DRUG DEVELOPMENT AND INDUSTRIAL PHARMACY, 13(7), 1279-1291 (1987)

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A SIMPLE TECHNIQUE FOR MEASURING EXPANSION OF METHYLCELLULOSE FILMS IN WATER

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ABSTRACT

A simple and easy to assemble apparatus for the determination of expansion of methylcellulose films is described. The technique involved essentially the projection of the image of the film on to a screen and the measurement of the expansion of the film with time. From statistical analyses of the data obtained for film expansion, the method was found to be reliable. The size, shape and amount of methylcellulose/area of film were considered in the assessment of the technique.

INTRODUCTION

Cellulose derivatives are used either alone or in combination with other polymeric substances as coating materials (1-3) and as binders and disintegrants for tablet formulations (4-7). Films made from these polymers have been evaluated for mechanical properties such as tensile strength and elongation (8-11).

In a current study of tablet formulations involving methylcellulose as a binder it was necessary to examine the effect of this excipient on the granule and on the properties of tablets prepared from these granules. As a consequence, a simple yet reliable method to determine the swelling of the methylcellulose film in

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water was developed and this report gives the findings of the technique so applied.

EXPERIMENTAL

Material

Methylcellulose (British Drug Houses Chemicals, England) was used as the film forming substance.

Preparation of Film

A solution of 1% w/w methylcellulose in distilled water was prepared, by dispersing the methylcellulose in water at about 4°, the low temperature decreased the time taken to dissolve it. The solution was placed in an ultrasonic bath[1] to remove entrapped air bubbles. A known amount of methylcellulose solution was poured into a glass tray (23 cm x 23 cm) and allowed to spread over the entire surface, this was easily achieved at the laboratory temperature of 29±1°C. The glass tray had a flat surface, this was ascertained with the use of a spirit level to check the level of different parts of the surface of the tray. The tray with its content was dried at 60-70° in an oven for 4 hours which from preliminary experiments was found to be an adequate time to dry the film. The dried film was readily peeled off the tray and from a visual examination the film was seen to be clear, homogenous and continuous. The film after ageing for a day was cut very carefully into strips of requisite dimensions and stored in between cardboards. Freshly prepared films were used, although from preliminary investigations it was found that the expansion of films stored for 4, 7 and 14 days did not differ from that which was stored for a day.

Apparatus for Measurement of Film Expansion

A schematic representation of the apparatus used is given in Figure 1. It consisted of a 1 litre circular glass dish measuring

[1] Burndept Ltd., Erith, Kent.

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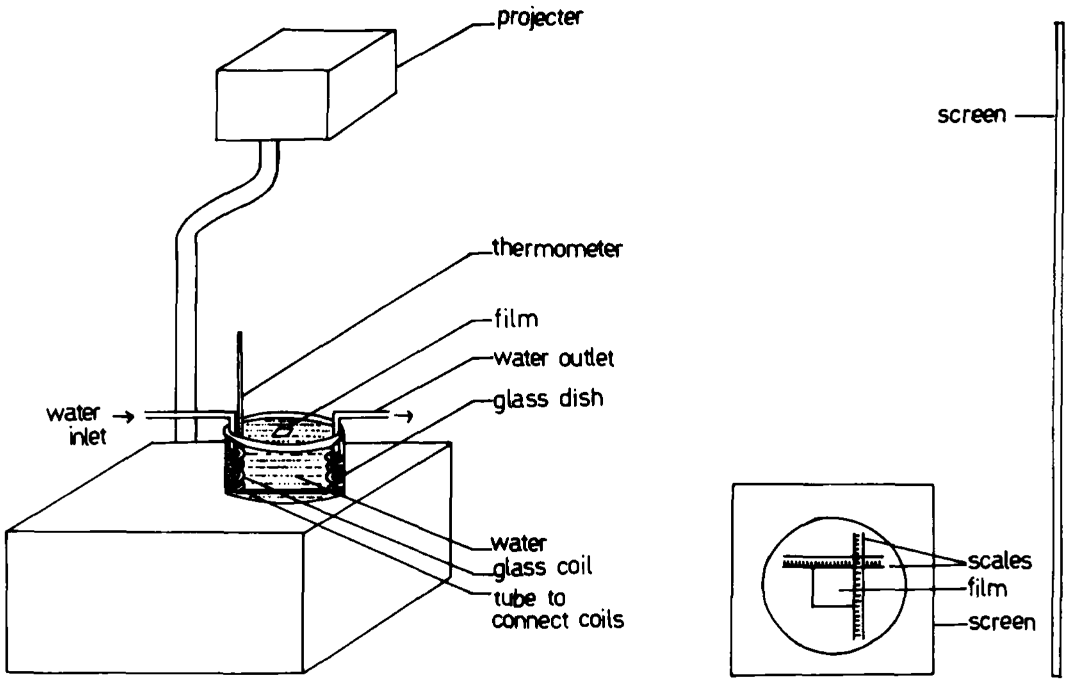

