Latex Dipping I. Relationship Between Dwell Time, Latex Compound Viscosity and Deposit Thickness

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The thickness of dry rubber deposited on straight and coagulant dipping has been investigated for three latex compounds, using variations in dwell time and compound viscosity. A relationship between these variables was obtained by an examination of the results and of the process.

Use of latex concentrate for making dipped articles is probably the oldest, and certainly the simplest, process in latex technology. Despite the long established practice of latex dipping, surprisingly little has been published on this subject, apart from patents and general descriptions of the process (e.g. Cook 1956, Noble 1953, Stern 1957 and Meher, 1954).

There are three processes of latex dipping:

(i) *Straight dipping.* The clean and dry former is lowered into the latex, slowly removed, inverted and dried. This procedure gives rise to very thin articles, say of 0.01 mm in thickness.

(ii) *Coagulant dipping.* In this case, the former is dipped into a coagulant solution (e.g. calcium nitrate, or cyclohexylamine acetate, in methylated spirit), withdrawn and then lowered into the latex compound. After a suitable dwell time (1–5 minutes), the former is slowly withdrawn, inverted and dried. This process gives articles of medium thickness (0.1 mm), and it is the most commonly used method.
It is customary to dip the former first into the compound and then dry it ('straight dipping') before dipping it into the coagulant, as this assists in the production of even deposits and also protects the formers.

(iii) *Heat-sensitive dipping.* When thick-walled products are required, a latex compound which gells above a certain temperature is used. The heated former is dipped into the compound and rubber is deposited on the surface. As before, the former is withdrawn, inverted and dried. This system is not discussed here.

In all these cases, however, the latex compound can be pre-vulcanised (in which case, only drying is required after dipping), or post-vulcanised where the article must be dried and vulcanised.

Vulcanisation of the dipped rubber articles will be no different from any other product and various formulations can be used to produce the required technological properties. However, the aspect of rubber deposition from the latex compound, whether pre- or post-vulcanised, has received little attention, and is yet of prime importance in producing articles of required thickness and therefore modulus. Published information includes some data on deposit thickness and latex concentration (Esser and Sinn 1963, Revertex Limited, 1966).

The purpose of this paper is to assess the relationship between deposit thickness, latex compound viscosity and dwell time of the former.

METHODS

All dipping experiments have been carried out at 25°C using a 'Cotswold' hydraulic dipping machine (upstroke) model MK IRL/20 and polished porcelain prophylactic dipping formers.

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The following procedure was adopted:

Straight Dip

Stage 1. The former was dipped into latex at the rate of 87.6 cm/min.

Stage 2. The former was withdrawn at 61 cm/min, inverted and dried in a circulating air oven at 70°C for 20 minutes. If required, post-vulcanisation was effected for 30 minutes at 100°C.

Coagulant Dip

Stage 1. The former was straight dipped as above, withdrawn and dried.

Stage 2. The former was, with dip coat, immersed in 20% cyclohexylamine acetate in industrial methylated spirit, at the rate of 130 cm/min, withdrawn immediately at the same speed and inverted for 1 minute.

Stage 3. The former was immersed in latex compound at the rate of 130 cm/min; at various intervals of dwell time, the former was raised about 1 cm at rate 61 cm/min. When completed, the former was totally withdrawn at the latter rate, inverted and dried 60 minutes at 70°C. Where necessary, post-vulcanisation of 30 minutes at 100°C was employed.

The rubber deposits were dusted with talc and were stripped from the former. Thicknesses were determined on a Mercer gauge, using a load pressure of 211 g/cm². The arithmetic mean of five measurements was taken for each thickness figure.

Viscosity determinations were carried out at 25°±1°C using the Brookfield Viscometer LVF model and Spindle 1. Precautions were taken to eliminate any thixotropic tendency of the compounds by subjecting them to high shear stirring for 4 minutes before measuring the viscosity. All viscosity determinations were done just prior to dipping.

A photograph of the former dipping into the latex compound is shown in *Figure 3*.

The following latex compounds were used:

Mix A: (Black Glove Compound)

Latex (as 60% HA concentrate)	100
Vulcastab LW (as 20% solution)	0.2
Potassium hydroxide (as 10% solution)	0.5
Sulphur (as 50% dispersion)	1
Zinc diethyldithiocarbamate (as 40% dispersion)	0.5
Zinc oxide (as 50% dispersion)	1
Nonox EXN (as 40% dispersion)	1
HAF black (as 10% dispersion)	0.25
Devolite clay (as 50% slurry)	20
Total solids (by measurement)	58.3 %

This compound requires post-vulcanisation.

