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EVALUATION OF TACK BEHAVIOUR OF COATING SOLUTIONS

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ABSTRACT

A new apparatus was constructed and a method was suggested to measure the tackiness of tablet coating solutions. The method is simple, rapid, reproducible and it can be used to fulfil either researchal or control investigations.

Water solutions /or dispersions/ of seven different coating materials widely used in the industry were investigated. On the basis of experimental results it

was concluded that increase in concentration of solution led to increase in tackiness. By the evaluation of the data gained from the tack values vs. concentration curves it was possible to choose the concentration of coating solution which proved to be the most favorable from the aspects of both producing requirements and tackiness.

The construction and the evaluation of the so called "Tack curves" showing the change of tackiness in time. The data of these curves add further useful informations to the optimization of the production parameters of coating.

INTRODUCTION

Film coating procedure is spread all over the world and there are large numbers of the film coated drugs these days. The quality of coatings and, to a certain extent, the quality of produts too are determined by the properties of the film coating materials. Therefore much effort has been devoted to the study of solubility, permeability, mechanical and other properties of films made of different film coating materials /1-10/. The linkage of coating film to the surface of tablet is important from the point of view of firm, homogeneous coating and the quality of product as well

/11-15/. The tack behaviour of coating solutions and not fully dry coatings play important part in working up manufacturing and in coating itself /14, 16-21/. In this latter respect there were significant the systematic investigations that dealt with the tackiness of solutions, first of all theoretically /18-20/.

The main purpose of the present study was to work up method for measuring of tackiness connected directly to the requirements of practice. By means of this method, further on, it was possible to assess the tackiness of some polymer solutions used as film-forming materials in coating of solid dosage forms.

On the other hand it is important to know how tackiness changes as a function of time, in order to optimize the process parameters and the film coating procedure. Such data give useful predictions concerning the changing of the tackiness of the coating solution on surface of the particles during the coating process.

EXPERIMENTAL

Materials

Eudragit L-100-55 /metacryl acid and metacryl acid methylester copolymer; Röhm Pharma Gmbh, Darmstadt/, Sepifilm-002 /cellulose substituted by hydroxypropyl--methyl groups and polyoxethen-8-stearate; Honeywill Stein Ltd, Wellington/, Pharmacoat-603 and -606

/hydroxypropyl-methyl-cellulose; Shinetsu Chemical, Tokyo/, Natrosol-HR /hydroxyethyl-cellulose; Hercules Gmbh, Hamburg/, Natrosol-HHR /hydroxyethyl- cellulose; Hercules Gmbh, Hamburg/, Natrosol-GR /hydroxyethyl--cellulose; Hercules Gmbh, Hamburg/.

The film forming materials were solved in distilled water with mixing, at room temperature in different concentrations. /From Eudragit L-100-55 water dispersions were made following industrial instructions/.

Tack Measurement Assembly

Tack measurements were made using a new apparatus constructed specially for this investigation. Schematic diagram of the apparatus used is shown in Fig. 1.

The measuring plate and the measuring probe were made of stainless steel. The working area of the probe and plate were polished. The probe was round, 1 cm^2 large, its vertical moving speed was 0,08 mm/s, weight was 47 g.

Measurement of Tack

10 µl of test liquid was dropped on the measuring plate while the measuring probe was in upper position. Then the probe was put against the test liquid i.e. against the measuring plate. After this an upward movement of probe was started. By the distraction of the surfaces, by its "neckdown" the digital volt meter shows larger and larger force value. The maximum force

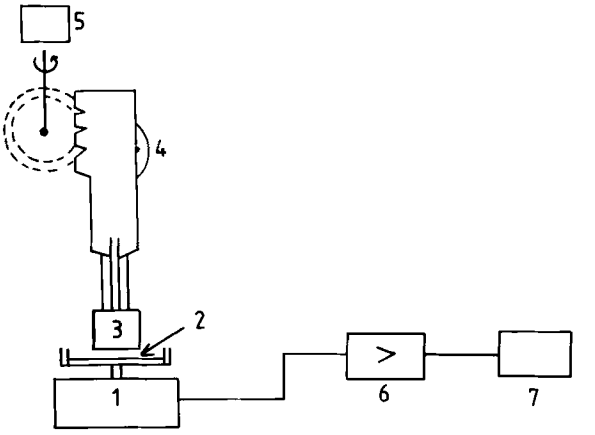

FIGURE 1

Schematic Diagram of Tack Measuring Appliance. Key: 1, load cell 40 N capacity; 2, measuring plate; 3, measuring probe; 4, knob for moving probe; 5, synchronic motor; 6, amplifier; 7, digital volt meter.

value shown at the separation of the test liquid is recorded by the digital volt meter. To qualify the tack behaviour of the test liquid the measured force was used, in terms of milli Newton /mN/. This was called "Tack value".

