A Survey of Factors Involved in an Experimental Study of the Drying of Sheet Rubber

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Aspects of the drying of sheet rubber are studied which include: the methods of preparation of wet sheet rubber and its dimensional changes during drying; the effect of drying conditions on drying rate, blister formation and technological properties; and the effect of relative humidity on the equilibrium water content. The data presented form the basis for further detailed investigations into the effect of drying conditions on drying rate and quality.

INTRODUCTION

Although the drying of sheet rubber is one of the more important processes in the production of dry rubber from field latex, only limited attention has been given to the theoretical aspects of the mechanisms involved. One of the most significant early contributions was by PIDDLESDEN (1937) who found two distinct phases of drying—a rapid reduction in water content down to about 10%, followed by a very slow rate of drying. It was postulated that this second phase was controlled by a diffusion mechanism, since the drying time was found to be roughly proportional to the square of the sheet thickness (a confirmation of an observation by VAN HARPEN, 1930) and also that the shape of the drying curve was in approximate agreement with classical drying diffusion theory (NEWMAN 1931, SHERWOOD 1929, SHERWOOD 1931). Observations, however, showed fairly wide variations from the average relationship between thickness and drying time, and consistent departures from the theoretical drying rate curve; the latter were attributed to dimensional changes and to the characteristics of the drying apparatus. Further, the day-to-day variability of drying characteristics did not allow an accurate assessment of the temperature co-efficient of the drying rate.

STEVENS (1948) expressed the view that the porosity of coagulum is reduced by compression during machining, and that the drying rate of an unmachined coagulum might be greater than that of machined sheet in spite of the former's greater thickness and initial water content.

PHILPOTT AND WALKER (1955) found that the inclusion in sheet of protein precipitants or tanning agents accelerated drying; coupled with the fact that drying is retarded both by removal of protein and the addition of sodium sulphite to latex (DE VRIES 1920), this clearly indicated the important rôle of non-rubber constituents.

In none of these investigations was the effect of relative humidity studied, but PIDDLESDEN (1937) anticipated, on the basis of the diffusion theory, that there would be no effect. The view was held in other quarters that increasing the relative humidity would prevent the formation of an impermeable skin on the surface of the rubber and thus increase the rate of drying.

In reviewing the need for further work it was clear that more information was required on the effects of temperature and humidity, since a knowledge of these two variables is vital to an economic assessment of the drying process: it was also evident that accurate data could only be obtained by paying attention to the many other factors likely to affect the drying characteristics. The investigations presented below in six sections form a preliminary survey of the effects of drying

COMMUNICATION 325

conditions on rates of drying and on the properties of the product.

As variations in sheet dimensions affect the drying rate, an investigation was made (Section 1) into methods of preparing wet sheet rubber and into dimensional changes of the sheet during drying. Section 2 is a report on the effect of drying conditions (temperature, humidity and air speed) on the rate of drying. Relative humidity is considered again in Section 3, where its effect on equilibrium water content is considered; techniques for determining the water content of sheet rubber are examined in Section 4. The last two Sections (5 and 6) are studies of the influence of drying conditions on blister formation and on the technological properties of the sheet.

1. FACTORS AFFECTING DIMENSIONS OF SHEET RUBBER

Dimensional changes in a sheet have a considerable bearing on its drying behaviour. Shrinkage in length and breadth reduces the area available for the evaporation of water, and changes in thickness affect the distance through which water in the interior of the sheet must diffuse before it reaches the surface.

Because of the marked effect of thickness, sheets for drying investigations should be as uniform as possible in this respect, and means for accurate measurement should be available. The thickness of a dry sheet is determined by the method of preparing the coagula, the degree of machining, and by dimensional changes during drying. A knowledge of the shrinkage of a material during drying also gives some indication of the mechanisms involved in water transport within the material.

Method of Preparation of Sheets and Measurement of Thickness

Preparation of sheets. Coagulating pans rather than partition-plate tanks were chosen since the coagulum can be removed from pans with less distortion, and also because there is less likelihood that the thicknesses of various parts of the coagulum will be affected differentially either by creaming of the latex or by compression due to buoyancy forces.

A pair of smooth rolls was used for machining, and the adjusting screws controlling the gap between the rolls were calibrated by measuring the thickness of lead strips passed between the rolls at the same time as strips of coagulum. To characterise the intensity of machining to which a coagulum is subjected (at constant d.r.c. of coagulation), use has been made of a machining ratio (M.R.), calculated by dividing the gap between the rolls by the depth of latex in the coagulating pan. To avoid undue variations in latex depth when preparing thin coagula, the pans were constructed of heavy gauge aluminium which ensured a flat base not liable to distortion.

Observations on wet machined sheets showed the usual feature of ridges along the edges (an unavoidable effect when using a coagulum of rectangular section), and also showed that unless there is a plentiful supply of water to the rolls the width of coagulum near the leading edge is less than that near the trailing edge. Furthermore even with a good supply of water the part of the sheet near the leading edge was more variable in thickness than was the remainder.

Because of the effects of pH of coagulation on the behaviour of coagulum during machining (see Table 1) it was considered desirable to coagulate at constant pH with a view to

TABLE 1. SERUM CONTENT (% OF DRY RUBBER) AND THICKNESS OF DRY SHEET (MM) FOR 8 SHEETS MACHINED TO M.R. 0.032

Property	pH of coagulation 4.0	pH of coagulation 4.5
Serum content 5 min. after machining	30, 42, 48, 47 *Average 42*	59, 56, 53, 54 *Average 56*
Dry sheet thickness	2.03, 2.27, 2.17, 2.17 *Average 2.13*	1.99, 1.98, 1.93, 2.03 *Average 1.98*