Mix B: (General Purpose Commercial Prevulcanised Latex)

"Revultex MR" (from Revertex Ltd.)	
Total solids (by measurement)	61 %

Mix C: (High modulus compound)

Latex (as 60% HA Concentrate)	100
Sulphur (as 50% dispersion)	2
Zinc diethyldithiocarbamate (as 40% dispersion)	1
Zinc mercaptobenzthiazole (as 50% dispersion)	1
Nonox EXN (as 40% dispersion)	1
Zinc oxide (as 50% dispersion)	5
Total solids (by measurement)	60.2 %

This compound requires post-vulcanisation. All the above concentrations are based on parts of dry weight.

The compounds were used both at the quoted solids concentrations and after dilution with distilled water to provide other total solids concentrations.

RESULTS

The results are shown in *Tables 1–3*.

DISCUSSION

Straight Dip

The variation of deposit thickness with latex viscosity is shown in *Figure 1*, where one curve adequately describes all three mixes.

If the viscosity is expressed as its logarithmic value, a reasonable straight line is obtained (*Figure 2*). Therefore it is concluded:

$$t = a_1 + K_1 \log(\eta 60) \quad \ldots\ldots\ldots\ldots(1)$$

where t refers to the straight dip deposit thickness

a_1 and K_1 are constants

and $\eta 60$ is the apparent viscosity (Brookfield) at 60 rev/min.

It has been shown further that such relationship holds at other rates of shear (and therefore viscosity). An alternative way of expressing this is to say that there exists a direct relationship between straight dip deposit thickness and latex total solids, as can be observed from the *Tables 1* to *3* and as shown previously (GORTON, 1962). The constants in the Equation (1) would depend on the mixes and, in this paper, only the pattern of dependence of thickness on other variables is of interest.

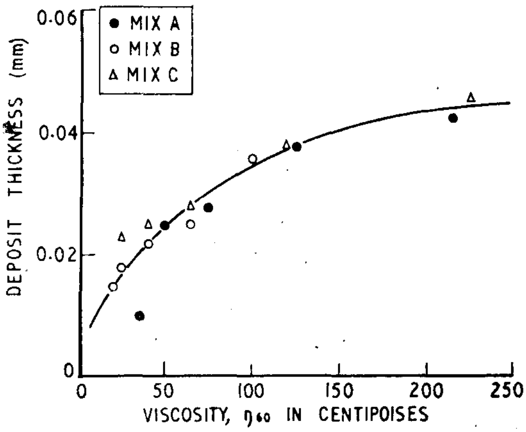

Figure 1. Straight deposit thickness and latex viscosity $\eta 60$.

Coagulant Dip

The total dip deposit here is dependent on two factors—dwell time and latex compound

TABLE 1. MIX A. THICKNESS OF DEPOSITS (MM), DWELL TIME (S) AND THE BROOKFIELD VISCOSITY (CP)

Total solids*	58.25%	56.32%	54.28%	52.31%	50.41%
Straight dip deposit thickness (mm)	0.043	0.038	0.028	0.025	0.010
Coagulant dip and deposit thickness (mm) at dwell time (s)					
10″	0.27	0.26	0.23	0.20	0.18
30″	0.37	0.33	0.30	0.24	0.25
60″	0.42	0.40	0.35	0.29	0.29
90″	0.48	0.44	0.39	0.33	0.35
120″	0.50	0.47	0.40	0.36	0.35
180″	0.55	0.52	0.45	0.41	0.38
300″	0.72	0.63	0.55	0.50	0.45
Brookfield viscosity (cp) Spindle 1					
$\eta 6$ rev/min	613	285	185	133	77.5
$\eta 60$ rev/min	217	123	77.0	51.5	36.3

*The measured total solids concentration differs slightly from that obtained by calculation due to the presence of non-active ingredients added to the latex and dispersions.

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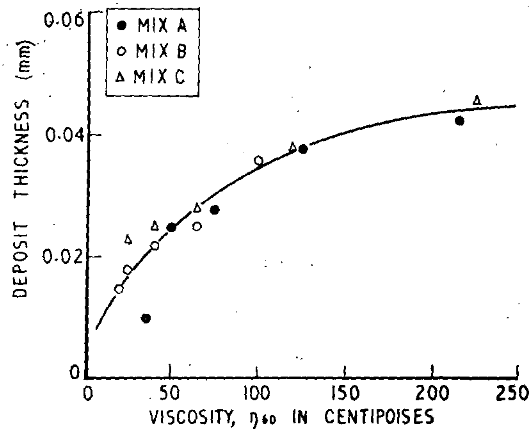

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TABLE 2. MIX B. THICKNESS OF DEPOSITS (MM), DWELL TIME (S) AND THE BROOKFIELD VISCOSITY (CP)

