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MEASUREMENT OF SOFT ELASTIC GELATIN CAPSULE FIRMNESS WITH A UNIVERSAL TESTING MACHINE

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ABSTRACT

An indication of the firmness of Soft Elastic Gelatin (SEG) capsules was obtained by measurement in a universal testing machine (Instron, Model 1122). Capsules were packed in both glass and polystyrene containers and then exposed them in closed containers to 25°C & 75% RH, 35°C & 75% RH, and 45°C & 75% RH for a period of 21 weeks. At the end of 2, 12, and 21 week periods, samples were withdrawn from environmental chambers and evaluated them for firmness by subjective as well as objective techniques. The deformation of capsules measured by the universal testing machine appears to be a reliable and sensitive method for measuring firmness of SEG Capsules.

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INTRODUCTION

Firmness is essentially a characteristic experienced by the senses and indeed it arises from the physical attributes of the product. The common practice of evaluating the firmness of capsules is by 'feeling' and comparing them to a so-called Control in the stability studies. The 'feel' test usually consists of taking a SEG capsule and squeezing it gently between fingers and sensing the amount of deformation in the capsule. The capsule that shows little deformation is characterized as 'firm' while the capsule that shows more deformation is classified as 'soft'.

As it is evident, this is a subjective evaluation of the amount of deformation for a given force applied on the capsule. A capsule that shows little deviation in deformation parameters to 'Control' is usually judged as a high quality product.

Since the softness of a capsule is used as a measure in the physical parameters evaluation of a stability study, an objective method measuring firmness of SEG Capsules is necessary.

EXPERIMENTAL

Oval Soft Gelatin Capsules, lot 85683 were packed in 100 cc glass and polystyrene containers with continuous thread metal closures and all the bottles were torqued down[1] to 20 inch-pounds.

Then, these bottles were kept in 25°C & 75% RH; 35°C & 75% RH; and 45°C & 75% RH environmental chambers. Samples (one bottle of each) were collected at the end of 2, 12, and 21 week periods, and allowed them to equilibrate to the ambient conditions in the laboratory.

Sensory Evaluation

This evaluation was performed to a 1 to 5 scale. In order to be consistent in the evaluation judgement, some criteria was given to each score in the scale. They are: 1 - firm; 2 - slightly firm; 3 - soft; 4 - very soft; and 5 - sticky. At least three capsules from each bottle were checked to assign a score to a given conditions.

Objective Evaluation

An universal testing machine[2] was used in an attempt to measure the firmness of SEG Capsules. This machine has a moving crosshead. A probe can be fastened to the crosshead so that it can squeeze the capsule. The resistance offered by the capsule to the free movement of probe is sensed in an electric bonded-wire strain gauge. The output is recorded on a high speed stripchart, which is driven synchronously with the crosshead.

Theory

This test consists of measuring the force required to

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1. Spring Torque Tester, Owens Illinois.

2. Instron Model 1122, Instron Engineering Corporation, Canton, Mass.

punch a probe to such a depth into the SEG Capsule that causes deformation. This is a simple testing procedure to measure firmness of capsules.

Bourne (1,2) postulated an equation that the force in a puncture test is a function of two factors. They are the area under the punch tip and perimeter of the punch tip. Then he related these factors through two equations:

$$\frac{F}{A} = 4\ Kp \cdot \frac{1}{D} + K_a + \frac{4c}{D} \qquad 1$$

$$\frac{F}{P} = \frac{KaD}{4} + Kp + \frac{C}{D} \qquad 2$$

Where F is deformation force, D is diameter of punch, P is perimeter of punch, Kp is a perimeter related to coefficient of the capsule, Ka is an area-related coefficient of the capsule, and C is a constant that is usually close to zero.

A rectilinear plot of F/A vs 1/D results in a straight line with a slope of 4 Kp. Similarly, a plot of F/P vs D yield a straight line graph with a slope of Ka/4. This is the way one can obtain area dependent and perimeter dependent coefficients of a given substance under study.

