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INFLUENCE OF MOISTURE CONTENT ON ADHESION FORCE OF SOME PHYSIOLOGICAL SUGARS

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Abstract

People involved in the cotton industries, from growers to spinners, have become concerned with the problem of stickiness encountered during the cotton processing from fibers to yarns. Some progress has been made with the fundamental knowledge of adhesion of sugar. But now, to progress in the knowledge of stickiness, an accurate method to measure the sugar moisture content is required.

Introduction

In the last 20 years, sticky cottons have substantially disrupted the cotton spinning process. This stickiness has two main causes, physiological and entomological sugars produced respectively, by the cotton plant and by insects. Many studies have been carried out to understand the practical and fundamental mechanisms of stickiness adhesion.

It has been also highlighted that the sugar moisture content is one of the most prominent parameters. The moisture content influences both sugar concentration and viscosity, which induces different sticky behavior. But, in fact, the measurement of moisture content in sugar is not very easy and it is quite difficult to have an accurate method.

In this paper, we present the measurement of moisture content, with the help of *nmr spectroscopy* rather than the *Karl Fischer* method, and the influence of moisture content on viscosity and adhesion of some physiological sugar.

Materials and Methods

Honeydew Cooking

Fructose, glucose, saccharose and melezitose are sugars selected to prepare synthetic honeydews (experimental plan figure 1). The adhesive properties of these honeydews are determined by means of a tack test. In this study, stainless steel is used as the substrate for the first adhesion test (tack). Sugars at different concentrations in water ranging from 60% to 85% ($\mathbf{C}_n$) were cooked, in a closed flask in an oil bath for 2 hours at 100°C, and conditioned at 4 different moistures (H_1, H_2,H_3,H_4 respectively 55% , 65%, 75% and 85%).

The rheological properties of synthetic honeydews are determined by means of rheometer (Bohling, cone plan) (viscosity: η): figures 2 and 3.

M_3 is a mixture of 4 sugars; and corresponds to the entomological honeydew of the aphid *Aphis-gossypii***:** 20% glucose + 25% fructose + 39% saccharose + 16% melezitose. After cooking, a determination of the water content of honeydews was performed. Two quantitative methods were used: Karl Fischer analyses and *nmr* spectroscopy (Nuclear Magnetic Resonance).

Karl Fischer: This dosage is based on chemical reactions combined with electrochemical detection. We had several difficulties with this method: due to honeydew crystallization at high concentrations and problem of honeydew dissolution in Fischer reactive because of its important viscosity. For this reason *nmr* analyses were preferred.

Nuclear Magnetic Resonance: nmr

Nmr spectra were obtained by means of Bruker 400 MHz equipment, Advance series, at ordinary temperature (28°C is 300K), in D_2O ("heavy water"), with a relaxation time of recycling of 10 S so that the magnetization of all the protons disappeared and so that the value of the integration of each proton corresponds to one: 1, with good precision.

We carried out the analysis of the fructose and **M_3** honeydews at several concentrations to proportion the exact quantity of water in these systems. A sample of 0.5 to 0.7g of each honeydew, to which we added the same "heavy water" weight, is stirred during a few hours (4 to 5 hours) to ensure a perfect homogeneity of the mixture before *nmr* analysis. The samples are then placed in special *nmr* tubes and analysed.

Results and Discussion

***nmr* Results**

Results obtained by ^{1}H *nmr* concerning the measurement of water content in synthetic honeydews are given in table 1. *nmr* spectroscopy is a very accurate method (98,4%), which enables us to determine honeydew water concentrations precisely. The results also indicate that preparation protocol of synthetic honeydews is suitable and preserves the chosen water content.

Synthetic Honeydew Conditioning

Synthetic honeydew samples were conditioned in climatic chamber where moisture (HR) and temperature are regulated to observe weight evolution vs. time. In fact, the temperature was kept constant (25°C) and HR was varying from 55% to 85%. Fructose and mixture honeydews, with different water contents from 15% to 40% of water, were conditioned at the four following HR: 55%, 65%, 75% and 85% for 2 to 4 weeks. Table 2 shows results of this analysis and figures 4 and 5 presents the evolution of water content honeydew vs. time for HR 55% and 85%.

