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Soft-enCapsulation of the Elastovisco-Plastic Materials

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Abstract— Soft-encapsulation is a process in which tiny particles or droplets are surrounded by a coating to give small capsules of many useful properties. In general, it is used to enclose solids, liquids, or gases inside a micrometric wall made of hard or soft soluble film, in order to reduce dosing frequency and prevent the degradation of pharmaceuticals and other Elastovisco-plastic materials. The reasons for Soft-encapsulation are countless. It is mainly used to increase the stability and life of the product being encapsulated, facilitate the manipulation of the product and control its liberation in an adequate time and space. There are more than 2000 varieties of soft-encapsulation available around the world; however they are often classified into twelve basic categories. Out of these twelve, three main categories have been selected for the present research studies. These categories were selected on the basis of their common texture classifications; very-hard, hard, and soft as well as their moisture and fat contents. The properties are reported based upon studies using indentation experimentation involving loading-unloading, and stress relaxation. The conventional indentation hardness values ranged from hard to soft. The hardness is universally proportional to the moisture content.

Index Terms— Elasticity, Materials Characterisation, soft solids, viscosity, plasticity, Drying, Encapsulation & Relaxation

1 INTRODUCTION

IN various fields of science and technology there arise problems associated with the determination and control of the characteristics of small particles; their sizes, shapes, degree of aggregation, and concentration as in [1]. Similarly, in environmental science, a pesticide may be soft-encapsulated to minimize leaching or volatilization risks. At the same time large variety of natural materials and modern products occur in the form of soft solids are represented in fig.1. These include muds, clays, pastes, foams, powders and many more foodstuffs. Apparently, Soft solids have also become the medium of choice for a variety of processing operations as in [9].

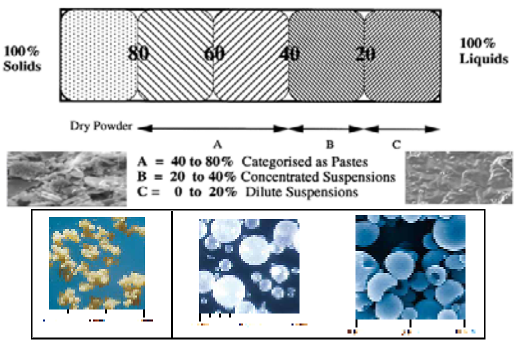

Fig. 1; Transitions from liquid to solids vice-versa.

Soft-encapsulation is a process in which tiny particles or droplets are surrounded by a coating to give small capsules of many useful properties. In general, it is used to incorporate food ingredients, enzymes, cells or other materials on a *micro metric scale*. Soft-encapsulation can also be used to enclose solids, liquids, or gases inside a micrometric wall made of hard or soft soluble film, in order to reduce dosing frequency and prevent the degradation of pharmaceuticals and other Elstovisco-plastic materials as in [10]. The reasons for Soft-encapsulation are countless. It is mainly used to increase the stability and life of the product being encapsulated, facilitate the manipulation of the product and control its liberation in an adequate time and space.

Fig2; Commercially available liquids & solids.

In some cases, the core must be isolated from its surroundings, as in isolating vitamins from the deteriorating effects of oxygen, retarding evaporation of a volatile core, improving the handling properties of a sticky material, or isolating a reactive core from chemical attack as represented in fig.2. In other cases, the objective is not to isolate the core completely but to control the rate at which it leaves the soft-capsule, as in controlled release of drugs or pesticides as in [2].

Spray drying serves as a soft-encapsulation technique when an active material is dissolved or suspended in a melt or polymer solution and becomes trapped in the dried particle. The problem may be as simple as masking the taste or odor of the core, or as complex as increasing the selectivity of an adsorption or extraction process. The main advantages are the ability to handle labile materials because of the short contact time in the dryer and in addition, the operation is economical. In modern spray dryers the viscosity of the solutions to be sprayed can be as high as 300 mPa, a dryer's flow chart is shown in fig.3. An encapsulation of functional additives for nano foods like fruit aromas, flavours, and perfumes.

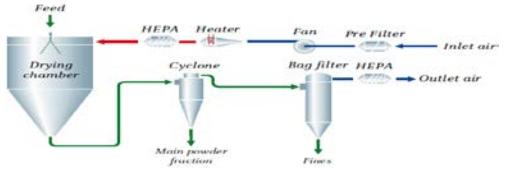

Fig.3; Flow diagram of Spray drying process.

In drying, water is usually removed by circulating air over the material in order to carry away the water vapour, while in evaporation; water is removed from the material as pure water vapour mixed with other gases.

2 MATERIALS and METHODOLOGY

In the work presented here, we have used a simple cone indentation test to characterise a model soft-solid material in terms of both its elastic and viscous properties. The sophisticated levels of these theoretical analyses and the level of understanding needed for routine measurements of material properties for quality control and other production procedures.