Prior to the start of the systematic investigations some preexperiments were made on different temperature. The results of these preexperiments showed that the change of temperature could lend more or less deviation to tack value. Therefore considering the aspects of industrial production, the measurements were made at 23°C /± 1°C/ and 38°C /± 1°C/, respectively. Each tack

value was given after the mean of ten runs in this study. The experimental tack values were within a margin of error of ± 9%.

Measurement of Tack in Connection with Evaporation /Drying/ Time

The measuring probe was put against the 10 µl of test liquid on the measuring plate. By this the uniform spread of test liquid on the surface of the measuring probe was brought about. After this the probe was raised from the plate and the test solution was allowed to evaporate for a given time. When evaporating time was over the probe was again put on the plate and the tack value was measured. During measurements the evaporation time of each measurement exceeded that of the previous one by 20 sec. When the tack values, after their initial increase, began to decrease measurements were completed. In order to get more exact data waiting /evaporating/ times were changed by 10 sec., near the maximum of the tack value vs. time curves. Minimum ten measurements were taken for every test solution at each time, at 23°C /± 1°C/. The experimental tack values were within a margin of error of ± 10%.

RESULTS AND DISCUSSION

Effect of Polymer Concentration on Tack

In accordance with the results of investigation of Eudragit L-100-55 water dispersions /Figure 2/ the

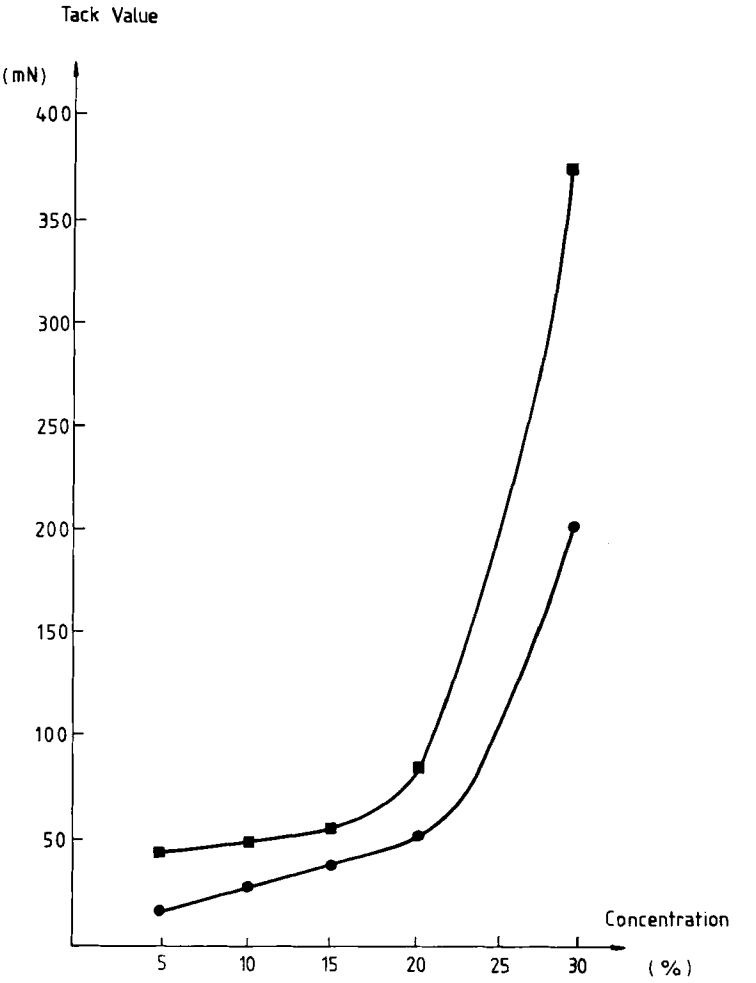

FIGURE 2

Tack Value vs. Concentration of Eudragit L-100-55 Dispersion. Key: ● 23°C; ■ 38°C.