In this study, four cylindrical probes with diameters, 0.500", 0.444", 0.377", and 0.314" were used. At all times of data collection, the following parameters were kept constant: Crosshead speed 0.5 inches per minute; strip chart speed 5

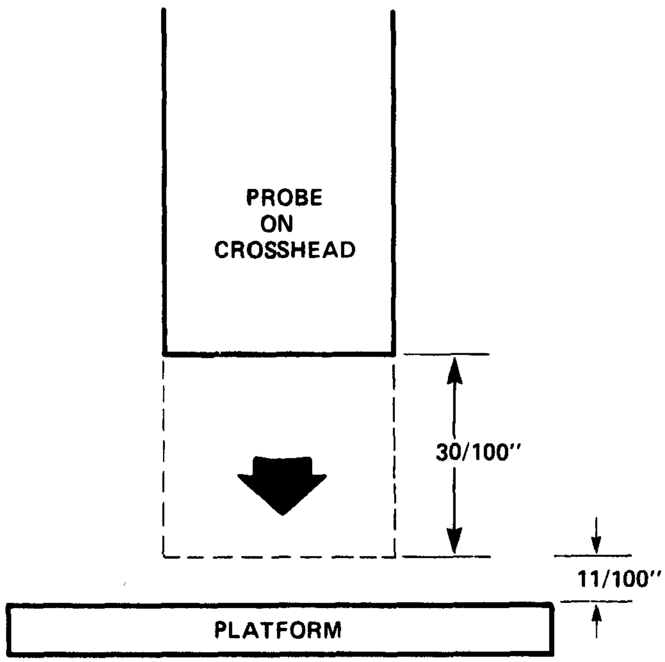

Figure 1: Schematic Represenation of probe on Crosshead relative to the platform. Dotted line shows the distance the probe set to move in the direction of the arrow. The clearance between the platform and the probe at its maximum travel is set at 11/100".

inches/minute; clearance between crosshead lower limit and platform 11/100"; and distance travelled by crosshead 30/100". These details are also clearly delineated in Figure 1. The crosshead probe is set up in such a way that it returns back to its initial condition after traveling 30/100 of an inch.

RESULTS AND DISCUSSION

Figure 2 shows effect of storage conditions and the container in which the product is packed. As it is evident from the graphs 2A, B, C, and D, the force is dependent on the