FIGURE 1 : Schematic representation of apparatus for measuring expansion of film in water

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15 cm in diameter and 7 cm deep, filled with distilled water up to a level of 6.5 cm. Two glass coils measuring about 1.5 cm wide and 6.5 cm long were interconnected and placed in the water in the dish. Water was circulated through the coils to maintain temperature of the dish at $37 \pm 0.5^{\circ}C$, the temperature chosen is related to that used for measuring disintegration and dissolution of tablets.

The dish was placed on the glass plate of the overhead projector. The film was allowed to remain on the surface of water. The magnified image of the film was projected on to a screen. Two transparent scales graded in mm divisions were used to measure the dimensions of the film image. The scales were held on the screen magnetically. The dimensions increased with time, due to swelling of the film.

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Measurement of Expansion of Film in Water

The methylcellulose film prepared as described in the above and cut very carefully to the required size and shape was placed on the surface of the water with the aid of a pair of forceps. The film in contact with water immediately unfurled and spread out. The dimensions of the film image projected on the screen were measured along one axis and also along an axis at right angles to the first axis. By means of an audio timing device, measurements of the expansion of the film with time were made. The edges of the film described a straight line. For each film, 5 replicates of measurements were carried out.

Variation of Amount of Methylcellulose in Film

Films containing two amounts of methylcellulose were prepared. They contained 0.628 mg/cm^2 and 1.256 mg/cm^2 of methylcellulose.

Variation of Size of Film

Three sizes of film were used, 1 cm x 1 cm, 2 cm x 2 cm and 2 cm x 1 cm.

Variation of Shape of Film

Two shapes were employed, squares and rectangular.

RESULTS AND DISCUSSION

Films of methylcellulose were found to swell when in contact with water. The results showed that the expansion of the film increased with time, initially, the expansion was fast and after attaining a peak expansion, the film either did not expand any more or expanded very slowly. Films, when allowed to remain on the surface of water for a long period, 2 hours, were observed to exhibit practically no change in their dimensions. The film when forced to submerge by pushing it into the water, to the bottom of the dish, did not dissolve even after a further period of 1 hour.

Tables 1-3 show the expansion of methylcellulose films (0.628 mg/cm^2) of two shapes, square and rectangular and of three sizes, 1 cm x 1 cm, 2 cm x 2 cm and 2 cm x 1 cm in water. Similarly, the

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TABLE 1

Linear Expansion of Square-shaped Methylcellulose Films (0.628 mg/cm²) Measuring 1 cm x 1 cm in Water at 37° with Time

Time (mins)	% Linear expansion along one axis (A) Film 1	Film 2	Film 3	Film 4	Film 5	Mean	± SD
1	6.06	5.97	7.46	6.06	4.55	6.02	1.03
2	10.61	10.45	11.94	12.12	9.09	10.84	1.24
3	13.64	13.43	14.93	15.15	13.64	14.16	0.81
4	15.15	14.93	16.42	16.67	16.67	15.97	0.86
5	16.67	16.42	17.91	18.18	18.18	17.47	0.85
6	18.18	17.91	17.91	18.18	19.70	18.38	0.75
7	18.18	17.91	17.91	18.18	19.70	18.38	0.75
	Correlation coefficient, r = 0.9320						
	% Linear expansion along axis perpendicular to (A)						
1	6.06	7.46	7.46	6.06	5.97	6.60	0.78
2	10.61	11.94	10.45	12.12	10.45	11.11	0.84
3	13.64	13.42	14.93	15.15	14.93	14.41	0.82
4	16.67	16.42	17.91	16.67	16.42	16.82	0.62
5	18.18	17.91	17.91	16.67	16.42	17.42	0.81
6	18.18	17.91	19.40	16.67	16.42	17.72	1.21
7	18.18	17.91	19.40	16.67	16.42	17.72	1.21
	Correlation coefficient, r = 0.8995						

data for expansion of films containing more methylcellulose, 1.256 mg/cm² are given in Tables 4-6. Statistical analyses of these data using the t-test, show that the observed t is less than the tabulated t value for 8 degrees of freedom and 5% probability level = 2.306 (Tables 7-8). Hence the Null Hypothesis is not rejected and therefore the differences are not significant.

