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EXPERIMENTAL ANALYSIS OF THE DIFFERENT PHASES IN THE DRYING OF NATURAL RUBBER



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1. INTRODUCTION

Latex has the aspect of a rubber particle dispersion in a water serum containing organic and mineral elements in solution. Its content in solid matter is approximately 30 to 40%, 90% of which being formed of rubber particles. The remainder is made up of non-rubber elements (proteins, sugars, etc.) [1]. The composition of latex is complex and prone to changes linked to various parameters: clonal characteristics, climatic influence, etc. From the latex collection on plantation to the industrial use under its various forms, numerous processes are involved to obtain a finished product. Drying is one of these intermediate processes; it is a delicate operation which greatly affects the product's quality.

To date, there has been little work carried out on the description of the internal structure and the mechanisms brought into play during the drying stage. The purpose of this study is to analyze serum transfers (water+solutes) in a natural rubber sample with three experimental methods:

- 1) kinetics of drying; 2) measurement during the kinetic study, of the temperature difference between the sample and that of the drying atmosphere; 3) measurement of the freezing point temperature of the serum contained in the product at different stages of the drying kinetics.

2. EXPERIMENTATION

2.1 Sample preparation

The latex used, whose dry rubber content (DRC) is 37%, comes from the Ivory Coast. Latex is then diluted with water in order to obtain a 15% reference DRC. Coagulation is obtained by adding a quantity of acetic acid with a 4.8 PH. The coagulum obtained requires a maturation period of about 20 hours during which a rubber particle partial solidification takes place. The coagulum is then laminated and washed to obtain an average 10 mm thick sheet, from which will be taken a 38 mm diameter sample with a punch. Before coagulation a thermocouple is inserted at the center of the sample.

2.2 Experimental device description

The measurement container consisting in a

beaker is located in a thermostable bath (Fig.1) thermo-regulated with an immersed thermostat. Partial pressure within the beaker is maintained steady with aqueous sulfuric acid. The thermocouple placed in the sample measures the Tf temperature, a second thermocouple located close to the sample measures the temperature of the drying air Ta.

2.3 Operating procedure

The sample is placed in the measurement container. As the experiment proceeds the sample mass and Ta and Tf temperatures are measured. At regular time intervals freezing trials are performed. The sample is taken out of the measurement container and placed in a refrigerating system at -28°C, meanwhile the thermocouple at the center of the sample measures its temperature progress. Prior to and following the freezing operation, weighings were taken to check that no significant mass loss occurred as a result. The freezing operation which lasts 20 minutes on average does not significantly alter the sample drying process. The experiments were performed under the following conditions: Ta = 49°C; relative humidity of the air hr=47%. At the end of the experiment, the sample reaches its steady state mass, it is then thoroughly dried above a concentrated sulfuric acid solution forcing the relative humidity of the air to be lower than 0.01%.

3. EXPERIMENTAL RESULT ANALYSIS

3.1 Drying kinetics. Variation of the temperature difference between the drying air and the sample

Figure 2 shows the kinetics of drying of the rubber sample as well as the temperature difference between the dry air Ta and the sample Tf, over time. Two drying periods can be observed from the water content changes; the first, called "constant rate period", between 80% and 33%; the second between 33% and 2.5% during which the drying speed markedly slows down is referred to as the diffusion period [2], [3]. The Ta-Tf difference over time confirms that two drying periods occur. Once the product temperature is set, Ta-Tf temperature deviation stabilizes at 9.8°C for a constant rate drying period then decreases for a diffusion period until reaching a low value (Ta-Tf = 0.3°C). During the constant

rate period, the sample surface is saturated with serum, a heat and mass transfer balance occurs on the product surface. To this balance corresponds a wet air temperature T_w which for the above mentioned air characteristics is 38°C ; this temperature is close to the T_f value measured ($T_f = 38,7^\circ\text{C}$).

3.2 Analysis of the freezing point temperature lowering of the serum contained in the sample

Figure 3 shows the refrigerating temperature variations over time of the sample at different drying moments. Two curve trends can be observed. The first which corresponds to curves C_1 to C_4 is characterized by temperature levelings, the freezing temperature is practically steady and equal to $-4,2^\circ\text{C}$. The second one (curves C_5 and C_6) is characterized by temperature rises linked to the appearance of overfusion. These rises progressively level off due to the water content decrease of the sample. For a water content below 7%, no characteristic signal making it possible to detect the change from the liquid to solid state of serum can be observed (C_{20}). Overfusion appears for a water content included between 53% and 33%. Overfusion seems thus to determine the limit between the two mass transfer mechanisms noticed in figure 2. This transition could be associated with a poral space shrinking on the sample surface, pores taking the so called "ink bottle" shape which favours the appearance of overfusion [4].