tackiness shows significant change at about 20% concentration. However, even the tack value of the 30% concentration solution, that is the highest concentration suggested by the producer, is not large. Therefore the tackiness significantly does not make the coating process parameters makes of even 30% Eudragit L-100-55 coating dispersion possible to be used in industry. The Figure 2 shows that the increasing of temperature resulted in a significant increase of tack values. It seems probable that its reason may be due to different solvation degree of dispersed polymer particles, to the change in the interaction of polymer-solvent system at higher temperature.

The data of investigation of Pharmacoat-603 and Pharmacoat-606 /Figure 3, 4/ give rise to an interesting comparison. The tack values of the samples made of Pharmacoat-606 - which is similar in setting up but larger in molecule weight than Pharmacoat-603 - are bigger by nearly one order of magnitude than the tack values of the same concentration solution of Pharmacoat--603. This finding refers to that the tackiness is in close connection with the size of the polymer molecule, as some more physical properties of polymers. The different properties of Pharmacoat-603 and Pharmacoat-606 also appear in tack values measured at different temperature. In case of Pharmacoat-603 the increase of

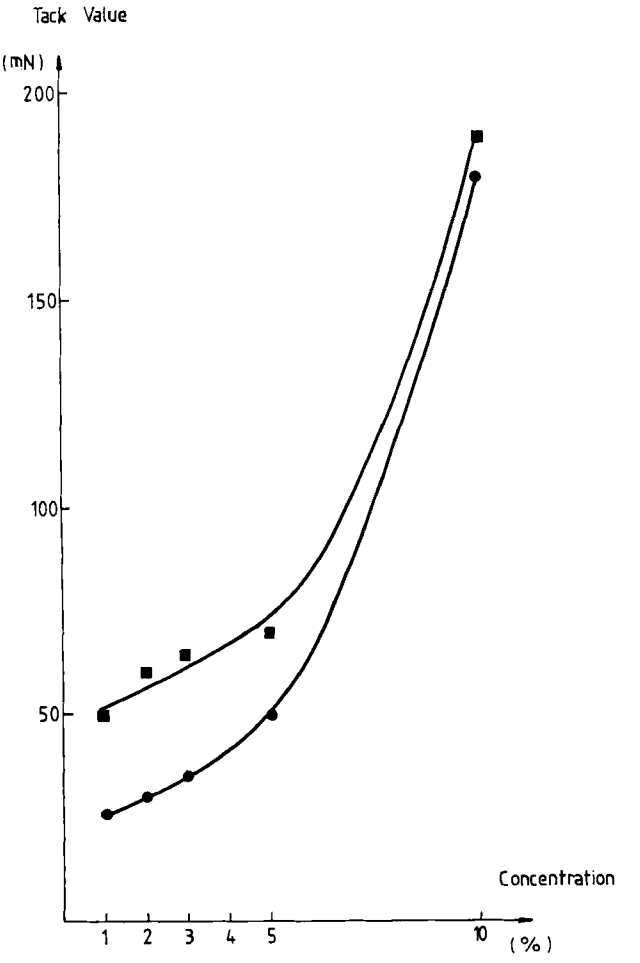

FIGURE 3

Tack Value vs. Concentration of Pharmacoat-603 Solution. Key: ● 23°C; ■ 38°C.

temperature results in relatively larger increase of tackiness at low concentrations than at higher concentrations. Just the opposite is experienced in case of Pharmacoat-606. The exact interpretation of this occurs is not known. The reason for this is also suppo-

