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Some emulsifying and suspending properties of a polysaccharide gum derived from *Mucuna flagillepes*, Papilionaceae

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The emulsifying and suspending properties of a new polysaccharide gum derived from an edible bean, *Mucuna flagillepes* have been investigated. The stability of the emulsion prepared with the gum was compared with that of emulsions prepared with acacia or tragacanth. Sherman's equations for concentrated emulsions were applied to determine the rate of coalescence k, and changes in mean cube globule diameter. Suspensions of sulphdimidine or zinc oxide prepared with tragacanth gum were compared with those prepared with mucuna gum. The final sedimentation height, H_u for each suspension was predicted using a biexponential relationship in the sedimentation pattern. The indication is that mucuna gum can be usefully employed as both an emulsifying and a suspending agent.

Mucuna flagillepes is a climber of the Papilionaceae family, grown on a subsistence level by some Igbos of the South East of Nigeria. The ripe, dry pods contain black and extremely hard seeds. The white cotyledons pulverized after roasting or cooking are added to broths for thickening. The gum has the disadvantage of darkening such preparations during a relatively short storage period. This darkening reaction is not peculiar to mucuna; it exists in root crops such as potato and *Dioscorea* species. The darkening reaction is obviously a serious disadvantage in the use of mucuna in any formulation. Considering the ease with which this bean can be cultivated and that the darkening reaction can be prevented by formulation, mucuna gum could find a place in the gum industry. The present investigation is designed to give some indication of its potential as an emulsifying as well as a suspending agent.

MATERIALS AND METHODS

Materials

The chemicals used were mostly Merck standard chemicals and include benzoic acid, gum acacia, gum tragacanth, zinc oxide, liquid paraffin, glycine, hydrochloric acid and ethanol (95% v/v). Nigrosin, glycerine and sulphadimidine were purchased from Fisher and Clonmel.

Preparation of mucuna gum

The tough dry seed coats were removed, the cotyledons were placed in Sorensen glycine buffer (pH 3·6) and autoclaved for 15 min at 121 °C. This helps to inactivate the enzymes usually present (Chudzikowski 1970). The cotyledons were then air-dried, pulverized in a hammer mill and screened through a 0·250 mm aperture sieve. A 10% w/v mucuna mucilage was prepared in the glycine buffer and allowed to hydrate for 24 h. The dispersion was then centrifuged for 30 min at 3000 rev mm^{-1}. Water was removed from the water-soluble polysaccharide retained in the supernatant by large volumes of 95% v/v ethanol using the method of Whistler & Max (1965).

Emulsifying property and emulsion stability

Batches of liquid paraffin emulsions containing 4:6 oil–water ratio were formulated with mucuna gum in concentrations between 0·4 and 1·0 w/v. The weighed amount of gum was sprinkled onto the measured amount of oil and water containing enough benzoic acid to give 0·1% w/v concentration of the preservative in the finished product. The mixture was blended for 15 min with a Silverson mixer (Gallenkamp). The emulsion obtained was further passed four times through a table homogenizer (Gallenkamp). Batches of liquid paraffin emulsions were similarly prepared with 12·5% w/v acacia and 1·0% w/v tragacanth respectively. 100 ml quantities of each emulsion were stored at 30 °C.

A globule count was performed on each emulsion 24 h and eight weeks after preparation. One exception was a batch prepared with 0·8% w/v of mucuna gum which was aged for four months. Dilution of

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the emulsions and the subsequent globule count was as described by Cockton & Wynn (1952). A drop of the thoroughly mixed and diluted emulsions was placed under the cover slip on a haemocytometer (BSS 748, Hawksley and Sons), each small chamber of which provided a volume of $2{\cdot}5 \times 10^{-4}$ mm^3. The staining of the aqueous phase with nigrosin permitted easy observation of the oil globules. The globules in 256 small squares were counted and the number N, derived from each mm^3 of oil and the rate of globule coalescence in each emulsion during the eight weeks of storage were determined from the globule count.

Suspending ability and suspension stability

Suspensions containing 16% w/v of zinc oxide or 10% w/v of sulphadimide were prepared using either mucuna gum or tragacanth in concentrations of 0·5, 1·0, 1·2 and 1·5% w/v. The required amount of the drug in fine powder was placed in the appropriate volume of the preserved mucilage and blended for 5 min with the Silverson mixer. This was further passed once through the table homogenizer. All suspensions were deaerated by negative suction pressure. A 100 ml volume of each suspension provided a height of 18·10 cm in a glass cylinder of internal diameter, 2·66 cm. This was stoppered and stored at 30 °C in a vibration-free compartment. The height of sedimented solid was observed by means of a cathetometer and recorded daily for the first week and weekly thereafter.