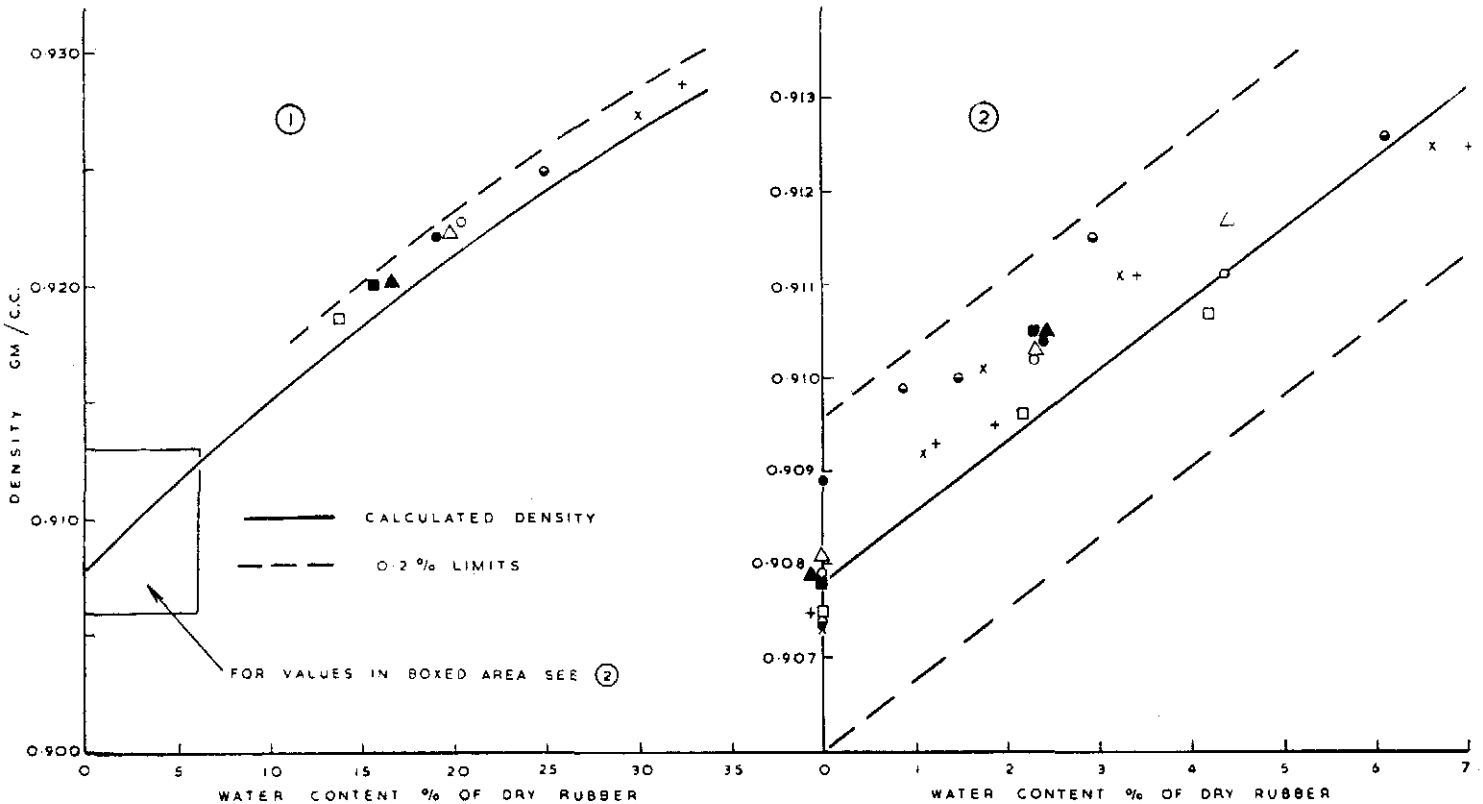

Figure 1. Density of nine samples of wet sheet rubber (c. 82°F), grams per c.c.

reducing day-to-day variation in the coagulum properties of a given latex.

Attention has been devoted almost entirely to coagula prepared from 15% d.r.c. latex and the emphasis has been on machining to a final M.R. of 0.032, since these conditions approximate to normal factory conditions.

Thickness measurement. The direct measurement of the thickness of freshly machined sheet is difficult owing to its softness and to the continued exudation of serum after it has left the rolls; in most cases therefore the thickness was calculated from the area of the sheet, its weight, and its density. The thickness of dry sheets was measured using a dial gauge mounted on a rigid arm above a cast-iron surface plate, the lower end of the gauge stem which is in contact with the rubber being 1 cm in diameter. The error introduced by the weight of the stem compressing the rubber was estimated by adding weights to the stem, taking thickness readings at each weight increment and extrapolating to zero weight the curve of thickness reading against total weight. The compression of dry sheets, ranging from 2 to 5 mm in thickness, caused by the weight of the stem alone was approximately 0.3%.

Dimensional Changes During Drying

A normal factory sheet, hung over a pole as in commercial rubber driers, shows a complex pattern of thickness variation. The most notable features, apart from the ridges formed by the edges of the coagulum, are the wide thin part in contact with the pole and the general tendency for increased thickness in the lower parts away from the pole.

Three mechanisms were thought to be responsible for this pattern—relaxation of machining stresses, shrinkage due to loss of water, and deformation caused by plastic flow of the sheet under its own weight—and each has been studied briefly.

Machining stresses. Rapid relaxation of the machining stresses in sample sheets machined to M.R. 0.032 was obtained with negligible water loss or effect of weight stress, by supporting the sheets horizontally on flat plates and exposing them to steam at atmospheric pressure. The shrinkage was 9% in length and 6% in width, causing an increase in thickness of about 17%.

Water loss. The shrinkage due to water loss was determined by measuring the density of sheets at various points in the drying cycle, using an immersion method similar to that described in B.S. 903; Part 14: 1950 (Procedure A), and the results for 9 samples are shown in Figure 1. The range of densities of the dry rubber (at equilibrium with laboratory atmosphere) is 0.9073 to 0.9089. WOOD (1938) quotes values from many sources at 77°F, the great majority of which lie in the range 0.905 to 0.919. Using Wood's selected value for the temperature co-efficient of volume expansion, this range becomes 0.903 to 0.917 at 82°F, the average temperature of our experiments, and the present results lie well within this range.

The solid lines in the Figure show the curve calculated on the assumption that the volumes of water and rubber are additive. The densities used for this calculation are 0.9960 for water (since only pure water is lost by evaporation) and 0.9078 (the average of the measured dry rubber values). All results are within 0.2% of the calculated curve. For all points above 1% water a much closer approach to the calculated curve would be obtained if this were calculated with a dry density 0.1% higher, but no conclusive explanation is offered for this.

The data show that as drying proceeds substantial voids are not left in the sheet; the rubber moves in to fill the space previously occupied by water.

Combined effect of machining and weight stresses. Assuming that shrinkage due to loss of water is uniform in all three dimensions, the linear shrinkage can be calculated from the above data. If the linear dimensions of a dry sheet are different from those calculated from the wet dimensions then deviations can be attributed to either machining or weight stresses.

Table 2 shows the actual dimensions as a fraction of those calculated assuming uniform

shrinkage for 8 sheets coagulated at 1 in. latex depth and pH 4.0 and pH 4.5, machined to M.R. 0.032, and dried at room temperature and at 120°F. The sheets were hung from one edge on a series of long hooks which were free to pivot, thus minimising restriction of width shrinkage. The wet dimensions were measured 5 minutes after the sheets left the rolls.