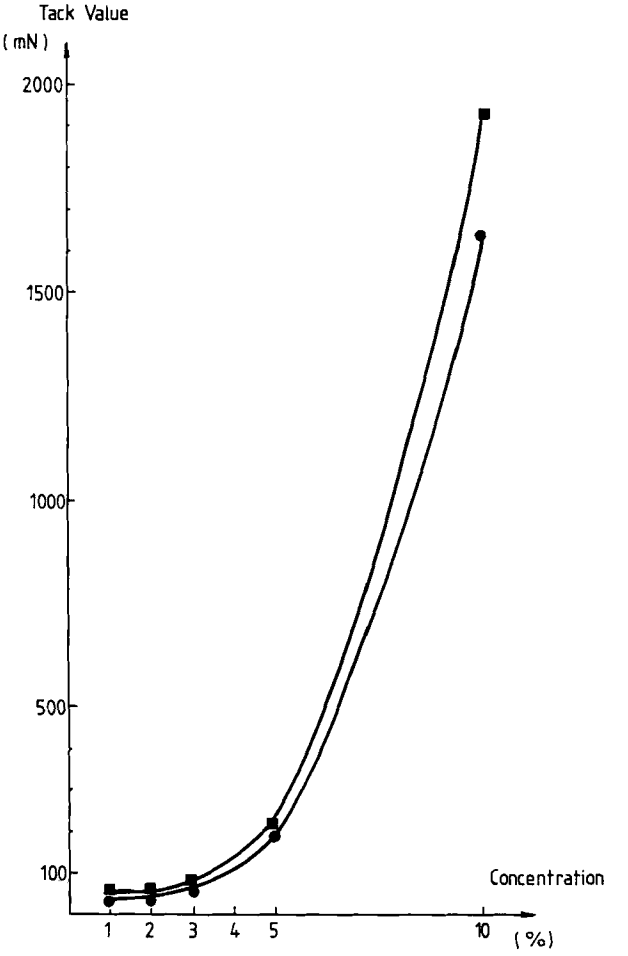

FIGURE 4

Tack Value vs. Concentration of Pharmacoat-606 Solution. Key: ● 23°C; ■ 38°C.

sed to be the molecular structure. Evaluating these results from the point of view of industrial production it can be declared that 10% concentration water solution of Pharmacoat-603 and 5% water solution of Pharmacoat-606 or some lower concentrations of these are suitable for coating processes.

The results of investigation of Sepifilm-002 /Figure 5/ show a significant increase of the tack values in the investigated relatively wide concentration field as the concentration of solution increase. The effect of the increasing of temperature is the most explicit at the highest concentration. /Remark: the sample containing 25% polymer was very densely fluent and hardly moving/. Approximatelly 10% aqueous solution seems to be suitable for coating of solid dosage forms.

The tack values of hydroxyethyl-cellulose polymers - Natrosol-HR, Natrosol-HHR, Natrosol-GR - changed considerably with the increase of the polymer concentration /Figure 6, 7, 8/. The anomaly observed at 3% and 5% concentrations of the Natrosol-HR and Natrosol-GR can probably be due to the change in the polymer solvation degree. These samples were also very densely fluent almost gelatinous. The changing of temperature practically did not lead to change in tack values. On the basis of the measured tack values the 1,0% aqueous solution of Natrosol-HR, 0,5% aqueous solution of Natrosol-HHR and 1,5% aqueous solution of Natrosol-GR seem to be suitable for the the production of film--coated preparations.

Effect of Evaporation Time of Solvent on Tack

Time depending tack values resulted in "Tack curves". The "tack curves" of Pharmacoat-606 solutions,