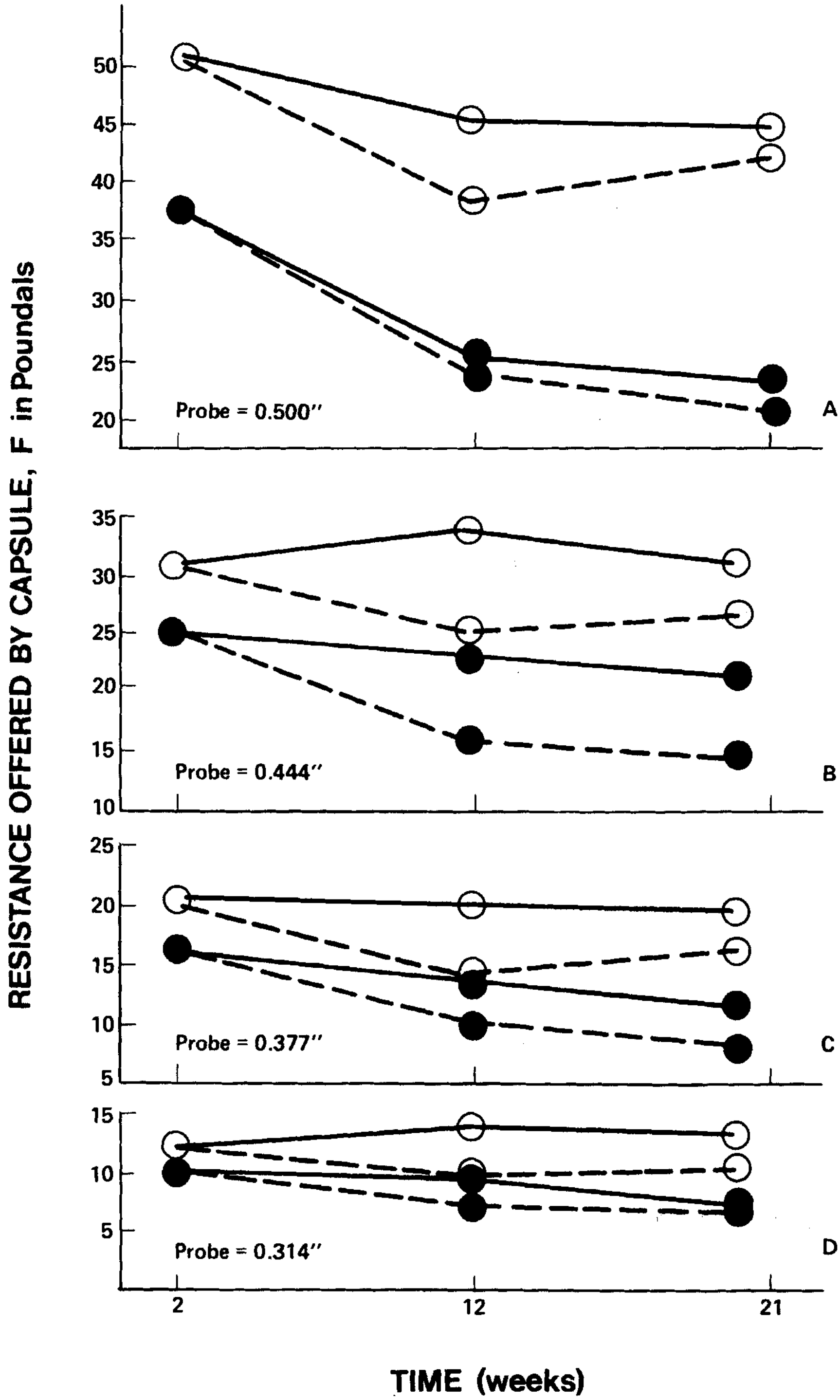

FIGURE 2

Soft Elastic Gelatin Capsules were sampled at 2, 12, and 21 weeks. The relationship between F, time, and container is shown (O glass container, ● polystyrene container, —— -25°C & 75% RH, and --- 35°C & 75% RH).

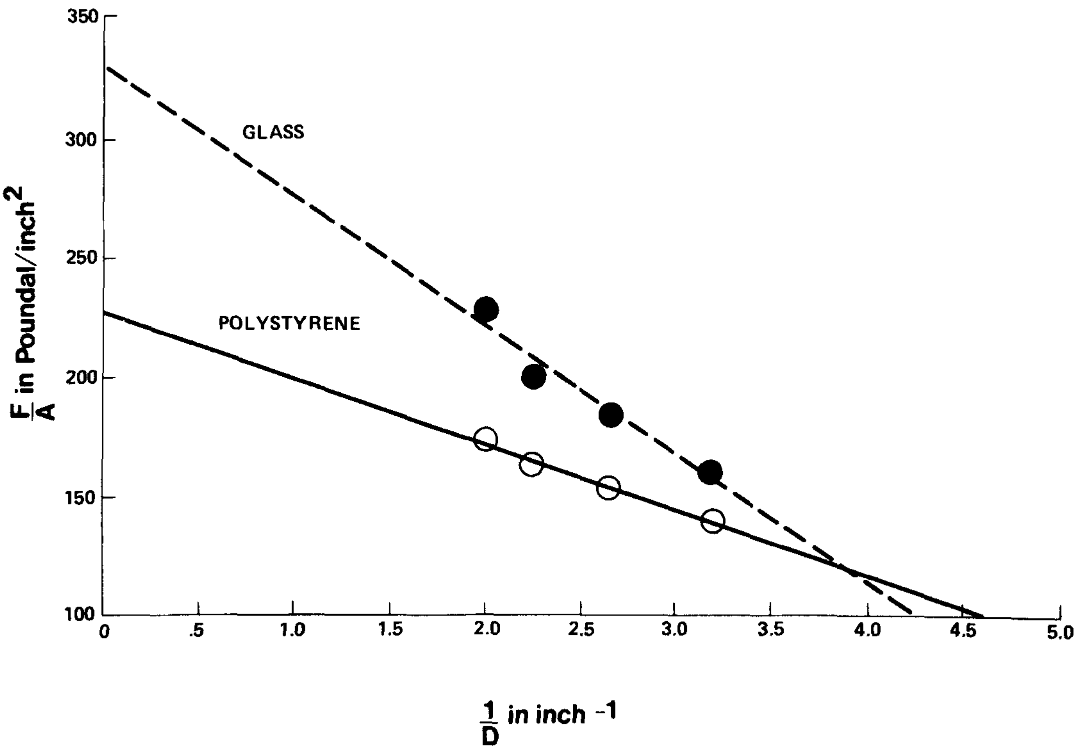

FIGURE 3

Typical plot for Equation 1. Slope of these curves provide perimeter related coefficient for Capsules, Kp. The data used for this plot is obtained for samples stored at 35°C & 75% RH.

probes surface. Furthermore, there is an interesting trend observed for capsules stored in glass at 35°C and 75% RH. The capsules tend to appear more firm at 21 weeks compared to 12 weeks. The reason for this is not quite evident at this time. In an effort to normalize parameter force, the data is fitted to Equations 1 and 2.