TABLE 2. ACTUAL DIMENSIONS OF DRY SHEET AS FRACTION OF CALCULATED DIMENSIONS. DEPTH OF LATEX 1 INCH

L = Length W = Width T = Thickness

Drying temp.	pH 4.0			pH 4.5		
	L	W	T	L	W	T
80–90°F	0.96	0.99	1.05	0.97	1.02	1.04
	0.98	0.99	1.03	0.98	1.00	1.03
120°F	0.96	0.99	1.08	0.96	1.00	1.07
	0.97	1.00	1.06	0.97	0.99	1.06

Although the accuracy of the experimental method is clearly not high, since the product of the length, width, and thickness values should be unity, the general effects can be seen. Owing to relaxation of machining stresses, both length and width are smaller than calculated and consequently the thickness is greater. At the higher temperature elastic stresses are more fully relieved, so giving a thicker sheet. No difference between pH's is established.

Table 3 shows further data for a range of thicknesses of coagulum all machined to M.R. 0.032 and dried at 120°F. The same effects are seen at all latex depths.

TABLE 3. ACTUAL DIMENSIONS OF DRY SHEET AS FRACTION OF CALCULATED DIMENSIONS. VARYING DEPTH OF LATEX

Latex depth, in.	L	W	T
½	0.94	0.98	1.09
¾	0.95	0.99	1.08
1	0.94	0.99	1.09
1¼	0.95	0.98	1.10
1½	0.94	0.99	1.09

The effect of a wider range of machining ratios is shown in Table 4.

TABLE 4. ACTUAL DIMENSIONS OF DRY SHEET AS FRACTION OF CALCULATED DIMENSIONS. VARYING M.R.

Final M.R.	L	W	T	Water content, % of dry rubber
0.14	1.10	0.94	0.95	151
0.094	1.06	0.97	0.99	107
0.067	1.04	0.98	0.96	81
0.047	1.01	1.00	0.98	57
0.032	0.98	1.01	0.99	41

There are definite trends in the width and length data which indicate that the weight stresses more than overcome the machining stresses at high machining ratios; in particular the actual length is greater than the calculated length.

Combined effect of water loss, machining stresses and weight stresses on small sample sheet. It was evident from the above that to produce a dry sheet of uniform thickness for experimental work it is necessary:

(i) to cut a sample sheet from the full sized wet sheet, avoiding areas known to vary in thickness
(ii) to minimise the effect of weight stresses by choosing a suitable length of sample and
(iii) to avoid a method of support which restricts width shrinkage.

Tests were therefore made to ascertain whether a sample sheet of 6 × 10 in. wet dimensions supported on four pivoting hooks 1½ in. long, would, under the effect of the various stresses, give a dry sheet of uniform dimensions.

Ten sample sheets were prepared by the standard method, machined to M.R. 0.032 and dried at 140°F; the thickness of each sheet was measured at 12 equispaced positions. The results showed no effect of weight stress, or of width restriction causing thinning near the top of the sheet, nor did further

storage of the sheets at 140°F for one week cause any reduction of thickness.

Sheets of these dimensions, supported in the way described, were finally selected for all experimental work on drying rates, the precise choice representing a compromise between many opposing factors. Average thickness nevertheless varied from 1.59 to 1.76 mm (± 5% of mean average) and statistical analysis showed significant differences (0.1% level) between the average thickness of individual sheets.

Intentional Restriction of Length and Width Shrinkage

Calculations showed that if contraction of width and length could be restricted the thickness would be reduced by about 20% compared with a sheet which is not restricted. A practical trial in which sheets were held in frames during drying confirmed this, and the drying rates of these sheets were correspondingly greater than those supported on hooks.

Summary and Conclusions

If weight stresses are negligible and shrinkage is not restricted, the surface area of a sheet (machined to M.R. 0.032 and containing say 40% water) is reduced by 14.5% owing to the relaxation of machining stresses and by a further 16% owing to water removal during drying. Relaxation of machining stress increases the thickness, and water removal reduces it; the overall effect is to produce a dry sheet 5% thicker than the original wet sheet.

The size of the sheet affects the weight stress, and the method by which it is supported affects the degree of shrinkage restriction. To obtain a sheet of uniform thickness for experimental work it is necessary to keep weight stresses low and not to restrict the width shrinkage at the top of the sheet which occurs if a pole is used as a support. This can be achieved by having a sample sheet 6 × 10 in. and supporting it on hooks which are free to pivot. Even using standardised coagulation, machining, sample cutting, and supporting methods there are still significant differences in dry thickness between individual sheets, and this emphasises the need for measuring the thickness of each sheet in a batch of nominally similar sheets.

The intentional restriction of shrinkage gives sheets of reduced thickness which, as a consequence, dry more rapidly. It is possible that, after further study, some means of using this effect to accelerate drying on a commercial scale could be found.

The measurement of shrinkage due to water removal has enabled the effect of machining and weight stresses to be assessed. The results have also suggested that since no voids are left in the rubber during water removal, capillarity is not much concerned in the mechanism of water transport within a sheet.

2. EFFECT OF DRYING CONDITIONS ON DRYING RATE

Before designing an experiment for a detailed examination of the effect of drying conditions, it is necessary to know the likely magnitude of the effect of extreme changes in the main variables (i.e., temperature, humidity and air speed) on the various stages of drying.

The classical theory of the drying of solids by evaporation recognises three phases of drying:

(i) A 'constant rate' period when the surface is completely wetted with water
(ii) A period of unsaturated surface drying when only part of the surface is wetted
(iii) A period during which the surface is almost dry and water diffuses slowly from the wet interior to the surface

The transitions between the first and second, and second and third phases, are termed the first and second critical points respectively.

PIDDLESDEN (1937) observed that the initial part of the drying curve for rubber was approximately linear, and that the drying rate was rapidly reduced below 10% water content. He concluded that the surface of the sheet was saturated with water in the initial stages and that the second critical point occurred at about 10% water. In observations of the behaviour of sheet in commercial

driers, however, it was noticed that dripping which had almost ceased at room temperature, was renewed when the sheet was placed in the drier. The possibility of condensation from the air in the drier could not be ruled out entirely, but it seemed more likely that spontaneous exudation or syneresis, similar to that found by SCHOON (1953) for unmachined coagula, was responsible and that it provided another mechanism by which a sheet could lose water in the initial stages.

The experiments recorded below fall into three categories: evaporation in the phase down to about 10%, syneresis, and drying below 10%.

Evaporation in the Initial Period of Drying

Earlier workers considered that the approximate linearity of the water content/time plot indicated that the effects of temperature and humidity on the drying of sheet would be similar to those on evaporation from free water surfaces. To establish this more fully the initial phase has been studied in more detail.

Experimental procedures. Studying this period presents some difficulties because of the very rapid rate of drying. Weighing the sheet in the air stream introduces weighing errors and frequent removal during drying interrupts the drying unduly. Another method which can be used to study changes in drying rate is to measure the temperature difference between the sheet and the drying air; at constant air speed and in the absence of radiation effects, the rate of heat transfer, and thus the rate of evaporation, is proportional to this temperature difference. The method has the disadvantage that absolute drying rates are not measured and accuracy becomes poor at low rates of evaporation. It is however a useful method of investigating the shape of the drying curve at high rates of evaporation, and has been used in these investigations.