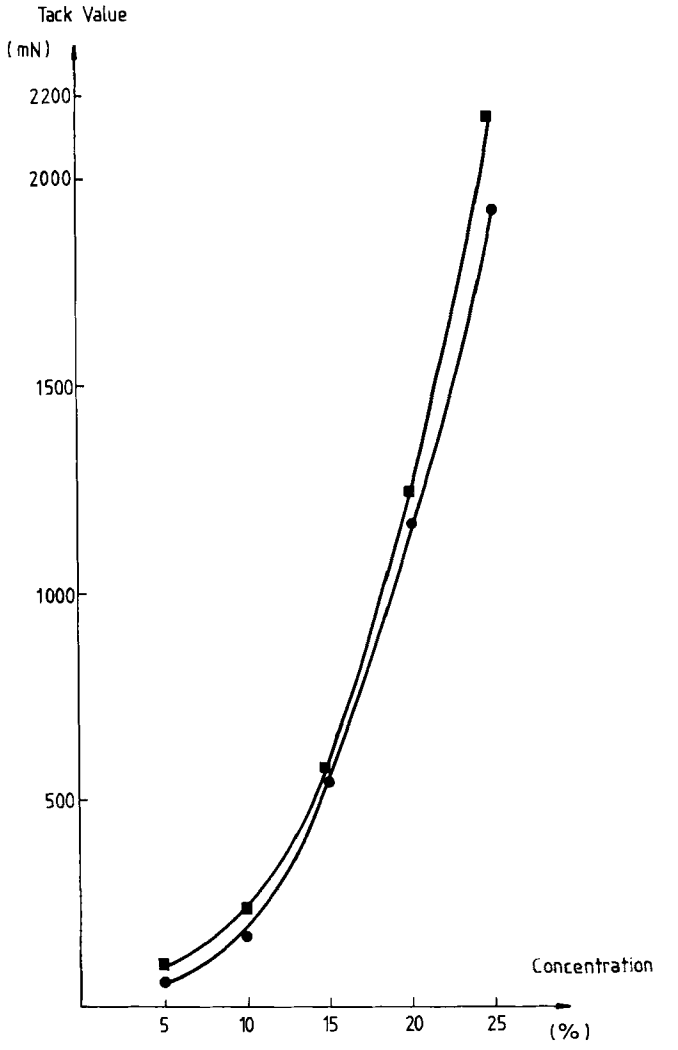

FIGURE 5

Tack Value vs. Concentration of Sepifilm Solution.
Key: ● 23°C; ■ 38°C.

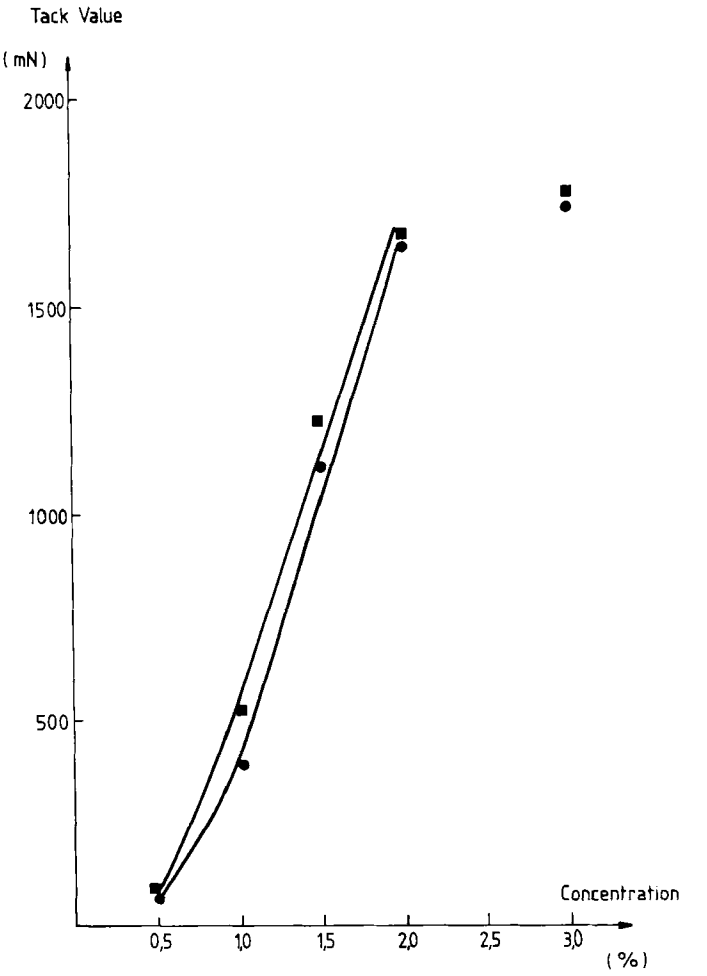

FIGURE 6

Tack Value vs. Concentration of Natrosol-HR Solution.
Key: ● 23°C; ■ 38°C.