3.3 Comparison of the three methods.

Figure 4 shows a synthesis of three analysis methods. According to the water content, the drying rate dw/dt , the $T_a - T_f$ temperature difference and the lowering of the freezing point, $\theta = T_0 - T$ ($T_0 = 273^\circ\text{K}$, T being the freezing temperature of serum), are shown in this figure which also indicates the three methods concur in locating at about 40% the water content during the transition from a constant rate period drying to diffusion drying.

3.4 Analysis of the solute concentration variation of serum through cryometrics

The lowering of the freezing point of the θ serum contained in rubber can be due to the presence of solutes and to the poral space structure. If θ_1 is the contribution due to the presence of solutes and the θ_2 that due to the poral space, then:

$$\theta = \theta_1 + \theta_2 \quad (1)$$

On the supposition the serum behaves as an ideal diluted solution [5]:

$$\theta_1 = \frac{RT_0^2}{L_0} \sum m_s \quad (2)$$

Where R is the constant of perfect gases, T_0 the freezing temperature of pure water, L_0 the latent freezing heat, M_0 the molar mass of pure water, m_s the solution molality.

Measurements taken on the free serum

recovered during syneresis, showed that the lowering of the freezing point is, in this case, approximately 4°C . This value being more or less equal to the lowering of the freezing point of the serum contained in the sample during the constant rate period, the influence of the poral space, during this phase, can be considered negligible: $\theta_2 \approx 0$. In these conditions, the relation (2) becomes:

$$n_s^* = \frac{L_0}{R T_0^2 M_0} \theta_1 w \quad (3)$$

Where n_s^* is the mole number of solutes per kilograms of dry rubber.

Figure 5 shows the n_s^* variation according to w . For water contents greater than approximately 40%, a line passing through the origin is obtained, thus verifying relation (3). During constant rate drying, the concentration of solutes in the serum within rubber, remains constant, thus explaining a mass transfer of serum towards the surface. The experiment deviation when compared to relation (3) during the diffusion phase ($w < 40\%$) can be explained in three ways: 1) poral space influence ($\theta_2 \neq 0$), 2) trapping of the solutes in structure, 3) water evaporation and transfer into gaseous phase towards the surface, which would be accompanied by an enrichment of the serum in solutes. Certain studies [2] tend to show that rubber remains saturated in liquid phase during the whole drying period, which would entail the elimination of the third hypothesis. The current research aims to assess all three hypotheses in order to describe transfers during the diffusion phase.

4. CONCLUSION

The three experimental methods set in operation clearly show two drying phases of rubber characterized by very different transfer mechanisms are well understood during the constant rate period, those occurring during the diffusion phase are still to be better defined.

REFERENCES

- [1]. LE BRAS J., Elements de Sciences et Technologie du Caoutchouc, 1951.
- [2]. GALE R.S., A survey factors involved in an experimental study of the drying of sheet rubber. J. Rubb. Res. Inst. Malaya, 16, 38, 1959.
- [3]. GALE R.S., Drying of sheet rubber in the falling rate period. Trans. Instn. Chem. Engrs., 15, 150, 1962.
- [4]. HOMSHAW L.G., L'eau et les solutés dans les milieux poreux: Etude par microcalorimétrie à basse température de l'effet des surfaces et de la porosité, Thèse de Doc-Ing., Université de Paris VII, 1980.
- [5]. GUGGENHEIM E.A., Thermodynamique, Dunod, Paris, 1965.