Tack Test: Adhesion Energy Measurement

Tack energy is the ability of two materials to resist separation after bringing their surfaces into contact for a short time under a light pressure [1]. It is a well suited quantity to analyse cotton stickiness. Tack test will consist in measuring the force required to extract a probe from a honeydew drop laid on a model surface. The adhesion energy is calculated by integration of the curve of force vs. displacement. For individual sugars (figures 6 and 7), both adhesion energy and maximum separation force decrease with increase of residual water content in artificial honeydews. It can be seen also that at a threshold of 30%, a strong decrease of adhesion occurs. This phenomenon has to be studied more accurately, taking in account the type of individual sugars and mixtures. For **M_3** honeydew (figures 8 and 9), the same phenomena are observed. It seems that the threshold in this case is located again around 30% of residual water content in honeydews.

Conclusions and Outlooks

Tack test seems to be a well-adapted technique to evaluate cotton stickiness. First results show that honeydew stickiness depends strongly on moisture, which seems to be the most relevant parameter, but also on the nature of sugar or compositions of mixtures. It also appears that the adhesive behaviour of a sugar mixture is similar to individual sugar constituting this mixture with quite a difference in adhesion energy values, especially for honeydews of high concentration.

In fact, it seems that **M_3** honeydew is stickier than fructose in these cases (higher adhesion energy). Tack test is considered as a microtest, which allows the study of very small quantities of sugar (a drop) and is closer to stickiness encountered during the cotton spinning. A very accurate determination of honeydew water content has been developed with the help of *nmr* spectroscopy.

Since the water content within the honeydew is the most important parameter regarding its adhesive properties, we will focus our attention on the determination of this water content in future work. Direct adhesion measurements of honeydew drops on model metallic or elastomeric planar surfaces as well as onto cotton fibres will also be performed.

References

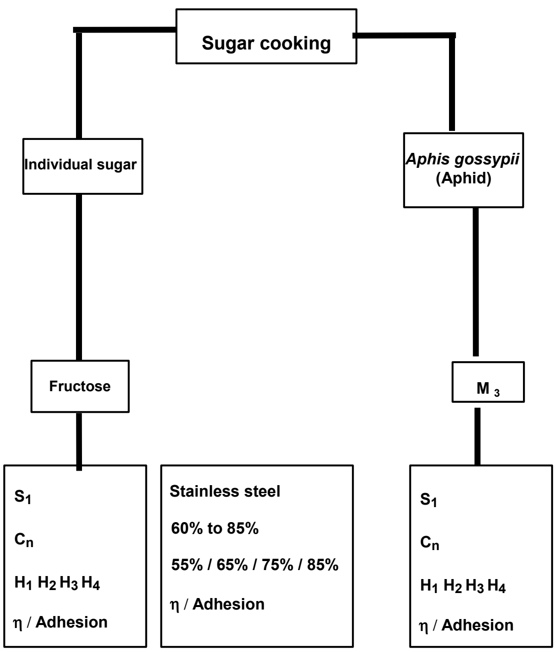
[1] SUNG-SEEN-CHOI and JH. JANG, 1988, “Testing the rolling tack of pressure-sensitive adhesive materials. Part I. Novel method and apparatus” J.Polymer, 39:pp5861-5866.

Table 1: Validation of the determination of synthetic honeydews concentration by ^{1}H *nmr*.

Nature of honeydew	$C_{initial}$	% $H_20_{initial}$	C_{final} measured via *nmr*
F_60	60%	40	61.5
F_65	65%	35	66
F_70	70%	30	71.1
F_75	75%	25	75.2
F_80	80%	20	80.7
F_85	85%	15	86
M3_60	60	40	61.6
M3_65	65	35	66
M3_70	70	30	71.7
M3_75	75	25	75.8
M3_60	80	20	80.9

Table 2: Honeydew concentration after conditioning.