Total solids	60.77%	58.09%	55.60%	53.06%	50.59%
Straight dip deposit thickness (mm)	0.036	0.025	0.022	0.018	0.015
Coagulant dip deposit thickness (mm) at dwell time (s)					
10″	0.25	0.22	0.19	0.17	0.15
30″	0.30	0.29	0.27	0.23	0.19
60″	0.35	0.35	0.30	0.28	0.23
90″	0.39	0.38	0.31	0.29	0.26
120″	0.41	0.38	0.34	0.30	0.27
180″	0.46	0.42	0.38	0.33	0.28
300″	0.58	0.49	0.45	0.37	0.28
Brookfield viscosity (cp) Spindle 1					
η6 rev/min	230	160	83.0	53.0	42.0
η60 rev/min	98.0	64.5	40.4	27.1	21.6

viscosity. The deposit thickness variation with viscosity is shown in *Figure 4* and, with dwell time, in *Figure 5*. The situation here is complicated by the use of the initial straight dip, and a cross section of the deposit appears as shown in *Figure 6* which shows five regions:

Region I – dry deposit from straight dip;
II – dry deposit of coagulant;
III – wet coagulum formed by 'straight dip', called 'first deposit';
IV – wet coagulum formed by coagulant, called 'second deposit'; and
V – latex compound.

TABLE 3. MIX C. THICKNESS OF DEPOSITS (MM), DWELL TIME AND THE BROOKFIELD VISCOSITY (CP)

Total solids	60.24%	57.74%	55.30%	52.88%	50.13%
Straight dip deposit thickness mm.	0.046	0.038	0.028	0.025	0.023
Coagulant dip deposit thickness (mm) at dwell time (s)					
10″	0.29	0.27	0.21	0.19	0.17
30″	0.38	0.31	0.27	0.24	0.22
60″	0.43	0.37	0.32	0.29	0.27
90″	0.47	0.40	0.35	0.34	0.29
120″	0.52	0.42	0.39	0.26	0.32
180″	0.55	0.45	0.44	0.40	0.34
300″	0.74	0.57	0.50	0.45	0.41
Brookfield viscosity (cp) Spindle 1					
η6 rev/min	563	260	160	90.0	47.0
η60 rev/min	223	118	67.0	39.5	24.0

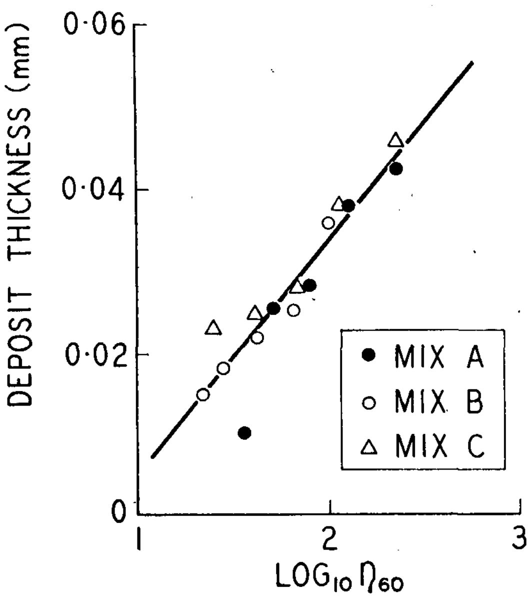


Figure 2. Straight dip deposit thickness and log_{10} *η60.*

The mechanism of the process is as follows:

The former, with straight dip deposit and dry coagulant, is immersed in the latex compound. The first deposit to form is primarily the same as a straight dip—hence Region III. Simultaneous with this deposition will be the dissolution of the coagulant and migration through Region III to the latex compound. Contact will then occur, and the first 'real' coagulant deposit will take place. Region IV will thus grow as the Interface IV/V extends away from the former. As the coagulum deposit is formed, two effects will hinder its growth:

(i) Release of serum and subsequent dilution of the coagulant.

(ii) Increase in the thickness through which the coagulant will have to permeate to reach the latex compound.

A further controlling effect is the passage of serum towards the former to assist the initial dissolution of coagulant, so that it is not 'starved'.

If T refers to the total coagulant deposit thickness at a given dwell time, t_1 refers as before to the straight deposit thickness and t' denotes the total coagulant deposit thickness.

$$T=t_1+t' \qquad \text{..............(2)}$$

Since the Region II vanishes and the thickness of Region III (t_3) is of similar magnitude as t_1

$$t' \approx t_3+t_c \qquad \text{..............(3)}$$

$$t'=t_1+t_c \qquad \text{..............(4)}$$

where t_c represents the true coagulant deposit thickness.

From Equations (2) and (4)

$$t_c=T-2t_1 \qquad \text{..............(5)}$$

Therefore, in order to examine the effect of viscosity and dwell time on the true coagulant deposit thickness (t_c), it is necessary to correct the total deposit thickness according to Equation 5.