Figures 3 and 4 are the typical plots of equations 1 and 2 respectively. Kp and Ka parameters were calculated for all

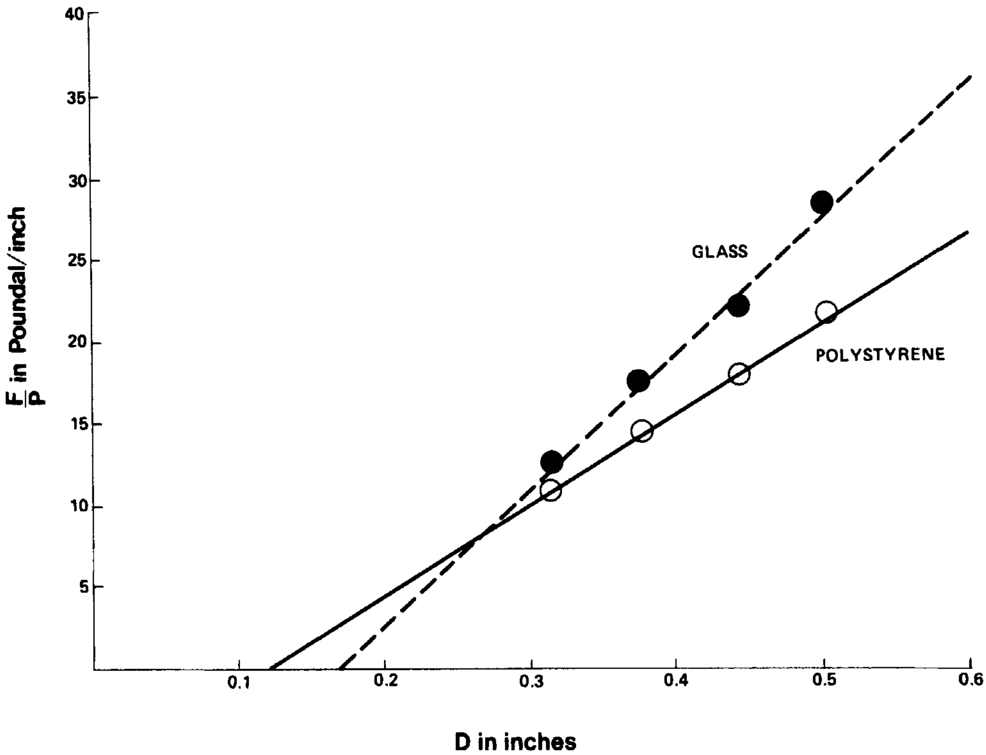

FIGURE 4

Typical plot for Equation 2. Slope of these curves provide area related coefficient for Capsule, Ka. The data used for this plot is obtained for samples stored around 35°C & 75% RH.

the collected data. Table 1 shows the parameter values, regression equations and correlation coefficients of regression equations for all the conditions studied except 45°C and 75% RH. Capsules stored at 45°C & 75% RH after two weeks are inseparably sticky and therefore were eliminated from the remaining study.

Figures 5 and 6 are the plots for Kp and Ka versus storage time. The rate of change in Kp or Ka is considerably reduced after 12 weeks when the product is stored in glass, while the

