, O db= 600 u.
C and D = 558 u , where O db= 0.0, △ db= 100, O db= 200 u.

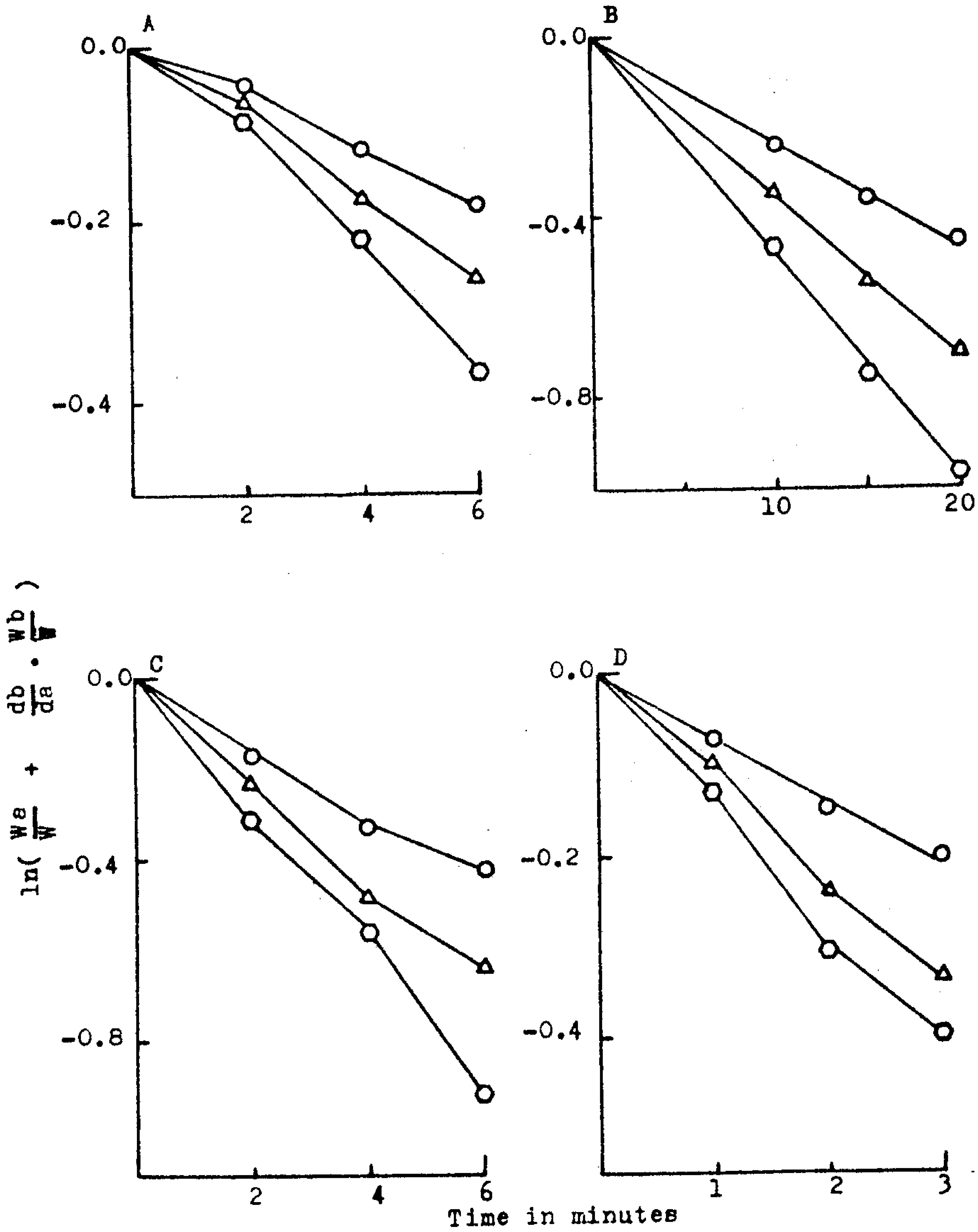

Figure 3. Effect of speed of rotation on the linearity of the equation.