Nature of honeydew	Moisture HR %	After conditioning	
		Water Content (equilibrium)	Honeydew concentration (equilibrium)
Fructose	55%	14	86
M_3	55%	13	87
Fructose	65%	15	85
M_3	65%	16	84
Fructose	75%	23	77
M_3	75%	21	79
Fructose	85%	33	66
M_3	85%	31	69



Legend:

$S_{1:}$ Surface

C_n: Concentration

$M_{3:}$ Mixture of 4 sugars corresponds to the entomological honeydew of the aphid: *Aphis-gossypii*

$H_{1...}$ $H_{4:}$ Moistures(HR)

η : Viscosity.

Figure 1: Experimental plan.

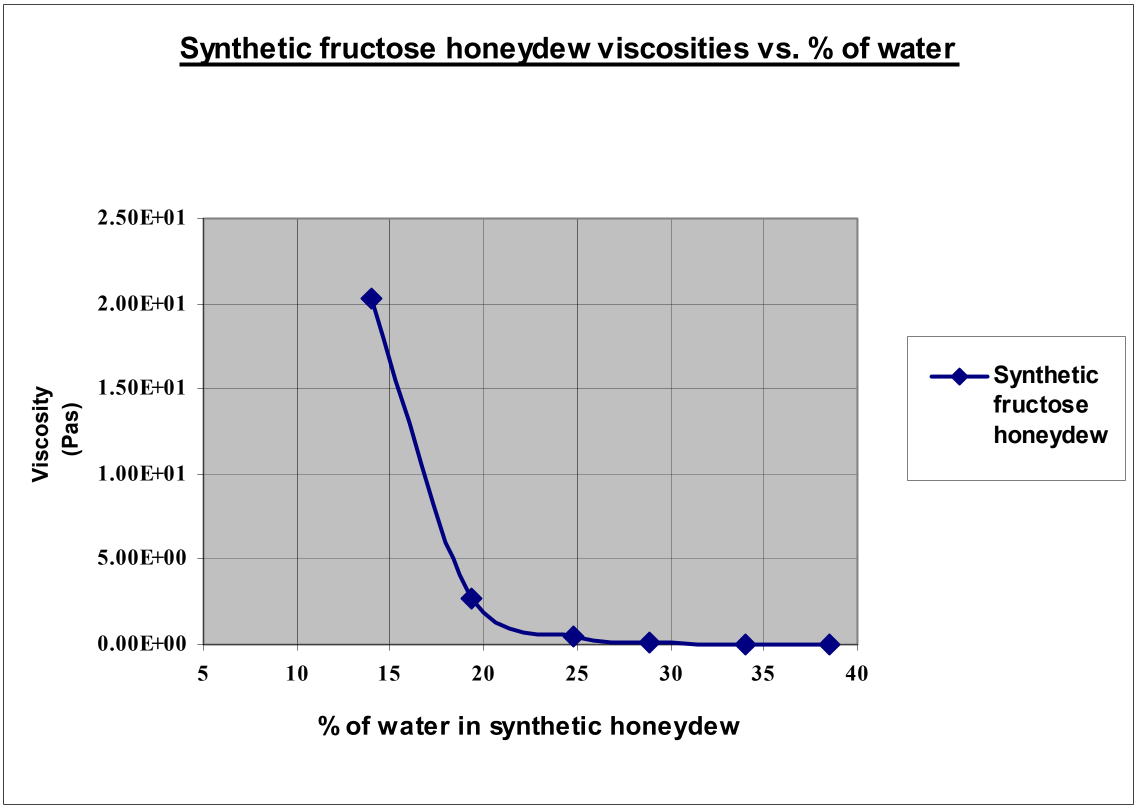


Figure 2: Viscosity vs % of water.