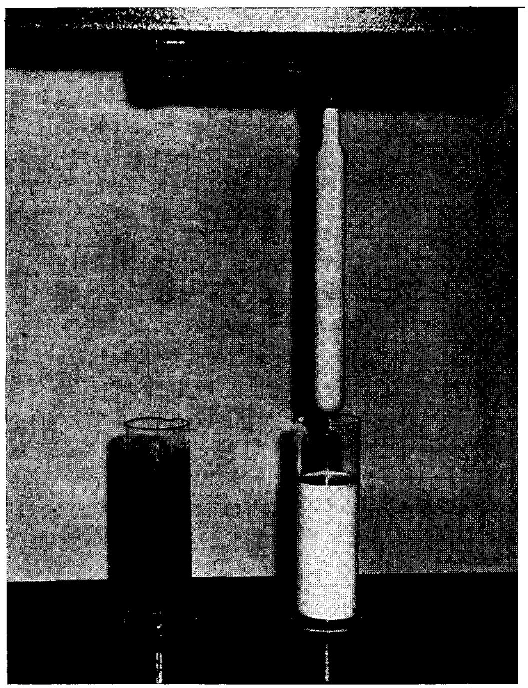


Figure 3. Dipping the former into latex compound.

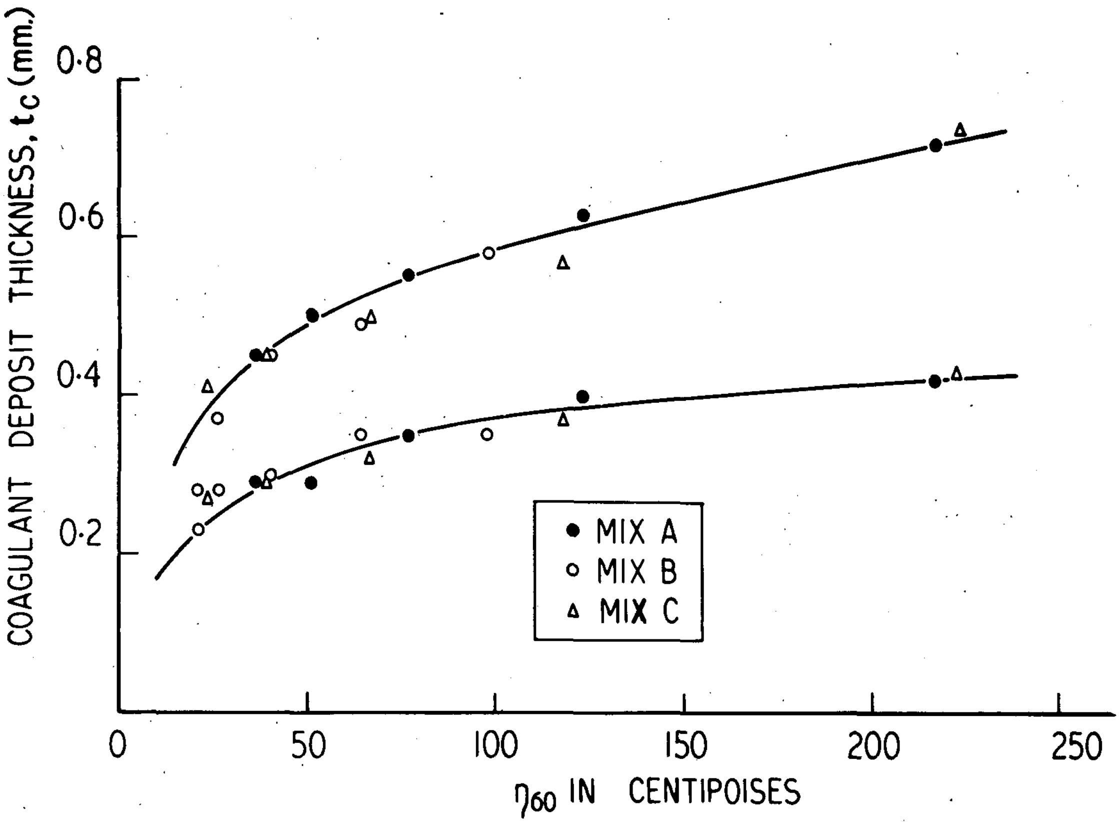


Figure 4. Coagulant deposit thickness and latex viscosity η60.