A and B= low speed

C and D= high speed

O db= 0.0 u

Δ db= 400 u

O db= 800 u

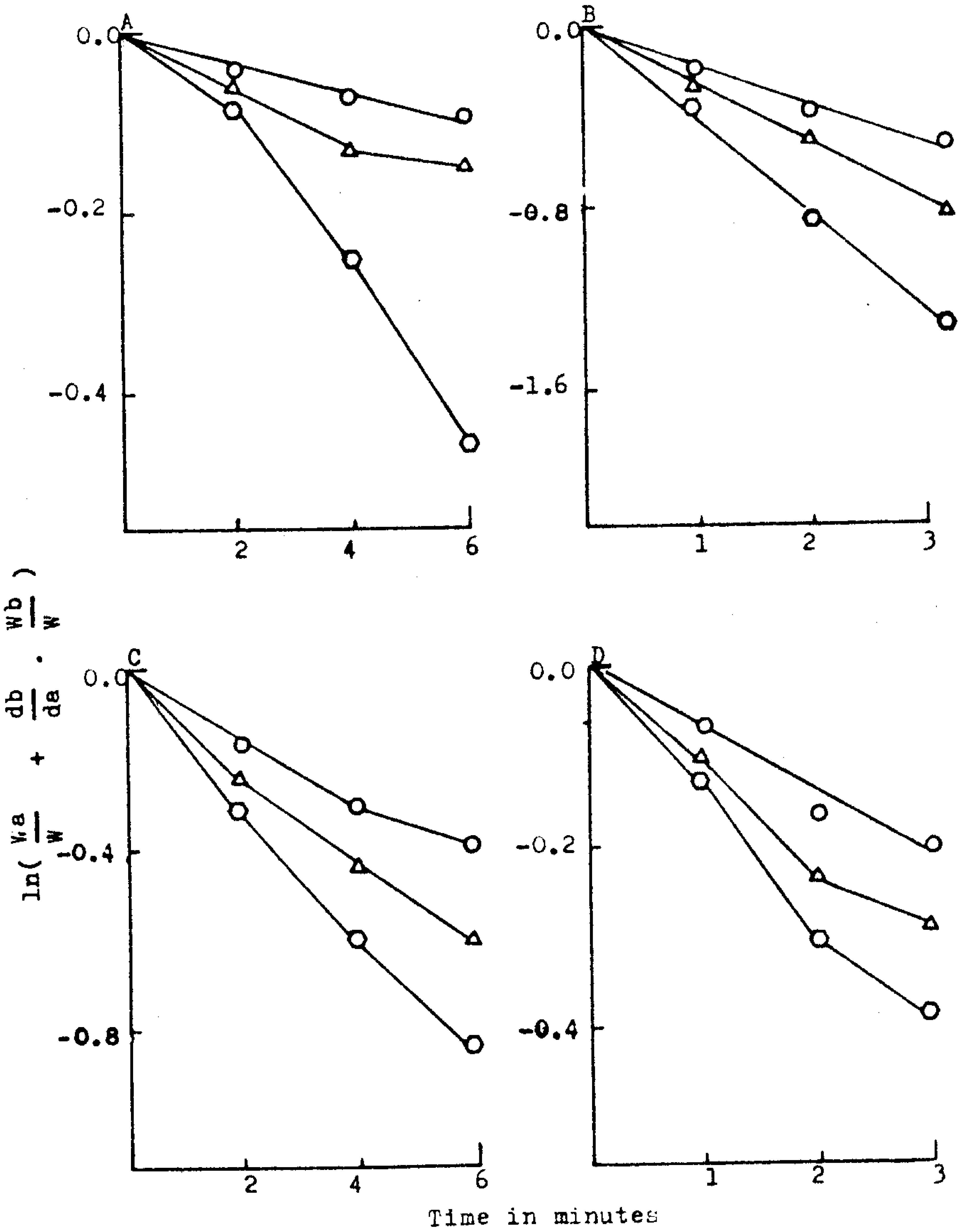

Figure 4. Effect of type of balls on the linearity of the equation.