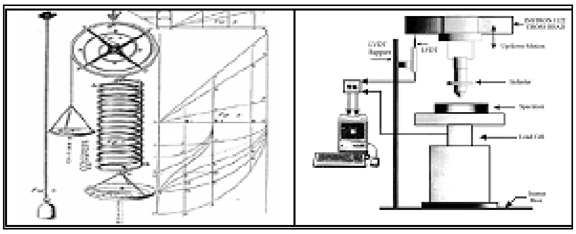

Fig.; 4. The Hook's law & indentation geometry.

The principal device used was a standard universal-testing machine (Instron 1122, Instron Ltd. High Wycombe, UK), equipped with a 50N transducer (bottom load cell) of accuracy ±0.01N. The reaction load was recorded at suitable intervals automatically using analogue sensors. Force and corresponding time data were recorded simultaneously by a host computer, via a terminal panel T31 (analogue to digital converter) connected to Instron interface as shown in fig.4.

A series of experiments has been performed in which cone included angles from 30º to 90º have been indented into a large block of a model soft-solid at a range of constant velocities. The indentations all proceeded at variable speed to a constant depth at which time the cones was fixed and the stress relaxation was observed. In all cases, the stress relaxation is very rapid at first, followed by a long gradually declining tail. Soft-Solids tend to flow their Stress depends on Strain rate like Pastes; Foods, Ceramics, Clays (terracotta), Concrete, Plasticine, .Powders, Gels, Emulsions, Foams, Paints. Hard-Solids tend to fracture (tough, brittle) their Stress depends on Strain; for examples metals, ceramics, and glassy polymers.

The initial data were obtained in the form of time series of load, and the corresponding displacement. Fig.5 shows typical examples of loading and unloading curves, where the total load (N) for the constant depth is plotted against the indentation displacement (mm).

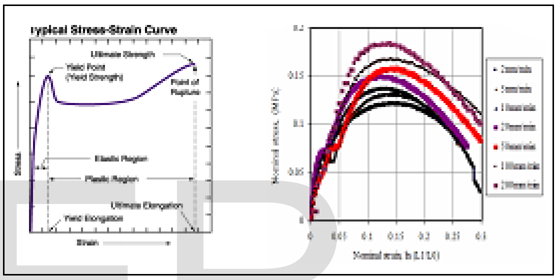

Fig. 5; Typical sets of data for load vs. Time.

The curves indicate that there is a little elastic recovery of the material during unloading, which tends to increase as the indentation rate is increased.

3 RESULTS & DISCUSSIONS

Drying is the final removal of water from material (usually by heat), non-thermal drying, as squeezing wetted sponge and adsorption by desiccant (desiccation) and then extraction, the process flow diagram can be seen in fig. 6.

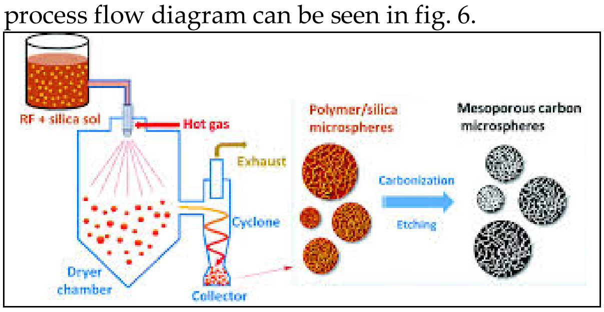

Fig.6; The drying process at micro & macro levels.

In pharmaceutical industries, drying is carried out for one or more of the following reasons: To avoid or eliminate moisture which may lead to corrosion and decrease the product or drug stability at different temperatures ranges, are shown in fig. 7. It clearly indicates their temperature dependencies on evaporation (kg/h) at gas rates

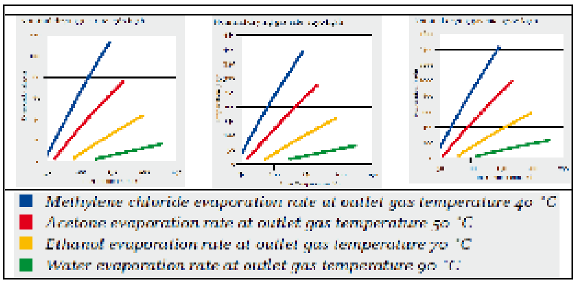

Fig. 7; Temperature dependencies on evaporation (kg/h) at different gas rates.

3.1 MATHEMATICAL INTERPRETATIONS

The hardness (plastic component), H, is commonly defined as [7];

$$H = \frac{L}{A} = \frac{L}{\pi a^2} \qquad (1)$$

where L is the reaction force, A is the contact area, projected onto the original surface, and a is the corresponding radius for axially symmetric indentations. The actual contact radius is distinguished from the apparent radius of contact, which corresponds to the value computed from the total depth of penetration, h_t.