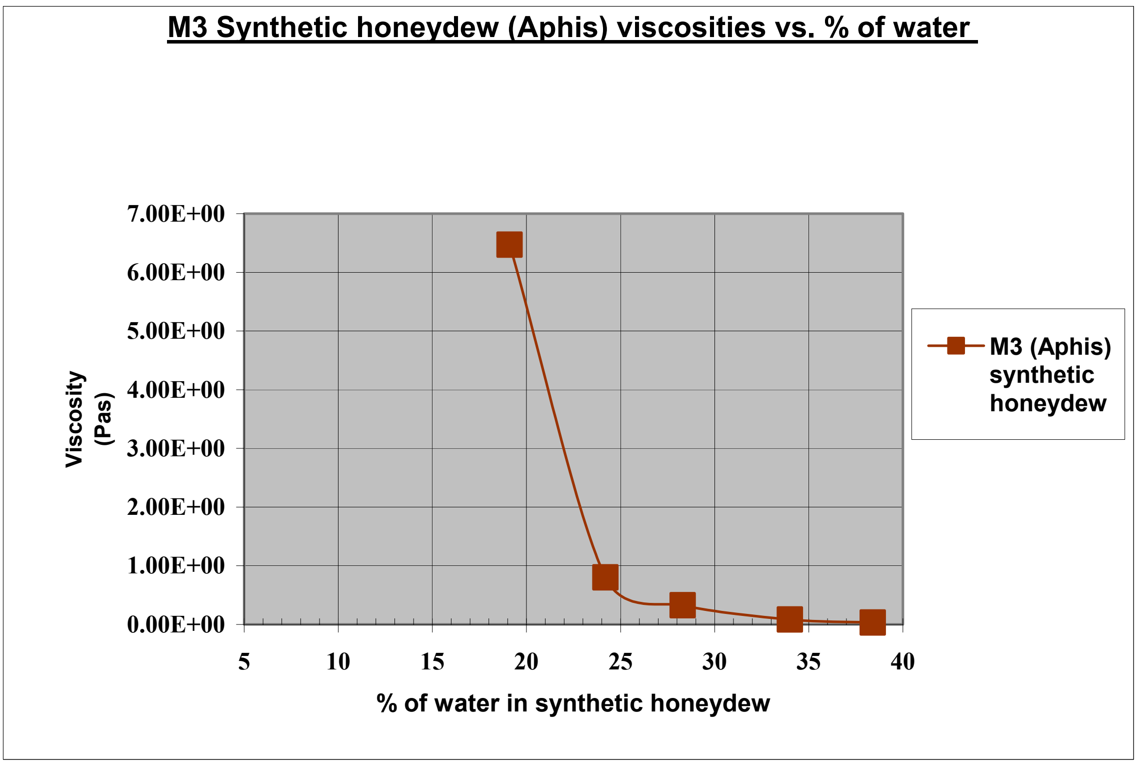


Figure 3: Viscosity vs % of water.

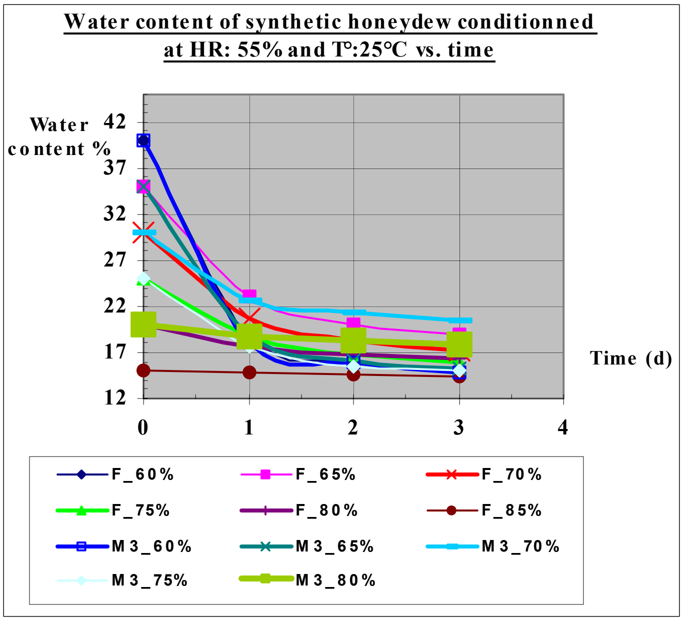


Figure 4: Water content of synthetic honeydews conditioned at HR: 55% and T°:25°C.