TABLE 1: SUMMARY ON DERIVING Kp and Ka VALUES

# of weeks stored	Storage Condition 75% RH	GLASS Linear Regression Eq.	GLASS Corr. Coeff.	GLASS Kp Poundal/inch	GLASS Ka Poundal/inch^2	POLYSTYRENE Linear Regression Eq.	POLYSTYRENE Corr. Coeff.	POLYSTYRENE Kp Pundal/Inch	POLYSTYRENE Ka Poundal/Inch^2
2	25°C	$\frac{F}{A} = 295.4-42.1\frac{1}{D}$	1.0	-10.4		$\frac{F}{A} = 218.6-24.7\frac{1}{D}$	1.0	-6.2	
		$\frac{F}{P} = -10.29+73.3D$	1.0		293	$\frac{F}{P} = 6.17+54.6\ D$	1.0		218
	35°C	$\frac{F}{A} = 273.9-34.98\frac{1}{D}$	0.98	-8.7		$\frac{F}{A} = 218.6-24.7\frac{1}{D}$	1.0	-6.2	
		$\frac{F}{P} = -8.3+67.31D$	0.99		269	$\frac{F}{P} = -6.32+55.27\frac{1}{D}$	0.99		221
12	25°C	$\frac{F}{A} = 329.6-50.3\frac{1}{D}$	0.93	-12.6		$\frac{F}{A} = 222.6-32.8\frac{1}{D}$	0.98	-8.2	
		$\frac{F}{P} = -13.6+84.8D$	0.99		339	$\frac{F}{P} = -8.4-56.24$	0.99		225
	35°C	$\frac{F}{A} = 248-39.4\frac{1}{D}$	0.91	-9.9		$\frac{F}{A} = 120.4-19.8\frac{1}{D}$	0.70	-4.95	
		$\frac{F}{P} = -10.61+64.1D$	0.98		256	$\frac{F}{P} = 2.3+30.87\ D$	0.98		124
21	25°C	$\frac{F}{A} = 323.6-52.5\frac{1}{D}$	0.93	-13.1		$\frac{F}{A} = 227.7-41.8\frac{1}{D}$	0.94	-10.5	
		$\frac{F}{P} = -14.36+84.2D$	0.99		337	$\frac{F}{P} = -11.5+59.5\ D$	0.97		238
	35°C	$\frac{F}{A} = 266.6-41.8\frac{1}{D}$	0.99	-10.5		$\frac{F}{A} = 127.5-17.02\frac{1}{D}$	0.73	-4.3	
		$\frac{F}{P} = -10.6+66.9\ D$	1.0		268	$\frac{F}{P} = -4.99+33.00\ D$	0.95		132

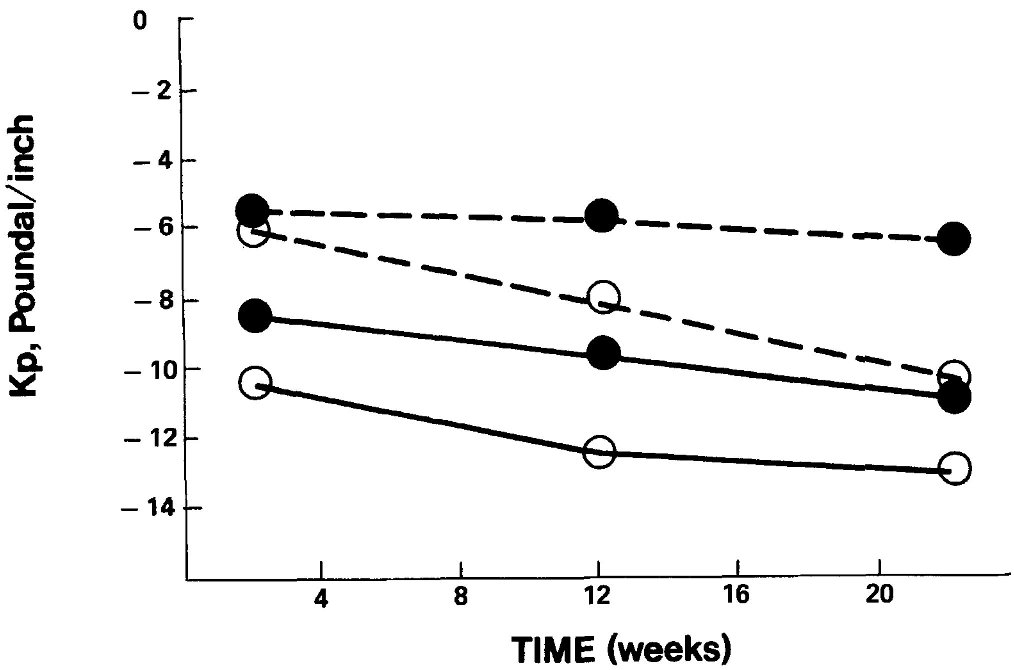

FIGURE 5

Relationship between Kp and various storage parameters (O glass, ● polystyrene, ——, 25°C & 75% RH, --- 35°C & 75% RH).

rate of change in Kp or Ka remained the same throughout the study period for polystyrene. This indicates that the capsules in glass undergo a change in mechanical strength in the initial period and then reach a state of equilibrium with respect to mechanical strength. On the other hand, when the capsules are stored in polystyrene container, the rate of decrease in mechanical strength is continued throughout the experimental period.