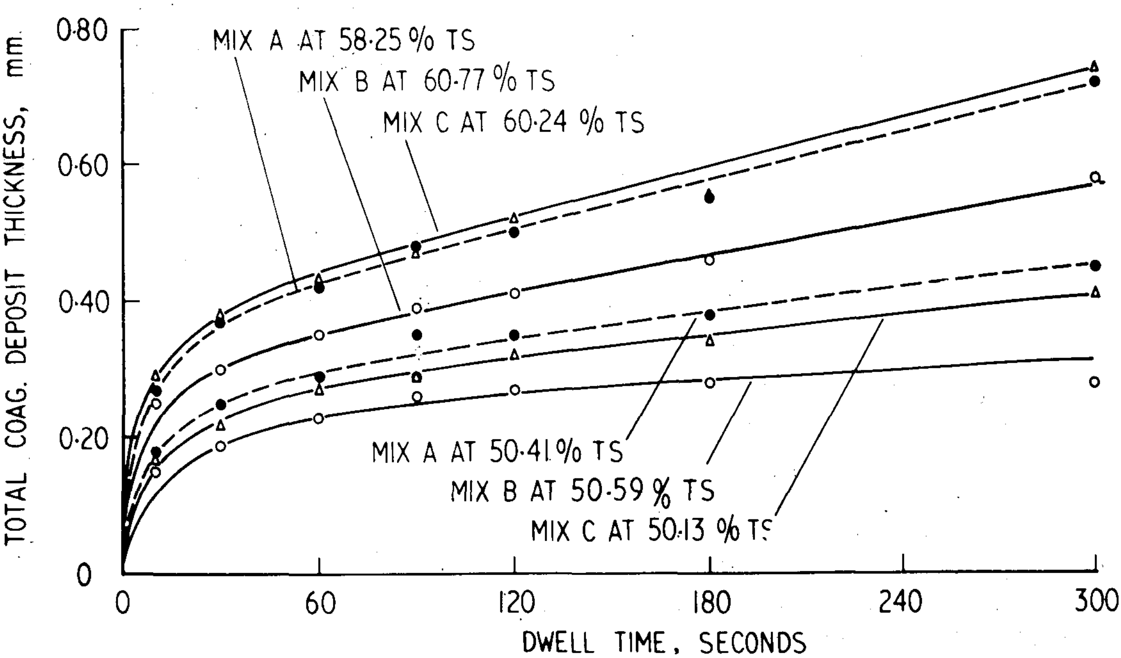


Figure 5. Total coagulant dip deposit and dwell time.

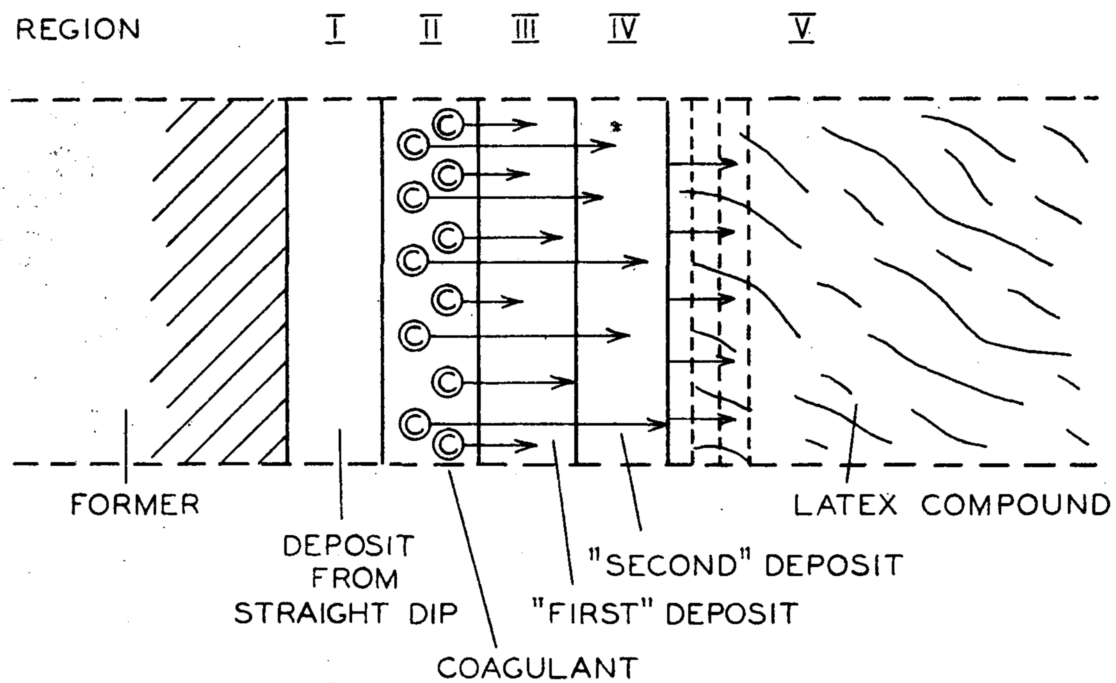


Figure 6. Cross-section of coagulant dipping deposition.