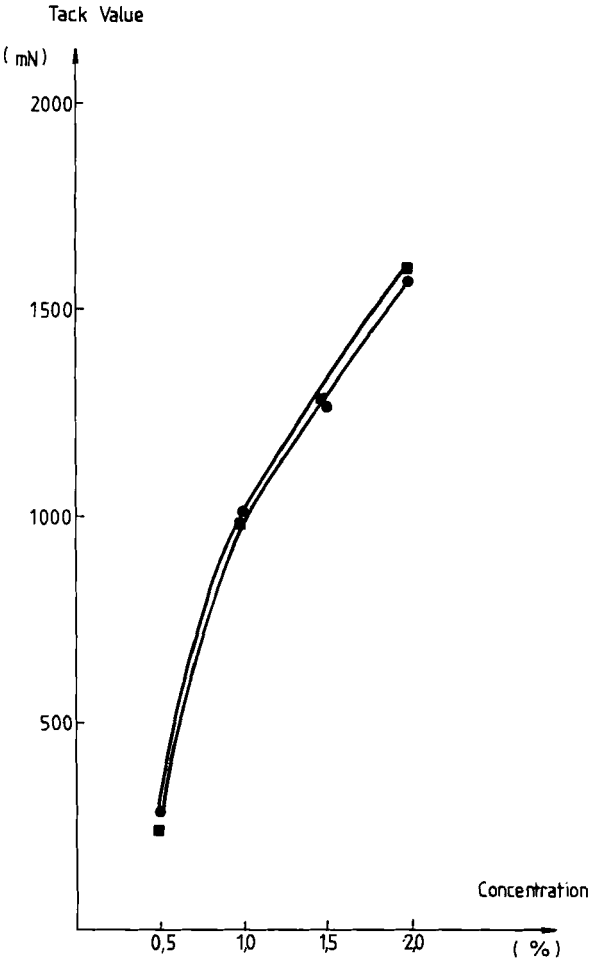

FIGURE 7

Tack Value vs. Concentration of Natrosol-HHR Solution.
Key: ● 23°C; ■ 38°C.

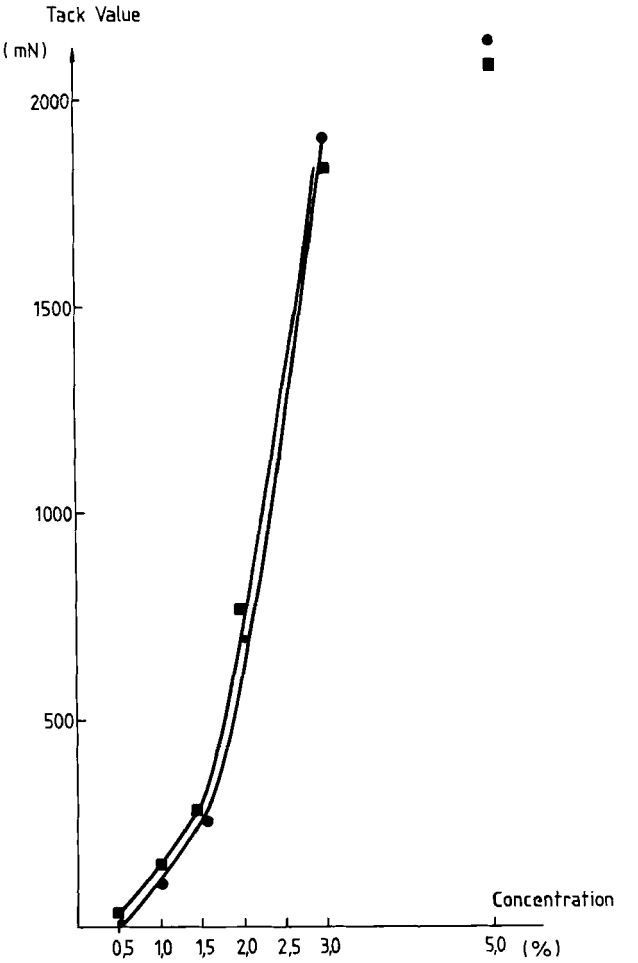

FIGURE 8

Tack Value vs. Concentration of Natrosol-GR Solution.
Key: ● 23°C; ■ 38°C.