A thermocouple made of 40-gauge wires was inserted into the latex in a coagulating pan just after acidification. The coagulum was then carefully machined with the wires in position. This gave a wet machined sheet with the thermocouple firmly embedded in the centre of the sheet, the wires emerging from the edge of the sheet. A further thermocouple to measure the air temperature was then attached. The current flowing in the opposed couple circuit was measured with a galvanometer, which gave a direct reading of temperature difference between the sheet and the air.

Discussion of results. Figure 2 shows the measured temperature difference for sheets dried at 140°F/61% R.H. and 137°F/16% R.H. The almost constant initial values equal to the difference between the wet and dry bulb temperatures of the air confirms that during this period the surface is saturated with serum of a vapour pressure almost equal to that of pure water (i.e. the sheet is behaving like a wet bulb thermometer). The sharp downward turn at the end of the constant temperature difference period is clearly the first critical point. The next section of the curve represents falling temperature difference: it is likely that the end of the straight portion of this section corresponds with the second critical point; this occurs when the temperature difference is about half the initial value. In a further experiment drying was stopped at this level of temperature difference and the water content was found to be 9%.

Data from experiments in which temperature measurements were not made but in which weight loss was measured suggested that the first critical point occurs at about 15% water content.

Syneresis

During the high humidity experiment described above (Figure 2) serum dripped from the lower edge of the sheet during the first part of the constant temperature difference period, and the mechanism of this effect was considered to be worth investigating in more detail. To eliminate evaporation effects the loss of weight of freshly machined sheets was studied at 100% R.H.

Experimental procedures. 7 × 3 in. samples were cut from sheets and hung in pairs in sealed 2-litre bottles each containing 500

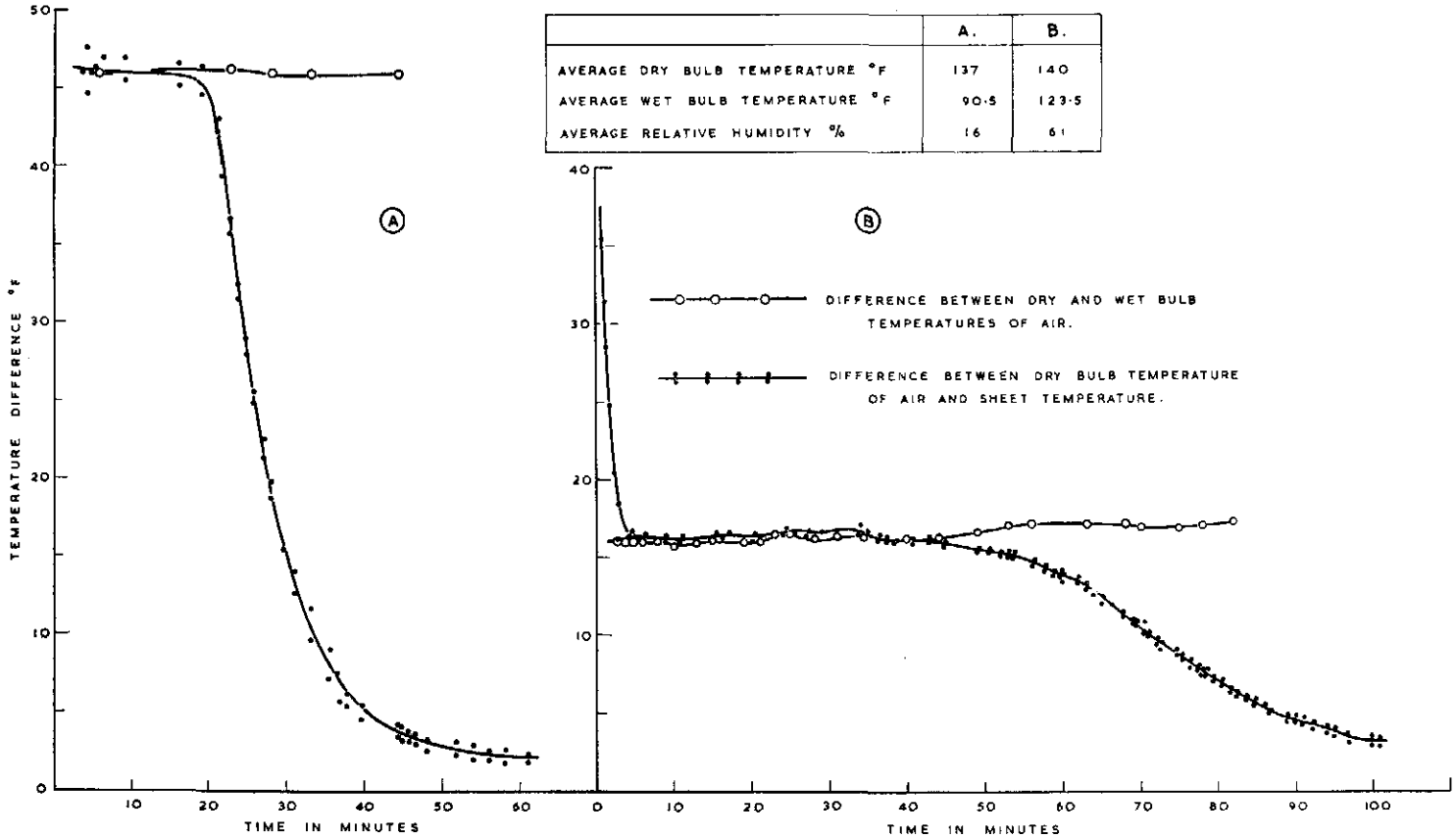

Figure 2. Temperature difference between sheet and air during initial stages of drying at 137°F/16% R.H. and 140°F/61% R.H.

c.c. of water. After about two hours some bottles were placed in an oven at an elevated temperature while others remained at room temperature. The bottles in the oven were lightly sealed during the heating period and were finally tightened when top temperature had been reached. The samples were removed periodically for weighing. Preliminary tests showed that removal for weighing did not seriously affect the measured weight loss.

Results. The following Tables (5 and 6) show the loss of serum for sheets machined to two levels of M.R., at two levels of pH. The results are plotted in Figure 3.