A and B = mixed balls
C and D = glass balls
○ db= 0.0 u
△ db= 400 u
○ db= 800 u

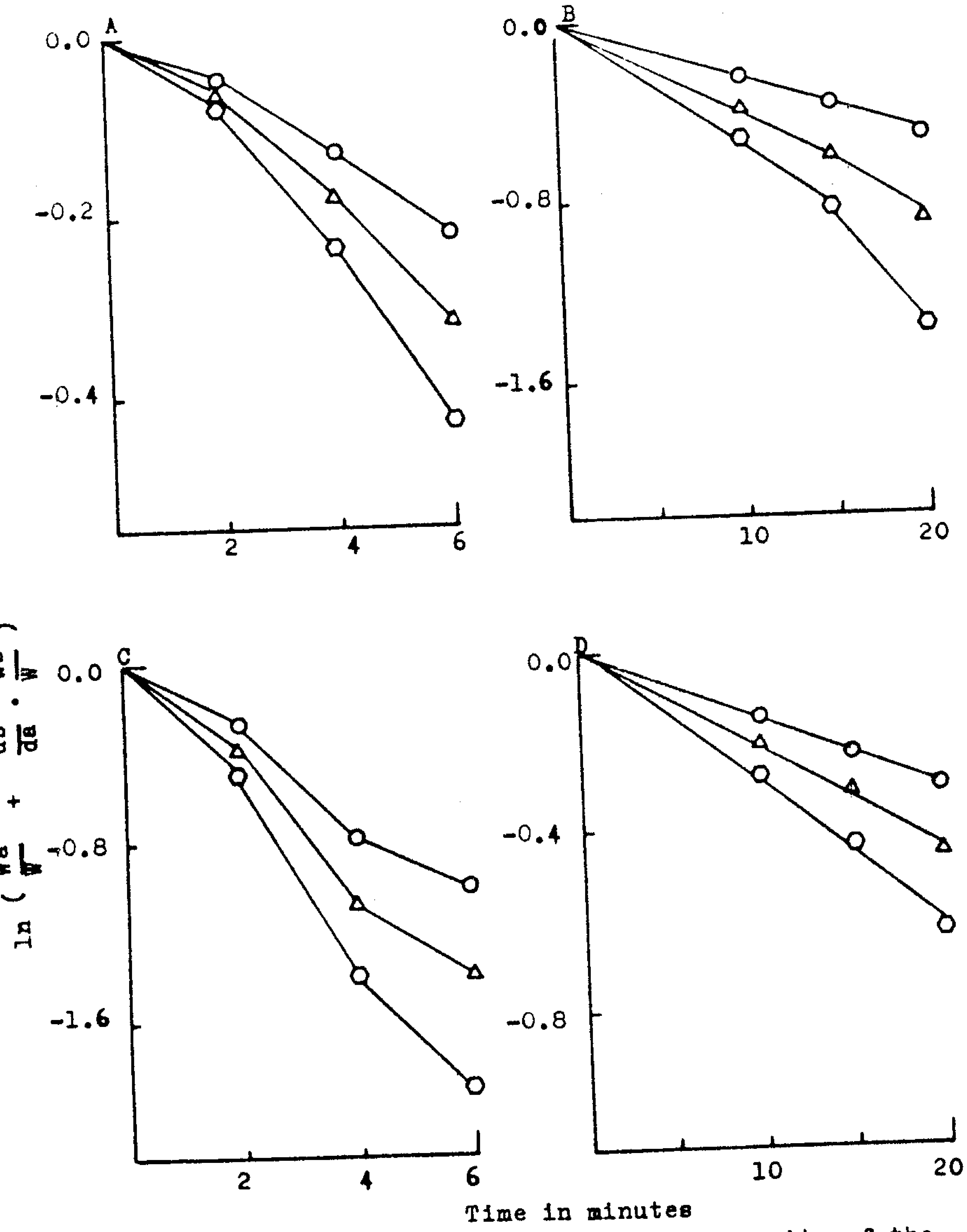

Figure 5. Effect of number of balls on the linearity of the equation.