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TABLE 2

Linear Expansion of Square-shaped Methylcellulose Films (0.628 mg/cm²) Measuring 2 cm x 2 cm in Water at 37°C with Time

Time (mins)	% Linear expansion along one axis (A) Film 1	Film 2	Film 3	Film 4	Film 5	Mean	± SD
1	9.02	9.63	6.67	6.06	9.92	8.26	1.77
2	13.53	14.81	10.37	10.61	14.50	12.76	2.13
3	15.79	17.04	13.33	12.88	16.80	15.17	1.95
4	18.05	18.52	14.81	15.91	18.32	17.12	1.66
5	18.80	18.52	15.56	17.42	19.85	18.03	1.63
6	18.80	18.52	16.30	18.18	19.85	18.33	1.30
7	18.80	18.52	16.30	18.18	19.85	18.33	1.30
	Correlation coefficient, r = 0.9060						
	% Linear expansion along axis perpendicular to (A)						
1	7.41	9.56	6.62	6.92	7.52	7.61	1.15
2	11.85	13.97	10.29	10.77	12.78	11.93	1.49
3	14.81	15.44	12.50	13.85	15.79	14.48	1.33
4	16.30	16.91	13.97	15.38	18.05	16.12	1.55
5	17.04	17.65	14.71	17.69	18.80	17.18	1.52
6	17.04	17.65	16.18	18.46	18.80	17.63	1.06
7	17.04	17.65	16.18	18.46	18.80	17.63	1.06
	Correlation coefficient, r = 0.9143						

The time taken for methylcellulose films to reach peak expansion and the expansion attained at that point are given in Table 9. The data obtained for these two parameters were about the same for films containing 0.628 mg/cm² or 1.256 mg/cm² methylcellulose, irrespective of shape and size. However, for films containing a greater amount of methylcellulose the time required

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TABLE 3

Linear Expansion of Rectangular-shaped Methylcellulose Films ($0.628\ mg/cm^2$) Measuring 2 cm x 1 cm in Water at 37° with Time

Time (mins)	% Linear expansion along one axis (A) Film 1	Film 2	Film 3	Film 4	Film 5	Mean	± SD
1	6.72	7.41	6.62	8.09	6.06	6.98	0.78
2	11.19	12.59	11.03	12.50	10.61	11.58	0.90
3	14.93	14.81	13.97	13.97	15.15	14.57	0.56
4	15.67	16.30	16.18	14.71	18.18	16.21	1.27
5	16.42	17.04	17.65	15.44	18.94	17.10	1.31
6	16.42	17.78	18.38	15.44	18.94	17.39	1.44
7	16.42	17.78	18.38	15.44	18.94	17.39	1.44
	Correlation coefficient, r = 0.8967						
	% Linear expansion along axis perpendicular to (A)						
1	6.06	5.97	4.55	7.46	5.97	6.00	1.03
2	10.61	10.45	10.61	11.94	8.96	10.51	1.06
3	13.64	13.43	15.15	14.93	11.94	13.82	1.30
4	16.67	16.42	18.18	17.91	14.93	16.82	1.30
5	18.18	17.91	19.70	17.91	16.42	17.42	1.33
6	18.18	19.40	21.21	17.91	17.91	18.92	1.42
7	18.18	19.40	21.21	17.91	17.91	18.92	1.42
	Correlation coefficient, r = 0.9402						

to achieve peak expansion was longer and the extent of expansion slightly smaller, indicating that swelling was slower with a thicker film.

The analysis of variance of the data for expansion of film of varying size is seen in Tables 10-11. The computed variance

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TABLE 4

Linear Exapansion of Square-shaped Methylcellulose Films (1.256 mg/cm^2) Measuring 1 cm x 1 cm in Water at 37° with Time