TABLE 5. SERUM CONTENT (PERCENTAGE OF DRY RUBBER) OF PAIRS OF SHEETS AFTER VARYING PERIODS OF STORAGE AT 100% R.H. M.R.0.094

Time hrs	Temperature	pH			
		4.0		4.5	
0	Room temp. c.85°F	113.0	121.0	140.0	153.0
2		95.6	104.0	120.0	134.0
4½		90.5	99.5	111.0	125.0
23		78.9	83.0	98.3	108.0
28½		76.6	82.2	95.3	105.0
47		73.1	78.4	87.2	94.5
0	c.85°F	119.0	114.0	148.0	143.0
2		101.0	95.5	133.0	129.0
4½		86.1	83.8	111.0	108.0
23	140°F	60.2	59.2	78.7	78.2
28½		59.3	58.7	77.1	76.9
47		57.2	56.9	73.7	72.6

TABLE 6. SERUM CONTENT (PERCENTAGE OF DRY RUBBER) OF PAIRS OF SHEETS AFTER VARYING PERIODS OF STORAGE AT 100% R.H. M.R.0.032

Time hrs.	Temperature	pH			
		4.0		4.5	
0	Room temp. c.85°F	47.4	50.9	61.3	68.4
2		33.3	36.1	45.7	49.7
4½		30.1	33.2	42.1	46.2
23		25.7	28.5	37.4	41.3
28½		24.2	26.9	36.1	39.7
47		22.3	25.2	32.4	35.4
0	c.85°F	47.9	48.0	64.3	58.9
2		36.7	36.3	48.8	44.7
4½		27.5	27.1	37.7	34.4
23	140°F	16.2	20.0	29.0	25.4
28½		15.7	19.8	28.3	25.3
47		15.2	19.3	27.7	24.8

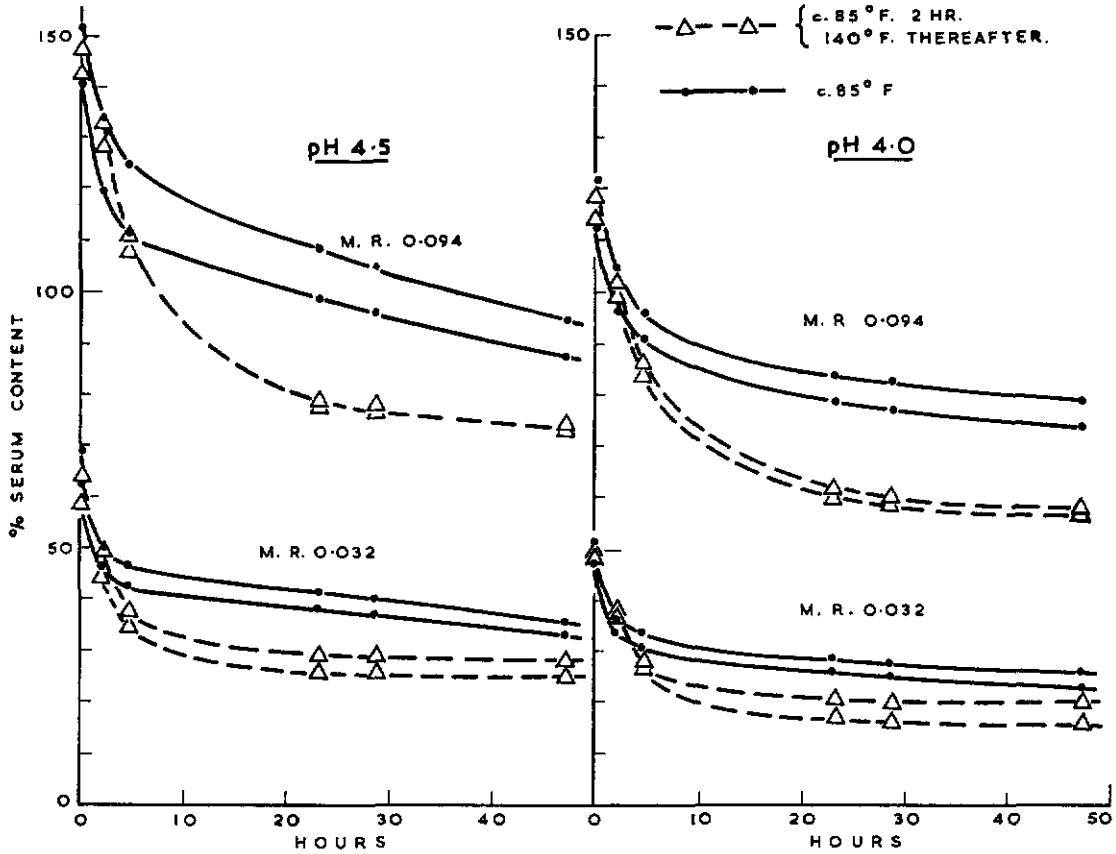

Figure 3. Exudation of serum from machined sheets. Serum content expressed as percentage of dry rubber.

Discussion of results. Sheets machined to M.R. 0.032 show losses of 11–19% during 2 hours at room temperature. During a further 2½ hours the extra loss at room temperature is 2.9–3.6%, and at 140°F 9.2–11.1%. This latter rate of loss is of the same order as the total initial rate of loss, by all mechanisms, obtained in commercial natural-convection driers operating at 140°F, and is therefore likely to contribute to the water loss in such driers.

The data also have some interesting features which give indications of the mechanism of syneresis. Sheets machined to M.R. 0.032 and held at 140°F almost reach equilibrium values, and those at room temperature could well be tending to the same average equilibrium value. Sheets with a high serum content do not reach equilibrium at either of the temperatures, but they are tending towards an equilibrium which is at a much higher level than for sheets with an initially low serum content. Each sheet however seems to have a separate equilibrium value which depends on its initial serum content. This applies for all sheets whether the variation in water content is due to differences in pH of coagulation, intensity of machining, or to random variations within the single sheet from which the sample sheets were cut. As also observed in the data in Table 1 the effect of increasing

the pH from 4.0 to 4.5 is to increase the serum content after machining.

A tentative explanation of this latter effect is that the coagulum prepared at pH 4.5 is softer and spreads out more easily during machining and is therefore subjected to less compressive stress than the firmer coagulum at pH 4.0.

It seems that the extent to which syneresis can occur depends to a large extent on the stresses set up during machining.

Drying Below 10% Water Content

Minor variations in serum composition or changes in the method of preparation are unlikely to affect the rates of drying in the constant rate period and it is probable that their effect would be noticed mainly in the diffusion-controlled period below 10% water content. In studying this phase in the drying process some attention was therefore given to measures for controlling the variation in non-rubber content and composition so as to reduce the day-to-day variability in drying rates. Such variations are considerable between latices from different clones and localities. The type of tapping cut and the time from the opening of a new tapping panel also influence the composition of the latex. An area of the Rubber Research Institute Experiment Station at Sungei Buloh (Tasks 7 and 8 of Field 25, Section V) was therefore selected as a source of latex for the experiments. The area was regarded as suitable because it was planted in 1931 with trees grown from seeds selected from a large number of high yielding trees on estates, and would be expected to give a fairly 'average' latex; also no manuring, stimulation or opening of a new tapping panel was expected for several years after the start of our experiments.

The control of pH was considered essential because of the effect of pH on the retention of non-rubber in sheet (LIPONSKI AND D'AUZAC 1955).