RESULTS

The number of globules, N, derived from a mm^3 of oil was calculated using the following expression (Cockton & Wynn 1952),

$$N = \frac{c}{256} \times f \times \frac{100}{p} \times \frac{10^4}{2{\cdot}5}$$

where c is the total count of globules, f the dilution factor of the emulsion and p the percent of oil in the emulsion. The root mean cube diameters, d of the globules at 24 h and after storage was calculated from

$$d \times 10^3 \sqrt{\frac{6}{\Pi N}}$$

The rate of globule coalescence, k was calculated from the expression (Sherman 1963)

$$N_t = N_0 \exp(-kt)$$

where N_0 and N_t are the numbers of particles per unit volume at zero time and time t. The changes in the globule sizes of the emulsions were determined from the relationship (Sherman 1963)

$$\ln D_t = \ln D_0 + \frac{kt}{3}$$

where D_t and D_0 are globule sizes at zero time and time t; k is the rate of coalescence and t is the time of storage. In all computations, 24 h was equated to zero time.

Table 1 shows the calculated values of N, k and d derived from the emulsions.

Carstensen & Su (1970) demonstrated that the sedimentation pattern of flocculated suspensions consist of an initial phase and a final phase. They found that the early decay pattern of the final phase may be used to predict the final sedimentation height long before it is attained. The suspensions of sulphadimidine and zinc oxide prepared with these gums were assumed to sediment in a manner characteristic of the final phase of decay. The changes in the sedimentation height during the first week of storage were used for predicting the final sedimentation, H_u. This was calculated for each type of emulsion by an iterative procedure in which $\log(x - H_u)$ vs time in days was plotted using selected values of H_u. The average sedimentation height of duplicates is represented by x. Tables 2(a) and (b) illustrate the data used for predicting the various H_u values. The numerical value that gives the best straight line (Fig. 1) is the expected H_u value. The use of H_u (7·00 cm) predicted for sulphadimidine suspended

Table 1. Total count, mean cube diameter and rate of coalescence of globules in freshly prepared and aged emulsions.

Gum and dilution factor		Total count c		Globule mm^{-3} ($\times 10^6$)		Mean cube diameter (d (μm)		Rate of coalescence k (day^{-1})
		t_o	t	N_o	N_t	d_o	d_t	
Mucuna	1 : 200	316	79	2·40	0·62	9·18	14·50	0·023
Tragacanth	1 : 100	138	96	0·54	0·37	15·25	17·20	0·006
Acacia	1 : 1000	1043	559	40·63	21·84	3·61	4·44	0·010

Table 2. The sedimentation height at different days showing values of H_u for graphical prediction of final sedimentation height.

(a) Sulphadimidine in 1·0% mucuna gum mucilage

Day	Sediment. hght x(cm)	log $(x-3·5)$	log $(x-3·7)$	log $(x-4)$
1	7·40	0·59	0·57	0·53
2	7·20	0·57	0·54	0·51
3	7·00	0·54	0·52	0·48
4	6·80	0·52	0·49	0·45
5	6·60	0·49	0·46	0·41
6	6·40	0·46	0·43	0·38
7	6·20	0·43	0·40	0·34
49	4·00*			

(b) Zinc oxide in 1·0% mucuna gum mucilage

Day	Sediment. hght x(cm)	log $(x-4·5)$	log $(x-5·0)$	log $(x-4·75)$
1	9·40	0·69	0·64	0·67
2	9·00	0·65	0·60	0·63
3	8·80	0·63	0·58	0·61
4	8·60	0·61	0·56	0·59
5	8·50	0·60	0·54	0·57
6	8·40	0·59	0·53	0·56
7	8·40	8·59	0·53	0·56
49	4·00*			

* Final experimental sedimentation height.

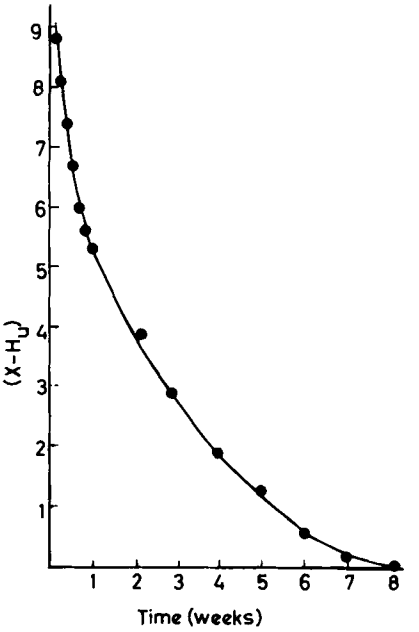

FIG. 2. The biexponential pattern of 10% sulphadimidine suspended in 1·2% mucuna; predicted H_u = 7·00 cm.

with 1·2% w/v mucuna gum and a plot of $x - H_u$ vs time (weeks) yields the graph shown in Fig. 2. Suspensions prepared with 1·5% of mucuna gum or 1·0% gum tragacanth showed little or no sedimentation but nevertheless showed some separation, as in other suspensions.