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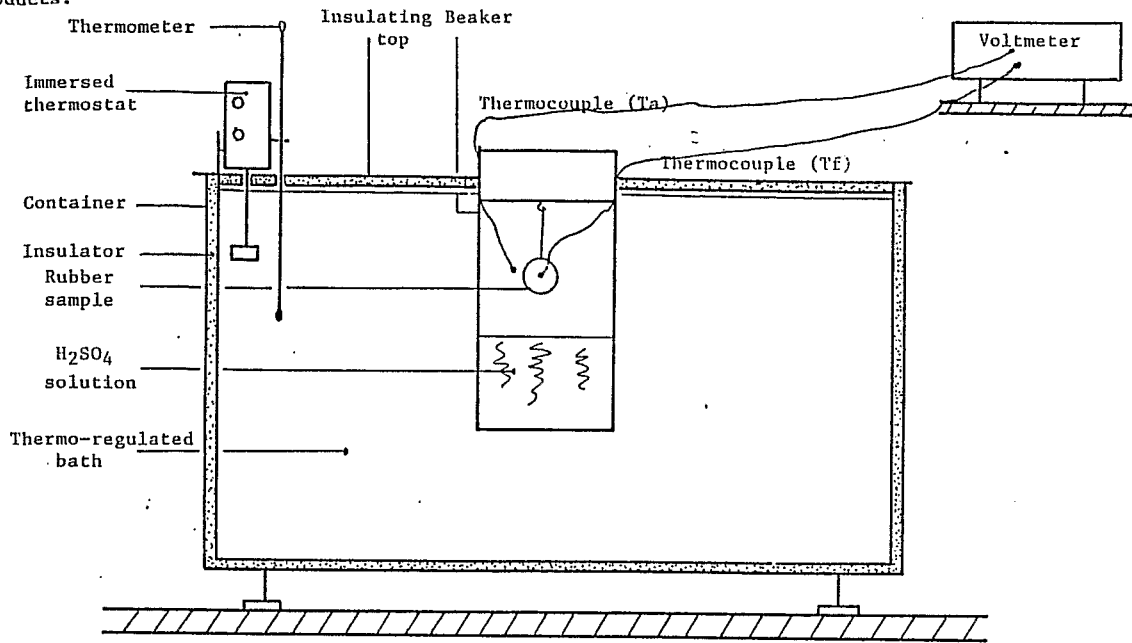


Figure 1: Diagram showing the rubber sample drying experimental device.

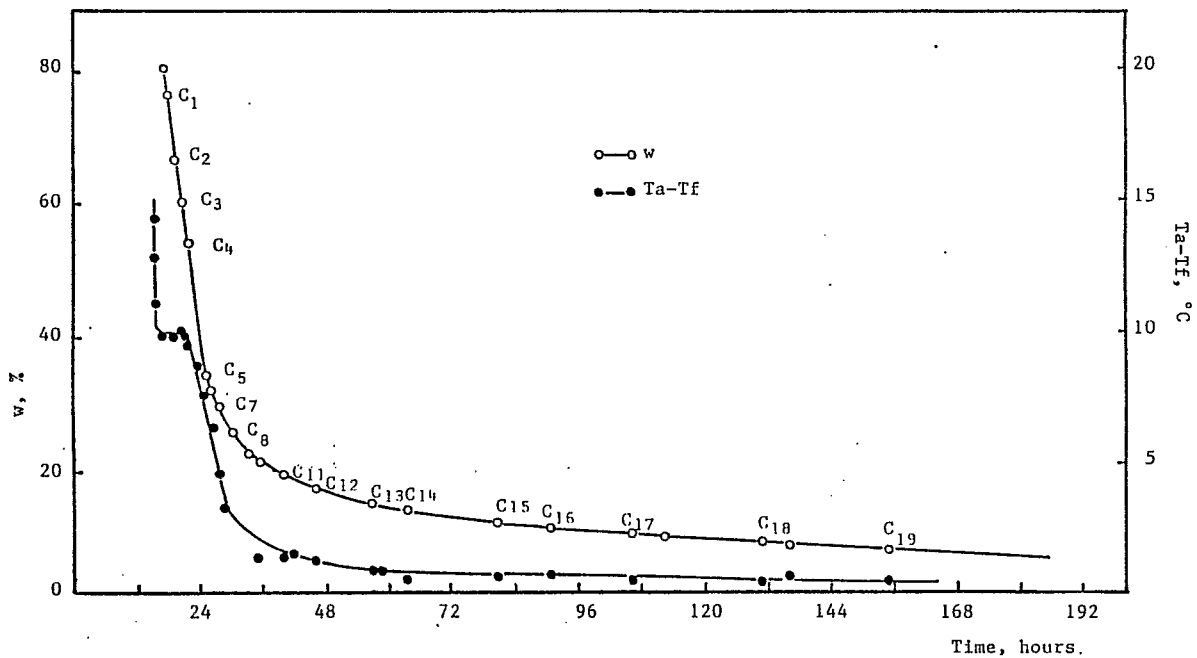


Figure 2: Variations of the sample water content and the temperature difference between the drying air and the sample over time. The subscript letter C_i identifies the different moments when the sample is taken out for freezing.