Experimental procedures. A preliminary experiment indicated that there might be some relationship between drying time and the order of acidification of the latex in the series of coagulating pans. To eliminate any doubts on this score an apparatus was constructed to enable two operators to acidify 16 pans of latex with an interval of only 15 seconds between the first and last pans, and to stir them simultaneously.

Coagula were prepared from the standard latex at a latex depth of 1 in. and pH 4.0, and machined to M.R. 0.032. Sample sheets were hung in the manner described in Section 1 for a fixed period at room temperature, and dried in an apparatus which enabled sheets to be weighed in a still atmosphere of temperature and humidity similar to that of the drying zone. The ten positions in the drying zone each had a separate air speed, the range of values being from 75 to 400 feet/minute.

Results. Figure 4 shows the results of three drying experiments carried out on different days, in each of which 10 sheets were dried at one time. The drying conditions for the three experiments were:

190°F, 5% R.H. (normal atmospheric absolute humidity)

140°F, 15% R.H. (normal atmospheric absolute humidity)

140°F, 70% R.H.

The units of the abscissa (time, θ, divided by the square of the thickness, t) provide an approximate correction for variations in thickness between individual samples. The curves give the outer limits of all points obtained with one batch of 10 sheets. The following Table gives the range of thicknesses, and the values of θ/t^2 for drying from 10% to 0.5% water, together with the percentage deviation.

Discussion of results. From Table 7 it can be seen that raising the temperature from 140°F to 190°F at constant absolute humidity has reduced the drying time by a factor of 2.65. Some of this effect is no doubt due to the reduced relative humidity at the higher temperature, since reducing the relative humidity from 70% to 15% at 140°F has reduced the drying time by a factor of 2.7, but no complete separation of the effects can be obtained from the present data.

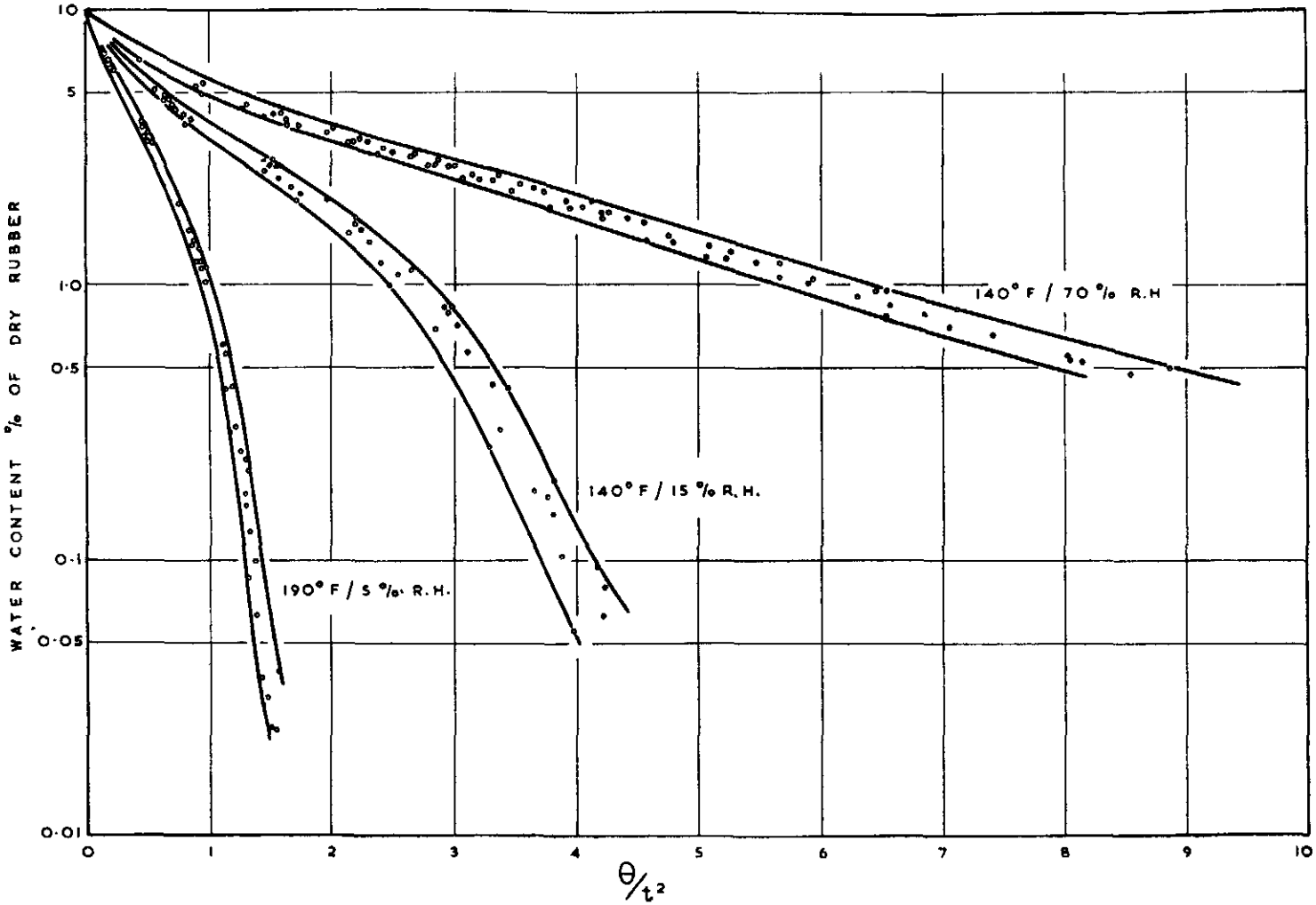

Figure 4. Drying below 10% water content; various drying conditions.
θ = *time in hours.* t = *thickness in millimetres.*

TABLE 7. DRYING BELOW 10% WATER UNDER VARIOUS DRYING CONDITIONS

θ = *time in hours.* t = *thickness in millimetres.*

Drying conditions	190° F/5% R.H.	140°F/15% R.H.	140°F/70% R.H.
Range of average sheet thickness, mm	1.98 to 2.15	1.59 to 1.76	1.75 to 1.88
Average θ/t^2 at 0.5% water, and % deviation	1.14 ± 4%	3.15 ± 6%	8.43 ± 5%

The very marked effect of relative humidity is of the greatest importance since it indicates that in commercial drying there will be an optimum humidity to give the most favourable economic balance between capital and fuel costs.

The range of variation in the corrected drying times between the 10 sheets in a single batch is only of the order of 10%. This variation is made up of experimental errors in the measurements of time, thickness, and water content, the possible inaccuracy in using the square of the thicknesses for correction of the time, and a variation in air speed ranging from 75 to 400 feet/minute. It can therefore be concluded that the effect of this range of air speeds is very small.