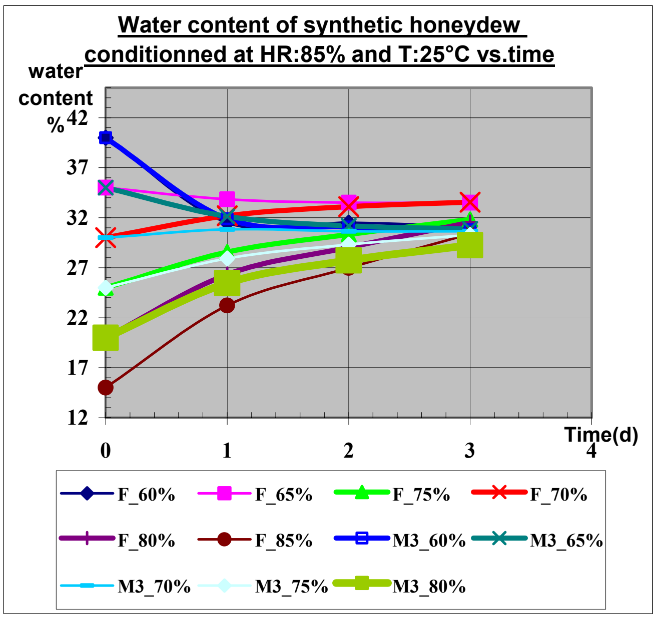


Figure 5: Water content of synthetic honeydews conditioned at HR:85% and T°:25°C.

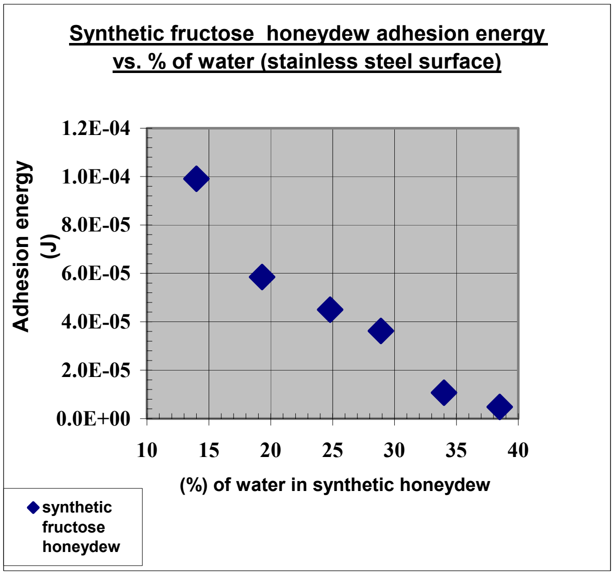


Figure 6: Fructose adhesion energy vs % of water.

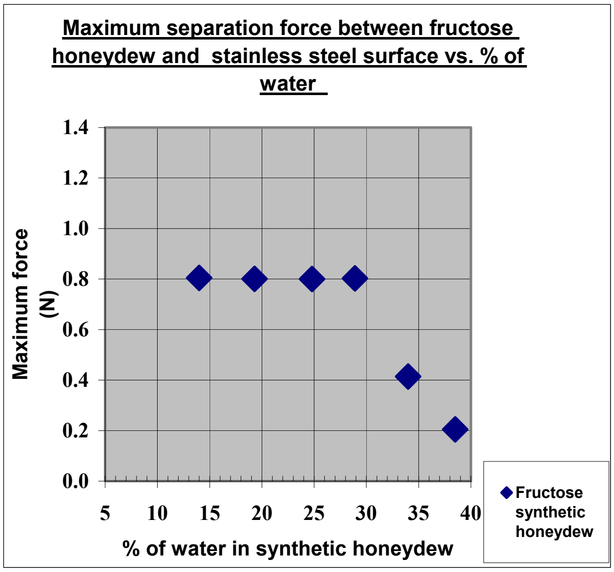


Figure 7: Maximum separation force between fructose honeydew and stainless steel vs % of water.