A and B= 15 balls
C and D= 5 balls
O db= 0.0 u
Δ db= 400 u
O db= 800 u

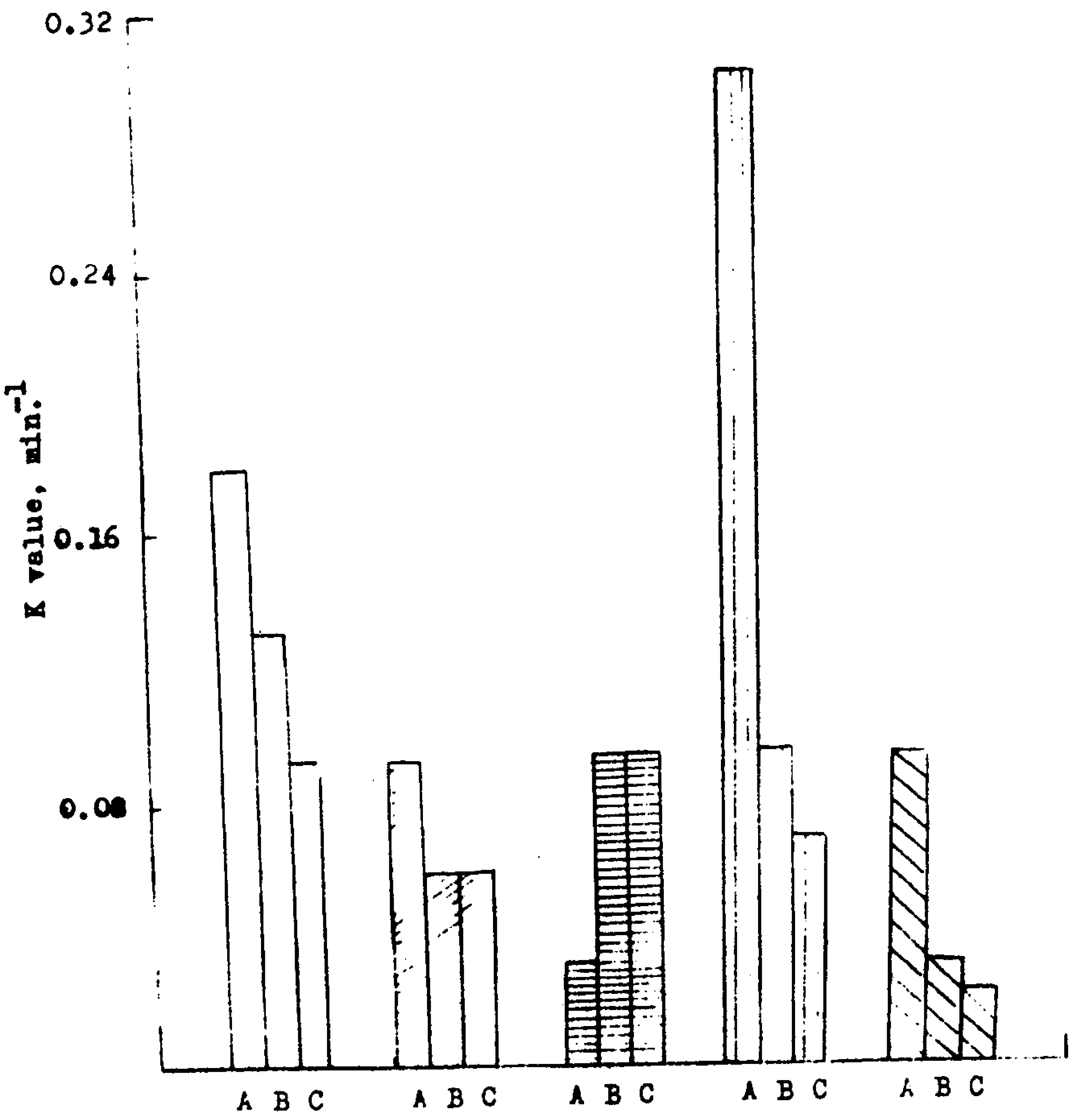

Figure 6. Effect of controlling factors on the K value of calcium sulfate granules.

▭ = proportion of the feed, A= 15 gm, B= 30 gm and C= 45 gm.

▨ = granule size, A= 1875 u, B= 1025 u, C= 558 u.

▤ = speed of rotation, A= low speed, B= moderate speed, C= high speed.

▥ = type of balls, A= mixed, B= porcelain, C= glass.

▧ =number of balls = 25, B= 15, C= 5.

REFERENCES

1) C.F. Harwood, N. Pipel (1968) J. Pharm.Sci., 57, 478-481).

2) D. Garderton, A.B. Selkirk (1969) J. Pharm. Pharmacol., 22, 345-349.

3) G. Gold, R.N. Duvall, B.T. Palermo, R. Hurtle (1971) J. Pharm. Sci., 60, 922-925.

4) E. Doelker, E. Shotton (1977) J. Pharm. Pharmacol. 29, 193-198.

5) W. Erni, W.A. Ritschel (1977) Pharm. Ind. 39, 284-290.

6) A. Mehta, M.A. Zoglio, J.T. Carstensen (1978) J. Pharm. Sci., 67, 905-908.

7) H.M. El-Sabbagh, A.H. Ghanem, H.M.Abdel-Alim(1982) the 17th. Egypt. Conf. Pharm. Sci., Cairo, Egypt

8) E.L. Parrot (1974) J. Pharm. Sci., 63, 813-829.