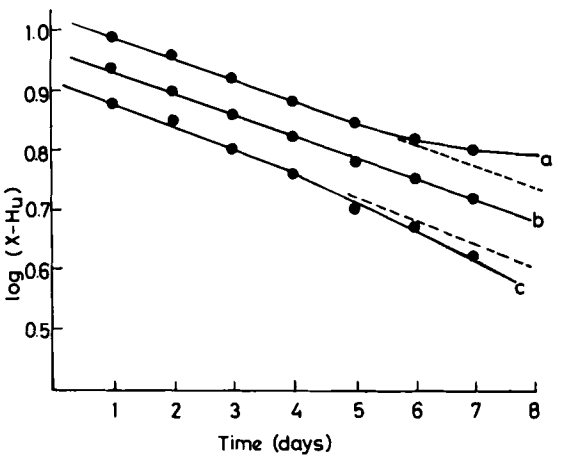

FIG. 1. The graphical prediction of the final sedimentation height, H_u of 10% sulphadimidine 1·2% mucuna mucilage: a = 6·00 cm; b = 7·00 cm and c = 8·00 cm.

DISCUSSION

Preliminary experiments indicated that an acid pH prevents the darkening in mucuna preparations. Benzoic acid was therefore used not only to preserve but also to stabilize these preparations. Acceptable emulsions resulted from the use of 4:6 oil–water ratio. All emulsions were subjected to the same processing. Although the emulsions prepared with 0·8% w/v of the mucuna gum were stable for four months and showed only slight creaming, test emulsions were prepared with 1·0% w/v of the gum. Those emulsions prepared with either 12·5% acacia or 1·0% of gum tragacanth provided the basis for comparison. Globule count performed on control emulsions aged for varying times were used for calculating the rate of coalescence, k, which, in turn was used for determining unknown initial or final globule sizes after storage periods longer than the test period of eight weeks. The close agreement with the experimentally determined globule sizes showed that the application of Sherman's equation to the system was justified.

Martin et al (1973) suggested that the most important factor in the stabilization of an emulsion is the physical property of the emulsifier at the interface. The order of magnitude of the rate of coalescence k, is mucuna > acacia > tragacanth

(Table 1). On the basis of this alone, the emulsion prepared with mucuna would seem to be the least stable. This was not borne out by the result of the storage test since the emulsion prepared with acacia creamed within this period. The emulsion prepared with acacia also had the smallest globule size (Table 1). The order of magnitude of the globule size is acacia < mucuna < tragacanth. An increase in emulsifier concentration reduces the globule size of an emulsion formulation (Rowe 1965) and a decrease in globule size should enhance emulsion stability. However, small particles have inherent instability and tend to mutually coalesce or coalesce with large particles (Sherman 1970). This and the possible weakness of the acacia film at the interface would account for the more rapid creaming in emulsions prepared with acacia.

The best suspending medium for a solid will permit the least sedimentation. Fig. 1 and Table 2 show the predicted sedimentation height H_u, of the suspensions. Zinc oxide suspended in 1·2% mucuna mucilage showed little or no sedimentation. The H_u value of 7·00 cm for sulphadimidine in 1·2% mucuna mucilage is about twice that for the same drug (3·7 cm) in a 1·0% mucilage of the same gum. This increase, brought about by a 0·2% difference in the concentration of the mucuna gum, could be accounted for by both viscosity changes and increased flocculation.

In general, higher concentrations of the gums reduced the rate and the extent of separation in the flocculated suspensions. This is essentially similar to the results of Kabre et al (1964). However, the sulphadimidine suspension prepared with 1·0% tragacanth showed a greater separation than zinc oxide suspended with the same concentration of tragacanth. Zinc oxide suspended in 1·2% mucuna mucilage showed less separation than sulphadimidine similarly suspended. Martin et al (1973), observed that a greater rate of separation may be present in highly flocculated suspensions. However, the suspensions prepared with higher concentrations of the gums (1·5%) showed little or no separation. This is not surprising because the high apparent viscosity resulting from the increased concentration of the gums will retard sedimentation of the flocs.

Conclusion

Carstensen & Su (1970) directed their investigation to the role of interparticular forces and the effect of such forces in the sedimentation of suspensions of moderate concentration. The present investigation shows that their technique is useful for a rapid evaluation of suspending agents.

Stable zinc oxide suspensions can be prepared with 1·2% of mucuna gum while sulphadimidine requires up to 1·5% of the new suspending agent. The new gum has obvious economic advantage in that the plant source can be cultivated several times in a season. Acacia mucilage is not very viscous and therefore it is not much used in suspensions. The tragacanth mucilage is more viscous but shows a rapid drop in viscosity during storage at a pH below 4·5. The mucuna mucilage has a comparatively high viscosity and it is stable at a pH below 4·5. Its stabilization by lowering of pH indicates that it can tolerate the presence of organic acids which may be necessary in some formulations. It may, therefore, prove to be the suspending agent of choice when a high viscosity and tolerance to acid pH are desired. The thixotropic character of the stored suspensions prepared with mucuna is a desirable property in suspending agents. At a concentration of 1·0%, the new gum yields a more stable paraffin emulsion than 12·5% acacia. The stability and nature of emulsions prepared with mucuna gum are similar to those prepared with tragacanth.

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