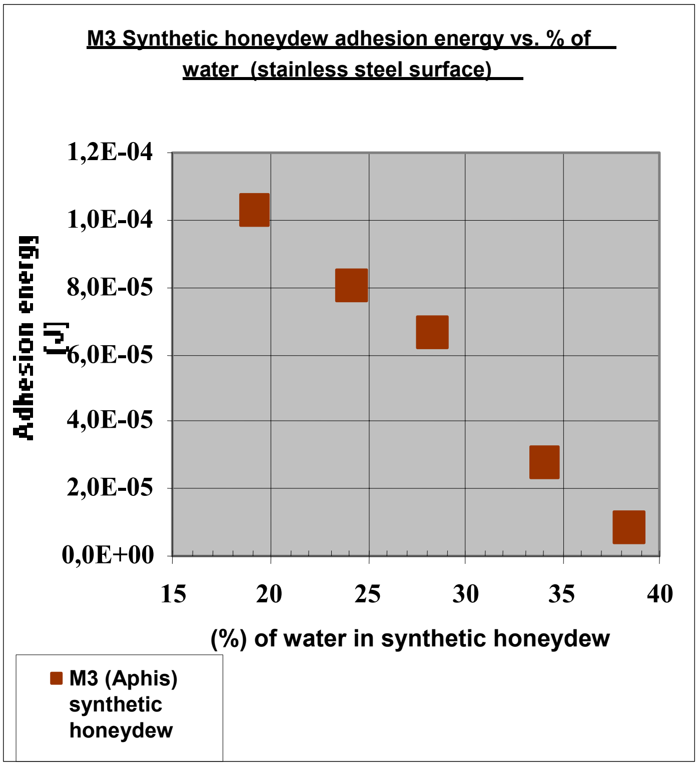


Figure 8: M3 adhesion energy vs % of water.

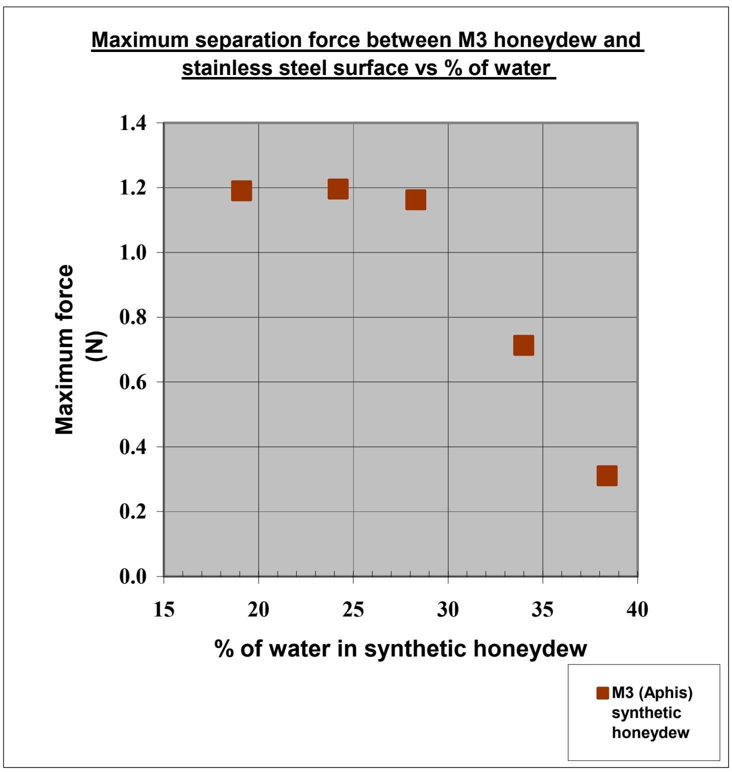


Figure 9: Maximum separation force between M3 honeydew and stainless steel vs % of water.