Figure 7 shows that the percent moisture gain is about five times higher in polystyrene compared to glass containers for the

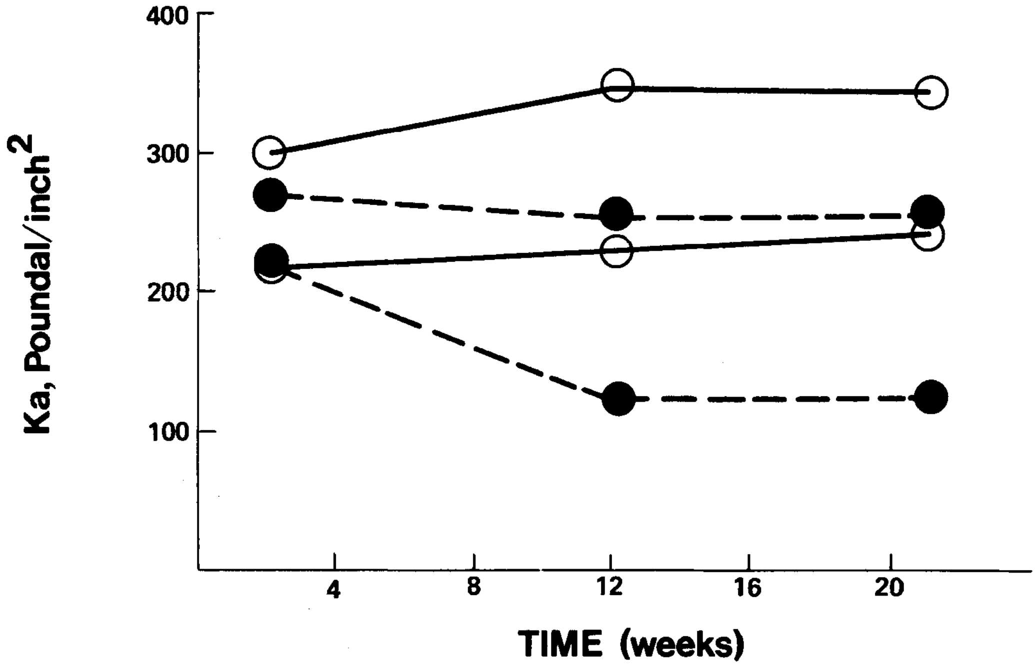

FIGURE 6

Relationship between Ka and various storage parameters (O glass, ● polystyrene, —— 25°C & 75% RH, and --- 35°C & 75% RH).

product tested.

Constant negative slope of Kp or Ka vs. time for capsules stored in polystyrene in Figures 5 and 6 can be attributed to the higher moisture pickup. As it is indicative from Figure 7, the moisture pickup by capsules stored in glass is considerably lower and it perhaps is the reason for decreased rate for Kp or Ka in Figures 5 and 6.

Figure 8 is a plot of Ka vs. sensory score and Figure 9 is Kp vs. sensory score. Ka or Kp values calculated for the