9) E.L. Parrot (1976) in "The Theory and Prctice of Industerial Pharmacy", 2nd edition, Lea and Febiger, Philadelphia, Chapter 15,

10) J.M. Coulson, J.F. Richardson, J.R. Bachhurrt, J.H. Harker (1978) Chemical Engineerings, Vol. 2, 3rd edition, Pergamon Press, Chapter 2.

11) R.W. Perry, C.H. Chilton (1978) Chemical Engineer's Handbook, 5th edition , Section 8.

العوامل المتحكمة فى قياس قوة سحق حبيبات كبريتات الكالسيوم بطريقة طاحونة الكرات

حمدى محمد عبد العليم ـ محمد حامد الشابورى
قسم الصيدلانيات ـ كلية الصيدلة ـ جامعة المنصورة ـ المنصورة

تم فى هذا البحث تحضير حبيبات كبريتات الكالسيوم المائية باستعمال محلول عديد فنيل البروليدون المائى . وقد قيمت القوة اللازمة لسحق هذه الحبيبات كذلك تمت دراسة العوامل التى قد تؤثر على هذه القوة مثل كمية الحبيبات وحجم الحبيبات وسرعة الدوران وعدد ونوع الكرات المستعملة .

وقد أثبتت الدراسة انه يمكن الحصول على خطية المعادلة بالسوائل الاتية :

١- زيادة كمية الحبيبات.
٢- زيادة زمن الطحن للحبيبات.
٣- تقليل زمن الطحن باستعمال كرات زجاجية أو خليط من الكرات صغيرة الحجم وكبيرة الحجم .
٤- تقليل زمن الطحن عند استخدام سرعة الدوران الكبيرة .

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