Effect of Dwell Time

Examination of the variation of coagulant deposit thickness with latex dwell time (*Figure 7*) shows that there is a direct relationship between t_c and the square root of the dwell time, i.e.

$$t_c = K_2\sqrt{D} \qquad \ldots\ldots\ldots\ldots\ldots(6)$$

where K_2 is a constant
and D denotes dwell time (min).

Effect of Viscosity of Latex Compound

The variation of coagulant deposit thickness with latex compound viscosity is shown in *Figure 8*. The points can reasonably be represented by straight lines, though some results are scattered. The results for highest and lowest latex total solids of each compound have been plotted. Viscosities were measured as before, and η 60 represents the apparent viscosity of the compound as measured with the Brookfield viscometer at 60 rev/min.

It is concluded that a direct relationship exists between deposit thickness t_c and the latex compound viscosity at a given dwell time as

$$t_c = a_2 + K_3 \log_{10} \eta \qquad \ldots\ldots\ldots\ldots(7)$$

where a_2 and K_3 are constants
and η refers to compound viscosity (centipoises).

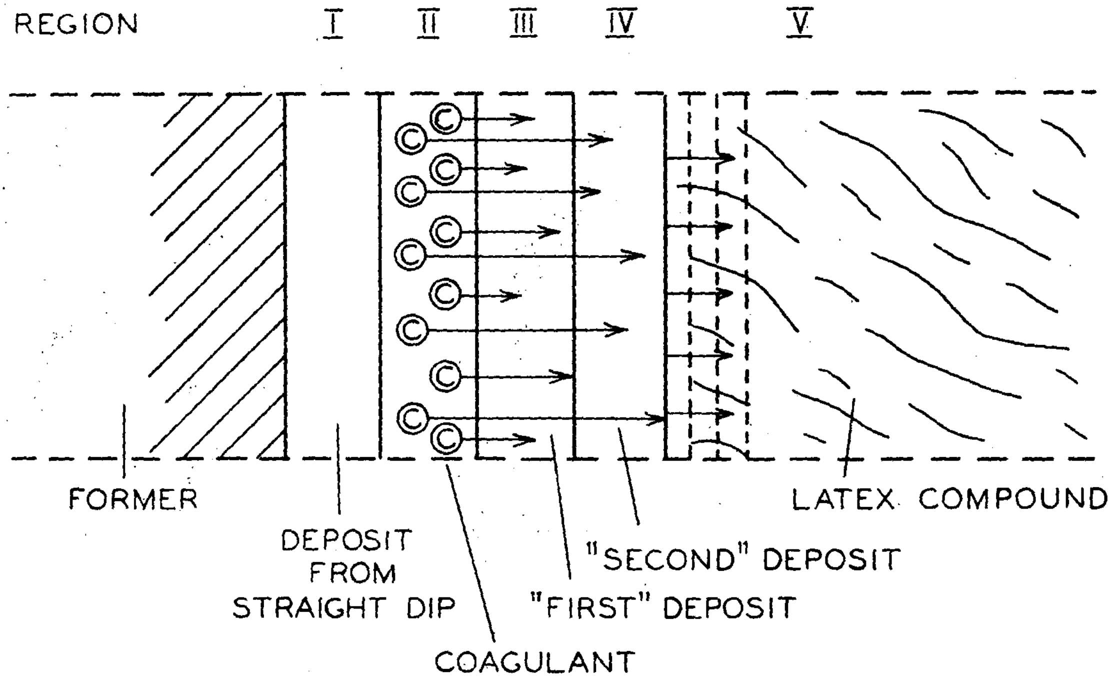
It has been shown that such relationship holds for other rates of shear.

Continuing Equations 6 and 7, a possible dependence for the coagulant deposit thickness on the product of $\sqrt{D}$ and $\log_{10} \eta$ is permissible as

$$t_c = a_3 + K_5 \sqrt{D} \log_{10} \eta \qquad \ldots\ldots\ldots\ldots(8)$$

where a_3 and K_5 are constants.