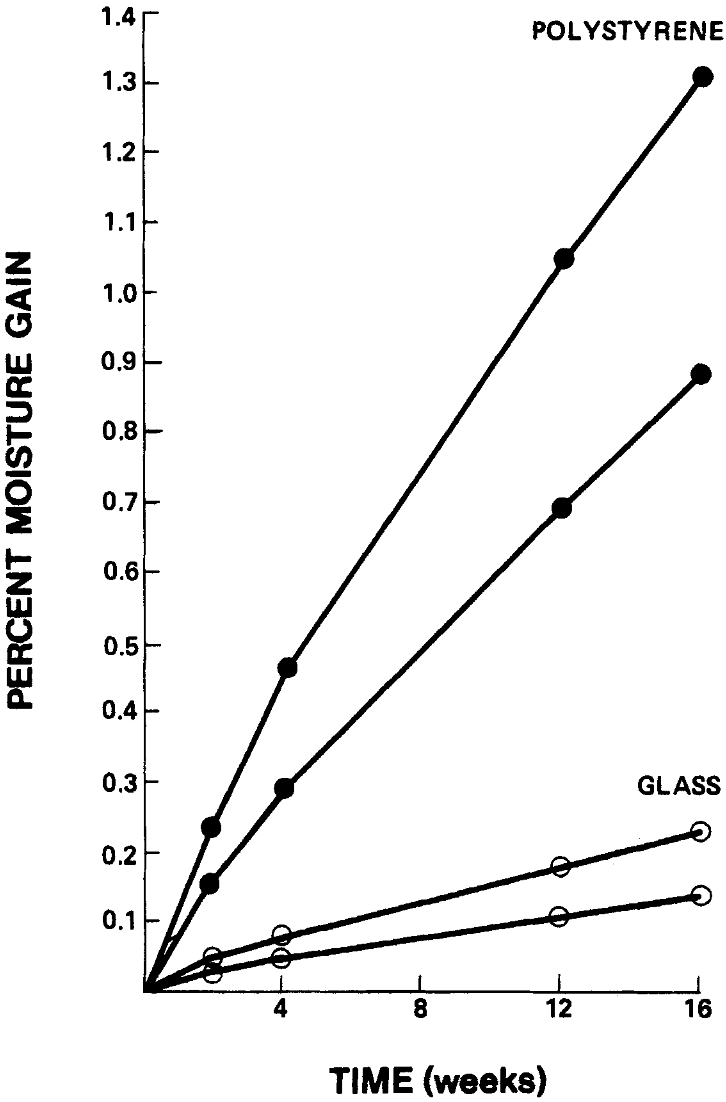

FIGURE 7

Percent Moisture Gain observed for both glass and polystyrene is shown for a period of 16 weeks.

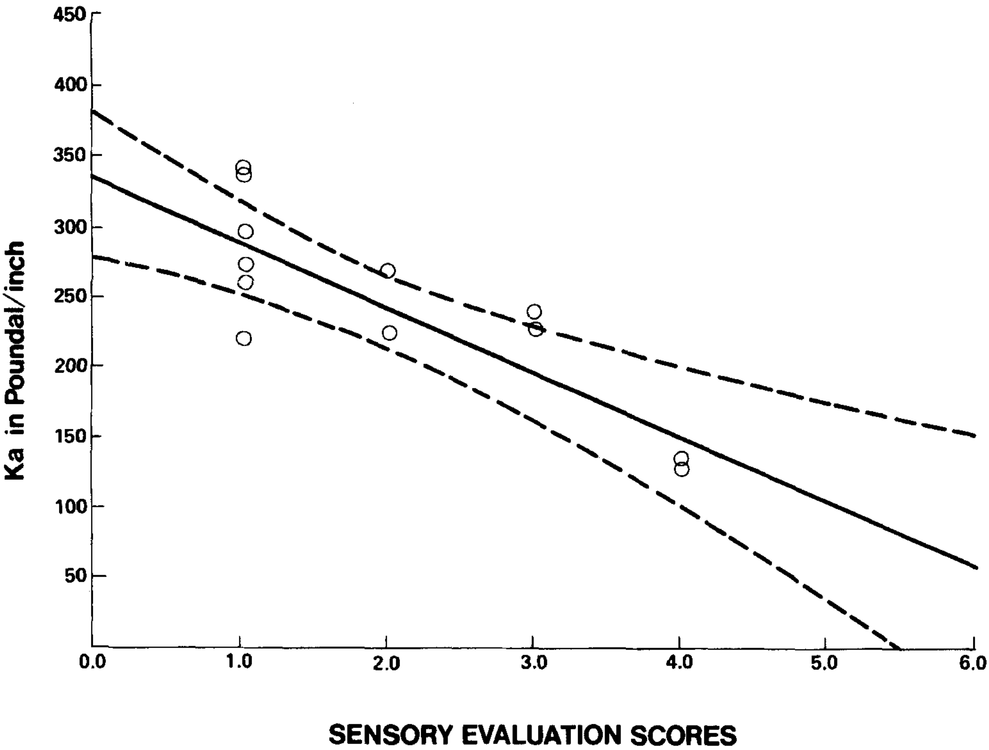

FIGURE 8

Ka plotted against sensory rating for 12 different points. The regression equation of the line is $\overline{A}^{F}$ = 335.46-46.06X, r= 0.82. The dashed lines on both sides of the regression line represent 95% confidence limits.