The reduced elastic modulus, E^*, is conventionally obtained from the elastic contact stiffness of the initial part of the unloading curve, as in [3], Fig.5;

$$\frac{\partial L}{\partial h} = S = 2E^* a \qquad (2)$$

where $E^* = \frac{E}{(1-\nu^2)}$, E is the Young's modulus, ν is Poisson's ratio and S is the contact stiffness upon unloading at the maximum penetration depth, h_t. as in [4].

for the cone, as: $a = h \tan \theta$ *(3)*

where θ is the semi-included angle of the cone, R is the radius of the sphere and h is the depth of indentation, which can be represented as $h = V(t-t_0)$, V is the penetration velocity, t_0 is the time zero offset and $(t-t_0)$ is the apparent time of the contact between the indenter and the specimen as in [8].

In the case of indentation, the plasticity index can be usefully expressed as the ratio of the elastic component of the work done to the plastic component of the work done. Using the parametric relationship for the loading curves, the plasticity index can be approximated in terms of the hardness and the reduced elastic modulus.

$$\psi = \tan\beta \frac{E^*}{H} \qquad (4)$$

where β is the angle of inclination of the indenter to the sample surface and E^* is again the reduced elastic modulus.

An elasto-viscoplastic model for indentation is shown in Fig. 4, which comprise of the conventional elastic and plastic descriptors. If the stress is removed after some time, the spring, no longer constrained, returns to its original length immediately, but that part of the displacement due to the dashpot creep remains. As long as a material is uniform, it can be expected that the nominal stress, σ, is related to some nominal strain rate $\dot{\gamma}$ by a simple relationship.

$$\sigma = C_P F(\dot{\gamma}). \qquad (5)$$

where C_P is the plastic constraint factor (a term to include geometric factors and interface friction) and F is a non-linear material function. Similarly, for an essentially elastic response to indentation, the nominal stress would be related to some nominal strain ε by a simple relationship.

$$\sigma = C_E E^* \varepsilon \qquad (6)$$

where C_E is the elastic constraint factor which also, like C_P, embodies geometrical and frictional effects, E^* is an effective elastic modulus.

It is supposed that, during the indentation phase, the elastic component of the strain is small, so that an approximate relationship for the mean indentation pressure is obtained as:

$$\sigma = \frac{L}{A} = C_P F\left(\frac{V}{a}\right) \qquad (7)$$

where V is the indentation velocity.

However, during the relaxation phase, the elastic stress is equally important, and a relationship for the indentation pressure is given as;

$$\sigma = \frac{L}{A} = C_P F\left(\frac{-\dot{P}}{A C_E E^*}\right) \qquad (8)$$

where L is the load registered, and $\left(-\dot{L}\right)$ is the rate of relaxation of the load. The equation (8) provides quantitative values of the power law index q and the intercept k, as in [5].

$$\sigma = -k\left(\dot{L}\right)q \qquad (9)$$

The data were analysed using equations (8) and (9) for the indentation and relaxation phases. According to equation (8) plots of mean indentation pressure against nominal strain rate for different speeds should collapse to a single master curve. Similarly, though less familiar, equation (9) indicates that the mean indentation pressure in the relaxation phase is a function of the rate of decrease of the load. Collections of such plots for the cone indenters are shown in Fig. 8.

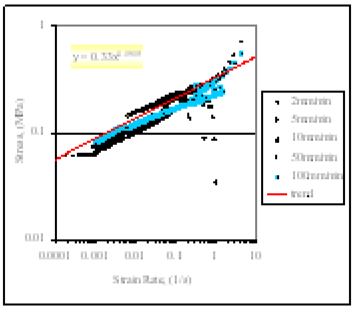

Fig. 8. Mean indentation pressure: (a) vs. nominal strain rate; (b) vs. rate of change of load. Each line represents a fixed indentation velocity.

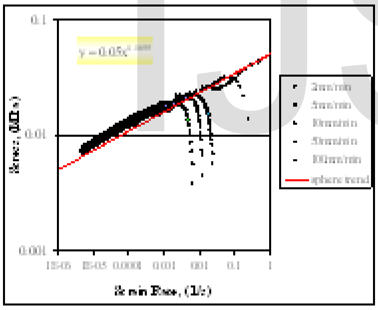

Fig. 9; Combined plot of data from indentation and relaxation.

The forms of the relationships (8) and (9) both show the shape of the viscosity function F. By choosing an appropriate value for the product of the elastic modulus and constraint factor, the two sets of data in Fig.5 & Fig 8 should be combined, for obtaining material indices is shown in Fig. 9

4 CONCLUSION

Spray drying is a method of producing a dry powder from a liquid or slurry by rapidly drying with a hot gas. This is the preferred method of drying of many thermally-sensitive materials such as foods and pharmaceuticals. Drying in most of the cases means the removal of relatively small amounts of water from solids. Online measurement for the study and optimization of the processing of dense paste was possible without sample dilution in contrast to optic methods.

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