The relationship between t_c and $\sqrt{D} \log_{10} \eta$ is shown in *Figure 9*, where all the points for *Tables 1* to *3* are plotted. Although for each mix a separate line can be drawn the general



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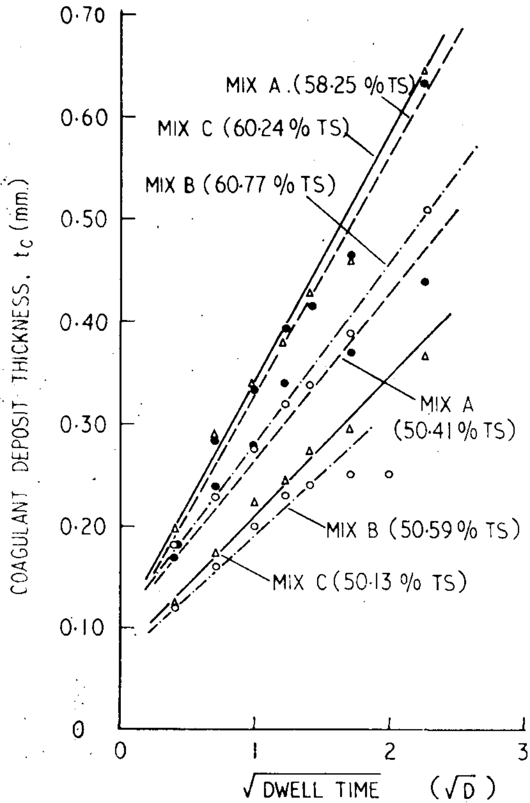

Figure 7. Coagulant deposit thickness and √ (dwell time).

trend shows that the constant a_3 for Equation (8) above can be considered as zero. Thus we have

$$t_c = K_5\sqrt{D}\log_{10}\eta \qquad \text{............(9)}$$

The total thickness of deposit following a straight dip with one coagulant dip is given as T_1

where $T_1 = t_1 + t'$

$$= t_1 + t_1 + t_c$$
$$= 2t_1 + K_5\sqrt{D}\log 10\,\eta$$
$$= 2(a_1 + K_1\log 10\eta) + K_5\sqrt{D}\log 10\eta$$
$$= 2a_1 + \log 10\eta\,(2K_1 + K_5\sqrt{D}) \text{ ...(10)}$$

where in a_1, K_5 are constants.

Thickness of more than one coagulant dip on a former straight dipped once can be assessed within reasonable limits accepting limitations imposed by other considerations.

If n further coagulant dips are carried out denoting by T_n the thickness of deposit

$$T_n = t_1 + nt'$$
$$= (n+1)t_1 + nt_c$$
$$= 2a_1 + \log 10\eta((n+1)K_1 + K_5\sqrt{D})$$
$$\text{............(11)}$$

Other situations of varying the numbers of straight dipping and coagulant dipping can be similarly assessed.

CONCLUSION

The total coagulant dip deposit thickness is directly dependent on the latex compound viscosity and the dwell time according to

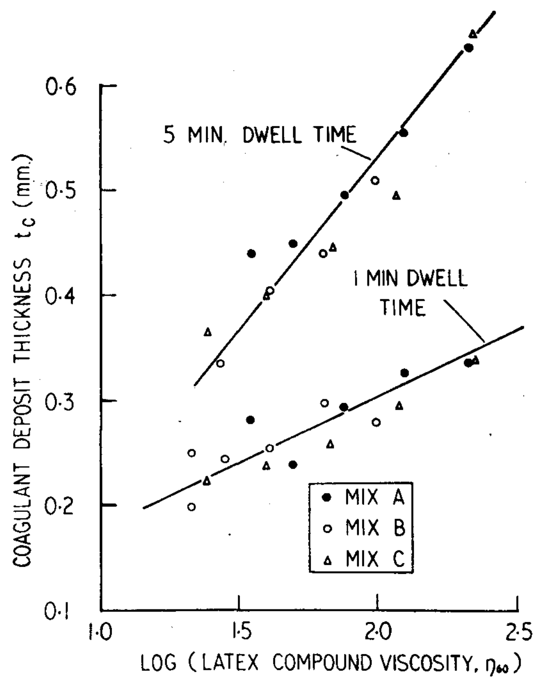

Figure 8. Coagulant deposit thickness and log $_{10}\eta 60$.