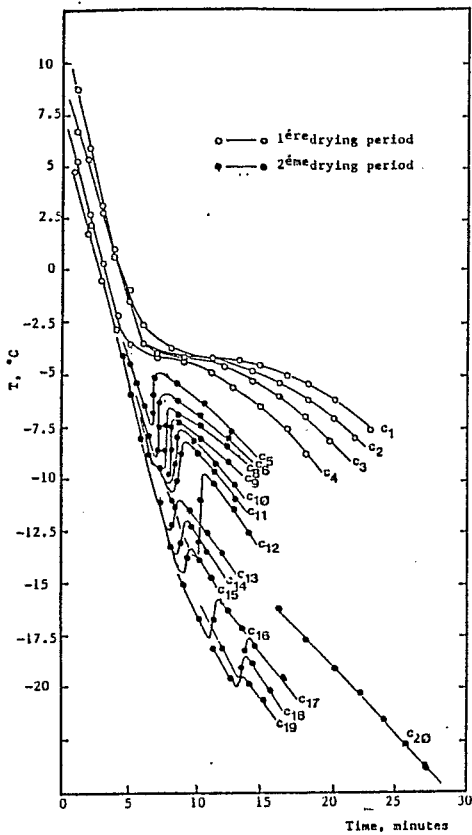


Figure 3: Cooling temperature variation over time at different moments of the drying kinetics of a natural rubber sample. The subscript letters C_i identify the different moments when the sample is taken out for freezing.

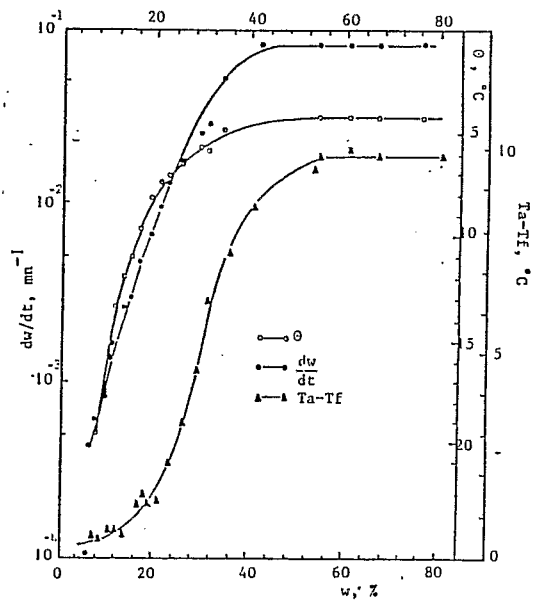


Figure 4: Characteristic curves obtained when drying a natural rubber sample.

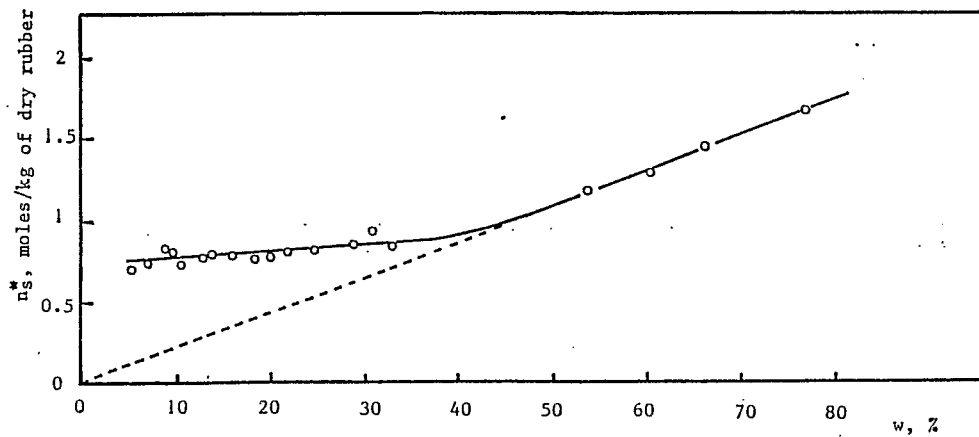


Figure 5: Variation of mole number of solute per kilograms of dry rubber in the sample according to the water content.