Conclusions

From temperature measurements of sheet during the initial stage of drying it can be predicted confidently that the evaporation rate during this stage will conform to the equations applicable to the evaporation from free water surfaces. The main features of such equations (PEARSE, OLIVER AND NEWITT 1949) are that the rate of evaporation per unit area will, in the absence of radiation or conduction effects, be determined by the difference between the wet and dry bulb temperature of the air, the air speed, and the dimensions of the sample.

The rate of loss of serum and/or water will however be affected by dripping from the sheet, which occurs when the rate of evaporation is less than the rate of syneresis. The balance between these two rates under different drying conditions has not been examined in detail but it is clear that raising the humidity at constant temperature and air speed will decrease the rate of evaporation and increase the rate of syneresis owing to the higher sheet temperature. It can therefore be expected that the variation in the rate of loss of water (particularly at low air speeds) will be less than if evaporation were the only mechanism of water loss, since at high humidities dripping will partially offset the reduced evaporation rate. Some insight into the mechanism of syneresis has also been obtained.

In the period below 10% water content when diffusion is the controlling factor, the effect of temperature (140°F to 190°F) is of the order to be expected from earlier work in a lower range of temperature, but the effect of humidity (15% to 70% R.H.) is unexpected and runs contrary to previously held views. The effect of air speed in the range 75 to 400 feet/minute appears to be small.

Calculations not presented in this paper indicate that from the point of view of thermal efficiency high temperatures are likely to be more economic, and that there would be an optimum humidity for the most economic operation.

It has thus been concluded that in future investigations the accent should be placed on temperatures higher than the normal 140°F and that the effect of humidity should be examined in more detail.

3. EFFECT OF RELATIVE HUMIDITY ON EQUILIBRIUM WATER CONTENT

Since it was considered that the effect of humidity on drying rate described in Section 2 was almost certainly associated with the change in equilibrium moisture content of the rubber, a brief examination of this property in freshly prepared air dried sheet was made.

Experimental procedures. Latex was diluted to 15% d.r.c. and coagulated at 1 in. latex depth and pH 4.5 with 1% formic acid, and machined to M.R. 0.032. From each of the sheets from seven coagulating pans four smaller sheets were cut. These twenty-eight sheets were assigned randomly to the various subsequent treatments. The sheets were dried at 140°F and then further dried to equilibrium over freshly dried silica gel at room temperature.

The dried sheets were then exposed to atmospheres of a range of constant relative humidities, obtained by using saturated solutions of various salts (WEXLER AND HASEGAWA 1954, WINK AND SEARS 1950). Half the sheets were maintained at 72°F, and the other half at 140°F. The samples were removed for weighing at intervals of several days. To correct for any absorption or desorption during weighing, readings were taken at short intervals and the required weight was obtained by extrapolating the readings to zero time.

In earlier preliminary experiments, mould had grown on the sheets at relative humidities above 80% and some additional sheets were soaked for 15 minutes in a 0.1% solution of *para*-nitrophenol after machining, and tested for comparison.

Results. The results are given in Table 8. The data show significant differences in water absorption due to humidity changes, but the picture is complicated by the occurrence of mould growth and the side effects of the *para*-nitrophenol treatment. The latter was effective in preventing mould growth at all

TABLE 8. EQUILIBRIUM WATER CONTENT. PERCENTAGE OF DRY RUBBER

Salt	Relative humidity % at 72°F	Relative humidity % at 140°F	Water content, %d.r. at 72°F	Water content, %d.r. at 140°F	Additional treatment
Potassium sulphate	97.0	—	2.61*	—	Nil
			2.72*	—	Nil
			2.04	—	P.N.P.
			2.00	—	P.N.P.
Potassium nitrate	93.0	—	1.31*	—	Nil
			1.49*	—	Nil
			1.06	—	P.N.P.
			1.08	—	P.N.P.
Ammonium sulphate	80.5	79.0	0.71	0.82	Nil
			0.69	0.87	Nil
			0.66	—	P.N.P.
			0.51	—	P.N.P.
Sodium chloride	75.5	74.0	0.58	0.77	Nil
			0.55	0.67	Nil
Magnesium nitrate	54.5	43.5	0.28	0.27	Nil
			0.25	0.28	Nil
Magnesium chloride	33.5	31.0	0.16	0.12	Nil
			0.16	0.15	Nil
Lithium chloride	12.0	11.5	0.06	0.02	Nil
			0.06	0.00	Nil

* Indicates presence of mould growth.