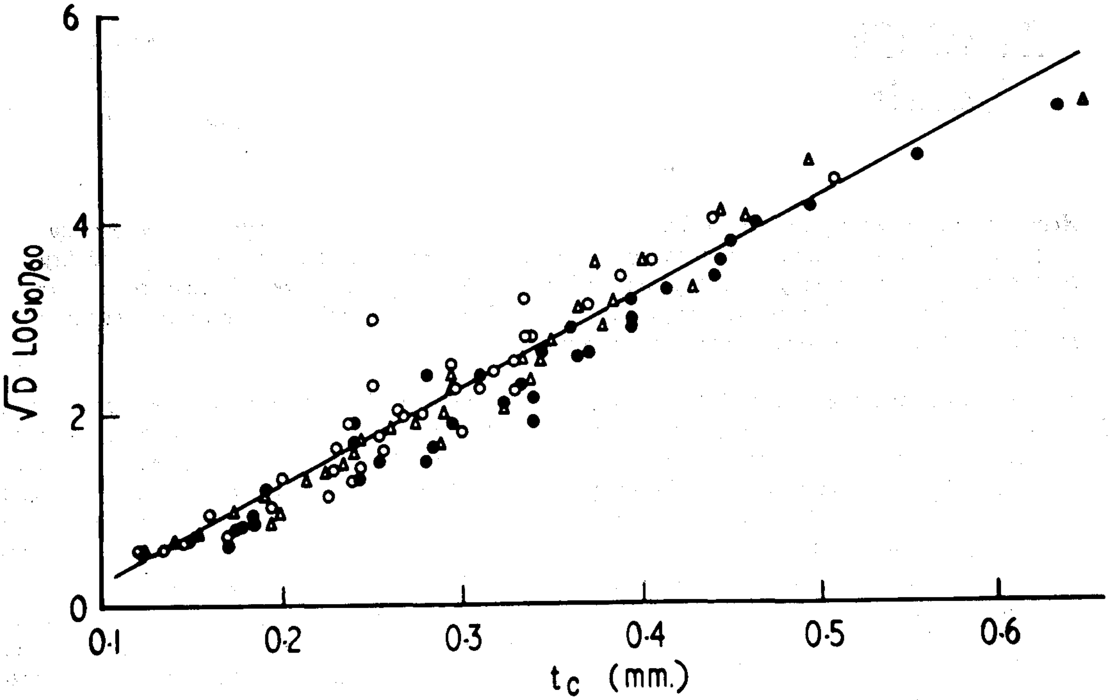

Figure 9. Graph of $\sqrt{D}.\log_{10}\eta 60$ *and thickness of coagulant dip* (t_c).

Equation (9). The constants should be estimated depending on the mix and other conditions involved.

It is considered possible to predict deposit thickness in latex dipping technology from a knowledge of dwell time and latex compound viscosity by an extension of the arguments advanced.

ACKNOWLEDGEMENT

The author acknowledges with gratitude the experimental assistance of Miss Ong Suan Sim. Thanks are also due to Mr Yee Yoke Thong for drawing the graphs, and to Mr G. C. Iyer and Mr Michael Goh of the Statistics and Publications Division for helpful discussions.

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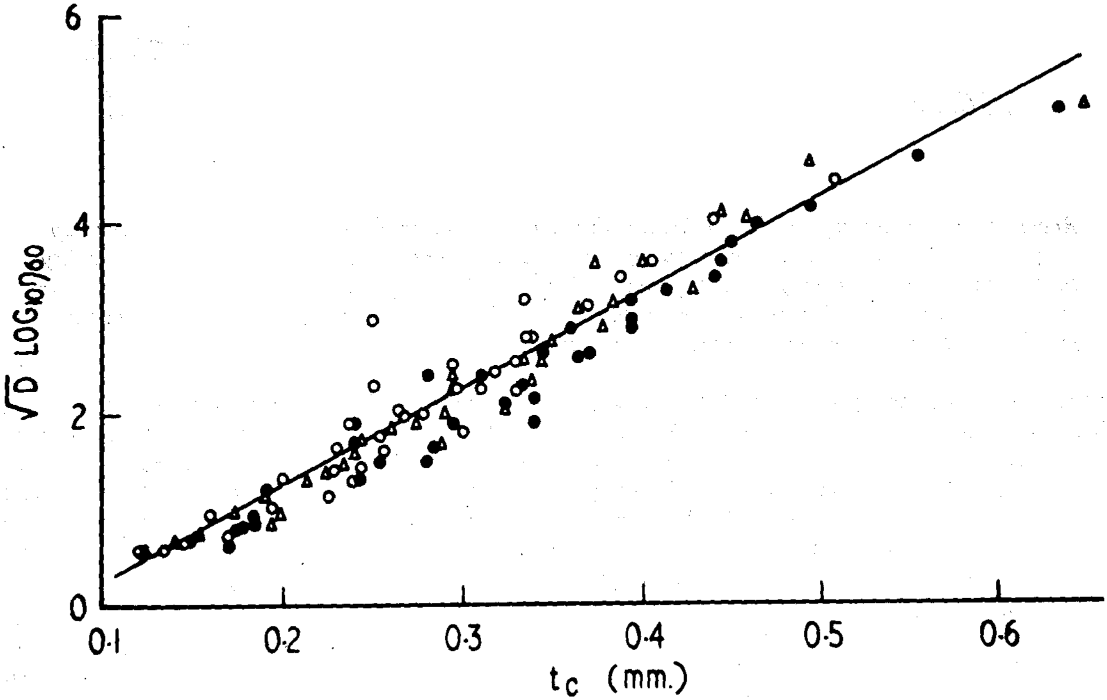

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