Capsules stored both in glass and polystyrene are plotted against sensory evaluation scores. The correlation coefficient for the regression line in Figure 8 is 0.82. In other words, the regression line represented 68% (r^2 X 100) of time by the data points. Similarly, the correlation coefficient for the line in Fig. 9 is 0.62 or the regression line is represented by the data points only 38% of the time. This indicates poor correla-

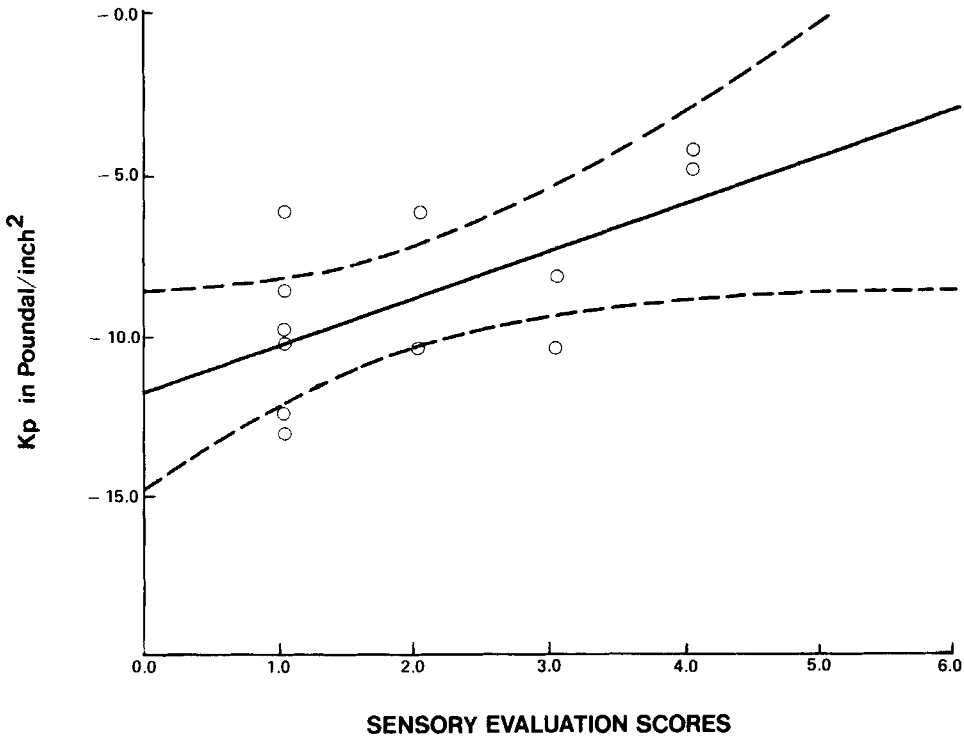

FIGURE 9

Kp plotted against sensory rating for 12 different points.

The regression equation of the line is $\frac{F}{P} = -11.76 + 1.48$, r=0.62.

The dashed lines on both sides of the regression line represent 95% confidence limits.

tion between sensory evaluation and objecive evaluation. Throughout the experimental study, the universal testing machine showed superiority in assessing the mechanical strength of capsules. Furthermore sensory evaluation is not quantifiable in a systematic manner. Thus, this poor correlation is attributed to the limitations of subjective evaluation.

Finally, when the capsules were stored at 45°C and 75% RH, the capsules begin to fuse together as early as 2 weeks after storage. The capsule fusing problem is progressively worse with time. During sensory evaluation all these samples received a score of 5.

Since this technique has the ability to quantify the firmness of Capsules, one can conveniently adopt this method to evaluate firmness for various occassions such as Stability Studies, screening of various contract manufactured products, quality control, etc.

CONCLUSION

1. SEG Capsules stored in glass containers retained their mechanical strength much better than the ones stored in polystyrene at both 25°C & 75% RH and 35°C & 75% RH.
2. Instron Universal Testing Machine provides a reliable method for the measurement of Capsule firmness (mechanical strength).
3. A relationship between moisture pick up and Kp or Kc appears to exist.

REFERENCES

1. Bourne, M.C., J. Food Sci., 31, 282 (1966).
2. Bourne, M.C., J. Texture Studies, 5, 459 (1975).