Time (mins)	% Linear expansion along one axis (A) Film 1	Film 2	Film 3	Film 4	Film 5	Mean	± SD
1	4.62	4.69	3.08	2.99	3.03	3.68	0.89
2	7.69	9.38	6.15	5.97	6.06	7.05	1.48
3	10.77	12.50	7.69	8.96	9.09	9.80	1.86
4	12.31	14.06	9.23	10.45	10.61	11.33	1.88
5	13.85	15.63	10.77	11.94	12.12	12.86	1.90
6	13.85	17.19	10.77	11.94	13.64	13.48	2.43
7	15.38	17.19	12.31	11.94	13.64	14.09	2.19
8	16.92	18.75	12.31	13.43	15.15	15.31	2.60
9	16.92	18.75	13.85	14.93	15.15	15.92	1.93
10	18.46	18.75	13.85	14.93	15.15	16.23	2.22
11	18.46	18.75	13.85	14.93	15.15	16.23	2.22
	Correlation coefficient, r = 0.9372						
	% Linear expansion along axis perpendicular to (A)						
1	6.06	4.55	4.48	4.41	4.48	4.80	0.71
2	9.09	9.09	7.46	7.35	5.97	7.79	1.32
3	13.64	12.12	8.96	10.29	10.45	11.09	1.81
4	13.64	13.64	10.45	11.76	11.94	12.29	1.36
5	15.15	15.15	10.45	13.24	13.43	13.48	1.92
6	16.67	16.67	11.94	13.24	13.43	14.39	2.16
7	18.18	18.18	11.94	14.71	14.93	15.59	2.64
8	18.18	18.18	13.43	16.18	14.93	16.18	2.07
9	19.70	19.70	16.42	16.18	16.42	17.68	1.84
10	19.70	19.70	16.42	16.18	16.42	17.68	1.84
11	19.70	19.70	16.42	16.18	16.42	17.68	1.84
	Correlation coefficient, r = 0.9487						

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TABLE 5

Linear Expansion of Square-shaped Methylcellulose Films (1.256 mg/cm^2) Measuring 2 cm x 2 cm in Water at 37° with Time

Time (mins)	% Linear expansion along one axis (A) Film 1	Film 2	Film 3	Film 4	Film 5	Mean	± SD
1	4.69	1.54	3.85	5.38	3.10	3.71	1.49
2	10.16	3.85	7.69	6.92	5.43	6.81	2.38
3	12.50	6.15	8.46	9.23	7.75	8.82	2.35
4	13.28	6.92	10.77	11.54	10.08	10.52	2.34
5	16.41	8.46	11.54	13.08	12.40	12.38	2.86
6	17.97	10.00	12.31	13.08	13.18	13.31	2.90
7	17.97	10.77	13.08	13.85	13.95	13.92	2.60
8	18.75	11.54	13.85	14.62	14.73	14.70	2.60
9	19.53	11.54	14.62	15.38	15.50	15.31	2.85
10	20.31	12.31	15.38	15.38	16.28	15.93	2.87
11	20.31	12.31	15.38	15.38	16.28	15.93	2.87
	Correlation coefficient, r = 0.9492						
	% Linear expansion along axis perpendicular to (A)						
1	4.58	3.03	2.27	6.87	3.85	4.12	1.76
2	9.16	6.06	7.58	7.63	6.92	7.47	1.14
3	11.45	9.09	9.85	9.92	10.00	10.06	0.86
4	13.74	10.61	11.36	12.21	12.31	12.05	1.17
5	16.03	12.12	12.88	13.74	14.62	13.88	1.52
6	17.56	12.88	14.39	15.27	16.15	15.25	1.77
7	18.32	13.64	15.15	16.03	16.92	16.01	1.77
8	19.08	14.39	15.91	16.79	17.69	16.77	1.77
9	19.85	15.15	16.67	17.56	18.46	17.54	1.78
10	20.61	15.91	17.42	18.32	19.23	18.30	1.78
11	20.61	15.91	17.42	18.32	19.23	18.30	1.78
	Correlation coefficient, r = 0.9513						

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TABLE 6

Linear Expansion of Rectangular-shaped Methylcellulose Films ($1.256 mg/cm^2$) Measuring 2 cm x 1 cm in Water at 37° with Time