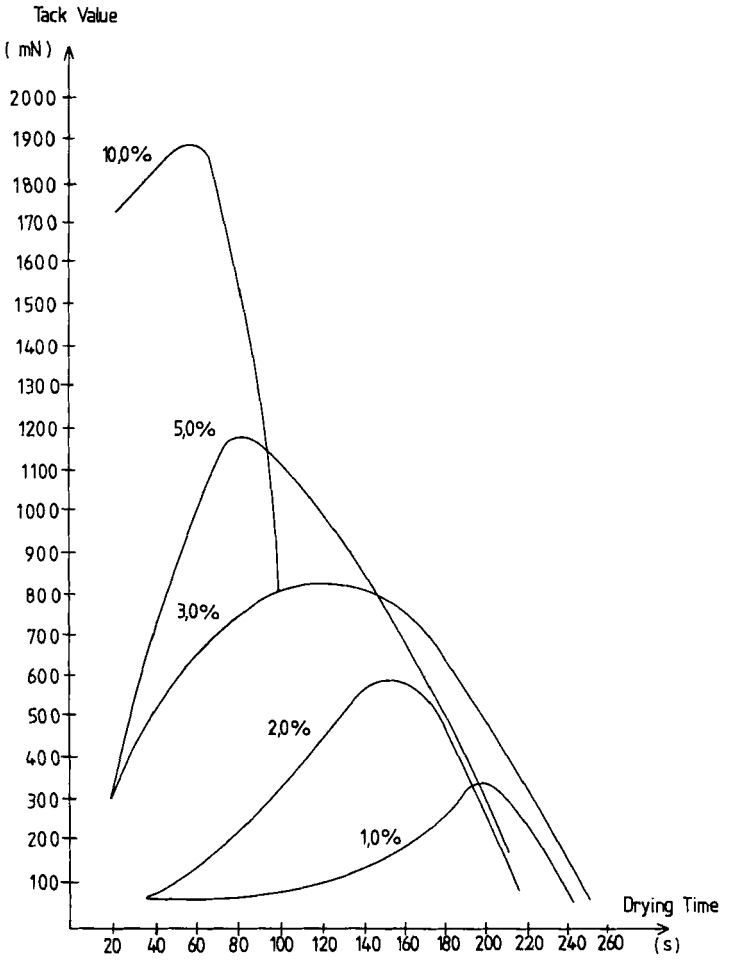

FIGURE 9

Dependence of Tack Value on Drying Time for Aqueous Solutions of Pharmacoat-606.

as characteristic curves , are shown in Figure 9. By means of the "tack curves" two characteristic data of changing of tackiness in time can be obtained. The tack value belonging to the maximum of tack curve is named "Maximum tack value" and the time belonging to this is named "Tack maximum time" /Table 1/. Analysis of the data of the Table 1 and the curves showed that as the concentration of coating solution increases, the tack curves moved to the left. Thus, together with the increase of the concentrations, the "tack maximum times" decrease. At the same time - similarly to the tack values - the "maximum tack values" increase as well.

The results of these investigations comparing with the industrial experiences of coating process, it was found that the "maximum tack value" and the "tack maximum time" give useful informations to the optimization of process parameters in a certain coating system. These are so called predictive parameters. They form important part of the highly automatized, programmed coating processes. However, it has to be taken into consideration that these data are only predictions concerning of industrial production. Therefore using up of these predictive parameters can only be considered together with the other producing parameters, in order to optimize the producing process.

TABLE 1

Tack Maximum Values and Tack Maximum Time Belonging to Them

Polymer	Concentration % /^{w}/w/	Tack Max. Values/mN/	Tack Max. Time/s/
Eudragit L--100-55	5,0	150	130
	10,0	170	120
	15,0	200	100
	20,0	430	40
	30,0	750	20
Pharmacoat--603	1,0	150	210
	2,0	160	200
	3,0	180	180
	5,0	750	120
	10,0	850	30
Pharmacoat--606	1,0	350	200
	2,0	610	160
	3,0	840	130
	5,0	1180	80
	10,0	1890	60

TABLE 1
/continue/
Tack Maximum Values and Tack Maximum Time Belonging to Them

Polymer	Concentration % /w/w/	Tack Max. Values/mN/	Tack Max. Time/s/
Sepifilm-002	5,0	390	90
	10,0	690	50
	15,0	785	30
	20,0	1710	20
	25,0	2520	10
Natrosol-HR	0,5	1370	80
	1,0	1970	60
	1,5	1990	50
	2,0	2000	30
	3,0	2300	20
Natrosol-HHR	0,5	740	70
	1,0	1440	40
	1,5	1470	30
	2,0	1780	20
Natrosol-GR	0,5	370	100
	1,0	940	80
	2,0	2140	60
	3,0	2460	30

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