Time (mins)	% Linear expansion along one axis (A) Film 1	Film 2	Film 3	Film 4	Film 5	Mean	± SD
1	4.72	4.69	3.15	3.88	3.15	3.92	0.78
2	10.24	7.81	6.30	6.98	6.30	7.53	1.64
3	11.81	10.94	7.87	8.53	9.45	9.72	1.64
4	14.96	12.50	8.66	10.08	11.02	11.44	2.41
5	16.54	14.84	11.02	11.63	11.81	13.17	2.40
6	16.54	15.63	12.60	13.95	13.39	14.42	1.62
7	18.90	17.19	14.96	14.73	14.17	15.99	1.99
8	18.90	17.97	17.32	14.73	14.17	16.62	2.07
9	19.69	18.75	18.90	15.50	14.96	17.56	2.17
10	20.47	19.53	19.69	16.28	15.75	18.34	2.16
11	20.47	19.53	19.69	16.28	15.75	18.34	2.16
	Correlation coefficient, r = 0.9643						
	% Linear expansion along axis perpendicular to (A)						
1	4.69	3.13	4.69	6.25	3.13	4.38	1.31
2	9.38	6.25	7.81	6.25	4.69	6.88	1.78
3	10.94	9.38	7.81	9.38	7.81	9.06	1.31
4	12.50	10.94	9.38	10.94	7.81	10.31	1.78
5	14.06	12.50	10.94	12.50	9.38	11.88	1.78
6	14.06	14.06	10.94	12.50	10.94	12.50	1.56
7	15.63	14.00	12.50	14.06	10.94	13.44	1.78
8	17.19	15.63	12.50	15.63	10.94	14.38	2.57
9	17.19	15.63	14.06	15.63	12.50	15.00	1.78
10	18.75	17.19	14.06	15.63	12.50	15.63	2.47
11	18.75	17.19	14.06	15.63	12.50	15.63	2.47
	Correlation coefficient, r = 0.9627						

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TABLE 7

T-test for Data Obtained for Expansion Along Two Axes of Methylcelluose Films (0.628 mg/cm^2) of Different Size 1 x 1 cm (t-1), 2 x 2 cm (t-2), and 2 x 1 cm (t-3) for Different Periods of Contact with Water at 37°

Time (mins)	1	2	3	4	5
t-1	1.00	0.40	0.48	1.80	0.10
t-2	0.69	0.71	0.65	0.98	0.85
t-3	1.70	1.72	1.19	0.75	0.38

TABLE 8

T-test for Data Obtained for Expansion Along Two Axes of Methylcellulose Films (1.256 mg/cm^2) of Different Size 1 x 1 cm (t-1), 2 x 2 cm (t-2) and 2 x 1 cm (t-3) for Different Periods of Contact with Water at 37°

Time (mins)	1	2	3	4	5	6	7
t-1	2.20	0.83	1.11	0.93	0.51	0.63	0.98
t-2	0.40	0.56	1.11	1.31	1.04	1.28	1.49
t-3	0.67	0.60	0.70	0.84	0.97	1.91	2.14

ratio, F is less than F (2,27) for 5% significance level, which is 3.35. Thus, the differences among films of varying size, 1 cm x 1 cm, 2 cm x 2 cm and 2 cm x 1 cm and within the same size film are not significant. It can therefore be said that expansion is not dependent on the dimensions of the film and not on whether it is square or rectangular for a film containing a given amount of methylcellulose/area, or in a broad sense, thickness of the film.

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TABLE 9

Peak Expansion of Methylcellulose Films in Water at 37° and Time Taken to Attain It

Amount of methyl-cellulose/ area of film	Size of film (cm)	Shape of film	Peak expansion (%)	Time taken to reach peak expansion (mins)
0.628 mg/cm^2	1 x 1	Square	18.05$\pm$ 1.01	5.20$\pm$0.79
	2 x 2	Square	17.98$\pm$1.18	5.30$\pm$0.67
	2 x 1	Rectangular	18.16$\pm$1.57	5.40$\pm$0.70
1.256 mg/cm^2	1 x 1	Square	16.65$\pm$2.09	8.60$\pm$0.52
	2 x 2	Square	17.12$\pm$2.58	9.90$\pm$0.32
	2 x 1	Rectangular	16.99$\pm$2.62	9.60$\pm$0.70

TABLE 10

Analysis of Variance for Data Obtained from Peak Expansion of Methylcellulose Films (0.628 mg/cm^2) of Varying Size, 1 x 1 cm, 2 x 2 cm and 2 x 1 cm

Source of variation	DF	SS	MS = SS/DF	F-ratio
Among films	2	0.17	0.09	0.05
Within films	27	44.58	1.65	
Total	29	44.75		

TABLE 11

Analysis of Variance for Data Obtained from Peak Expansion of Methylcellulose Films (1.256 mg/cm^2) of Varying Size, 1 x 1 cm, 2 x 2 cm and 2 x 1 cm

Source of variation	DF	SS	MS = SS/DF	F-ratio
Among films	2	0.14	0.07	0.01
Within films	27	160.06	5.93	
Total	29	160.20		

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In conclusion, the data presented here indicate that the technique adopted to measure expansion of films is reliable and produces results which when analysed statistically show that the differences in film expansion along two axes is not significant. The method is applicable to films of different size and shape.

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