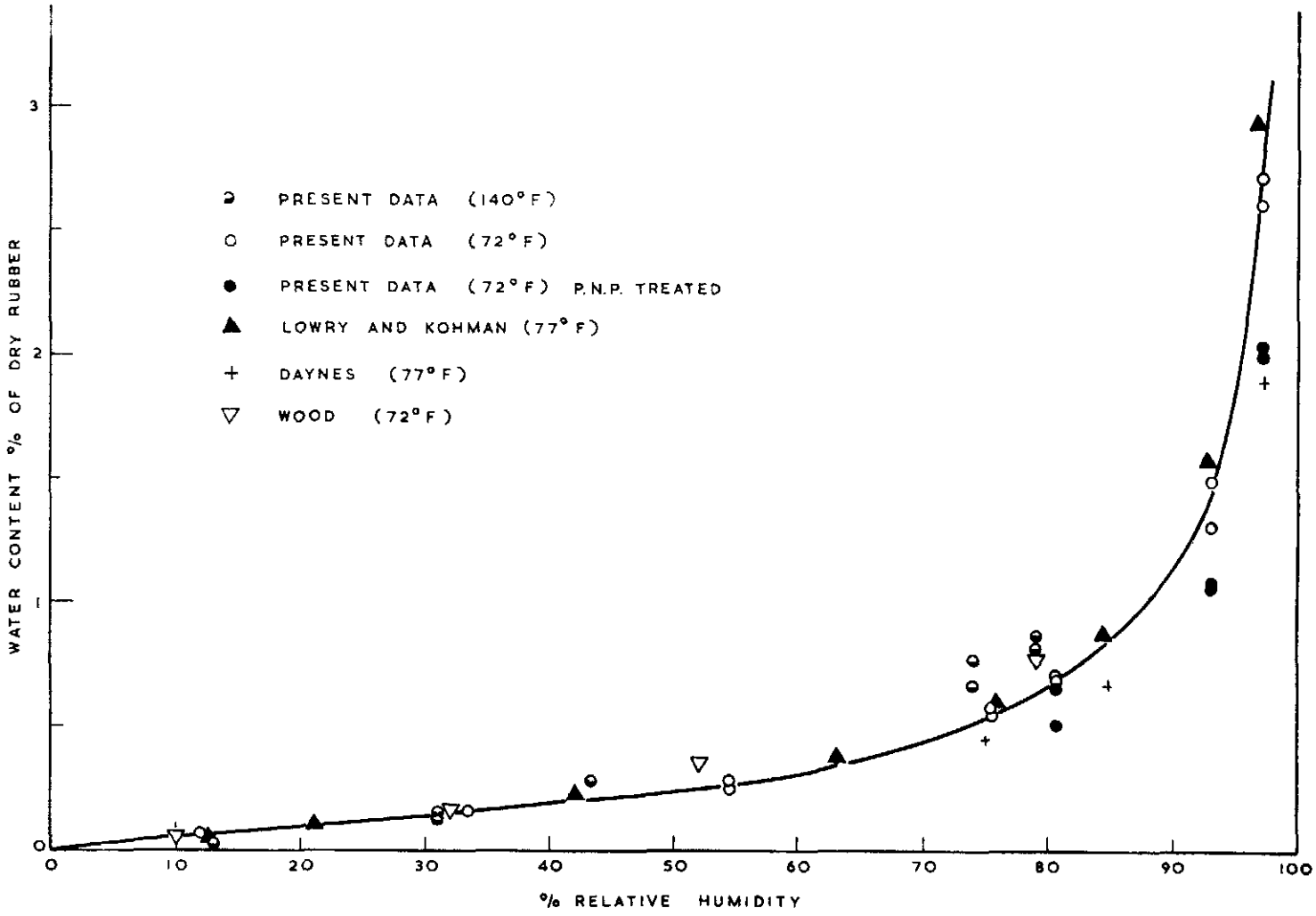

Figure 5. Equilibrium water content of sheet rubber.

humidities, but at 80.5% R.H. where neither treated nor untreated sheets supported mould growth, the water absorption was lower for the treated sheets. The data for treated sheets at 80.5% R.H. and above cannot therefore be regarded as an extension to higher humidities of the data for untreated sheets at 80.5% R.H. and below.

The results are plotted in Figure 5 and compared with data from other sources.

Our data show good agreement with those of LOWRY AND KOHMAN (1927) for smoked sheet, and the data of DAYNES (1932) and WOOD (1952) follow the same general trend. The points for Wood's data were calculated from the change in weight after 30 days.

No great accuracy is claimed for the high temperature results but they do at least show that the effect of raising the temperature is likely to be small.

Discussion and Conclusions

The data have shown that the equilibrium water absorptions of freshly prepared air dried sheet are similar to those of fairly fresh milled smoked sheet (WOOD 1952), and also to those of sheet used in investigations in consuming countries which is likely to have been stored for a period. It is therefore possible to draw some conclusions in relation to our work from the more extensive work of previous investigators in the consuming countries.

In particular LOWRY AND KOHMAN (1927) showed that intensive water washing of commercial smoked sheet reduced the water absorption at 98% R.H. almost in proportion to the weight of water extractables remaining in the rubber. On this basis the amount of washing received by wet rubber during and after machining should be carefully controlled if uniform hygroscopic properties are to be obtained.

In the experiments reported above all samples were translucent. The point at which a sheet becomes translucent is therefore not a satisfactory criterion for the end point of the drying operation from an experimental point of view, although it is the one used in commercial practice. A more satisfactory method is to determine the point at which the sheet reaches that water content which it would have when in equilibrium with a constant relative humidity. A relative humidity of 60% was chosen as approximating to the minimum atmospheric humidity in Malaya. In commercial operation any drying below this water content would not be of great use since the sheet, once removed from the drier, would re-absorb water from the atmosphere.

4. DETERMINATION OF THE WATER CONTENT OF SHEET RUBBER

The data in Sections 2 and 3 showed that the large decrease in drying rate caused by changing the humidity from 15% R.H. to 70% R.H. is associated with a change in equilibrium moisture content of about 0.4%.

Since in the main investigation it was planned to attempt to correlate drying rate with equilibrium moisture content, a convenient method was required for determining the dry weight of the samples on which to base the calculation of water content.

The direct method of moisture determination by azeotropic distillation was regarded as unsuitable, since only small samples can be conveniently handled and thus sampling from the sheets presents an added difficulty. Attention was therefore devoted to methods in which the whole sheet is weighed before and after equilibrating with an atmosphere having very low water-vapour pressure. These methods have the added advantage that they only determine the free moisture, i.e. the moisture which can be removed by air drying.

The problem resolves itself mainly into finding a convenient way of obtaining the low humidities required to reduce the equilibrium moisture content to a sufficiently low level.

Vacuum Drying and Drying with Desiccants

Experimental procedures. The vacuum apparatus consisted of a glass vacuum line using both mechanical and mercury diffusion pumps, to which the tubes containing the

TABLE 9. AVERAGE EQUILIBRIUM WATER CONTENT

Percentage of dry rubber at c. 85°F

In equilibrium with	d.r.c. of latex at coagulation	
	10%	25%
Vacuum 0.01 mm mercury	Assumed zero	Assumed zero
Silica gel containing 6.1% water	0.08	0.09
Silica gel containing 12.5% water	0.14	0.16
Laboratory atmosphere	0.36	0.44
R.H. c.80% (saturated ammonium chloride)	0.61	0.80

samples could be fitted. Pressure readings were taken with a MacLeod gauge. After pressure equilibrium had been reached the glass connecting-tubes were sealed off with a blow torch. The seals were broken immediately before placing on the balance pan for weighing, and readings were then taken at short time-intervals. The weight immediately after breaking the seal was obtained by plotting weight against time and extrapolating to zero time.

Desiccants were regenerated by heating to constant weight at 570°F for silica gel and 390°F for activated alumina. Whenever rubber samples or desiccants were weighed either tubes which could be rapidly sealed were used, or the weighings were corrected by using the plot of weight against time. Saturated salt solutions were used to give atmospheres of constant relative humidity. 75% sulphuric acid solution was used to obtain an atmosphere of 1.8% R.H. at 85°F, the relative humidity being calculated from vapour pressure data given by PERRY (1950).

Comparison of Vacuum Drying with Silica Gel and Alumina Drying

Vacuum drying yields the most reliable data but it is time-consuming and not easily adaptable to the handling of large numbers of samples. An experiment was, however, carried out to compare vacuum drying at a pressure of 0.01 mm mercury with drying over silica gel. Since it was suspected that the hygroscopic nature of the final sheet would vary with dilution of the latex, sheets were prepared by coagulation at 10% and at 25% dry rubber content, which represent the extreme limits for normal coagulation techniques. Both types of sheet had a nitrogen content of 0.34%.

The results in Table 9 show that the sheets coagulated at 25% d.r.c. are more hygroscopic than those coagulated at 10% d.r.c. Silica gel containing 6% of water reduces the water content of both types of sheet to less than 0.1%, assuming that vacuum drying gives the true dry weight. Silica gel and activated alumina were then compared for their effectiveness as desiccants. Table 10 shows the water absorptions of the desiccants at various relative humidities.

In Figure 6 the results are compared with those obtained by other workers. There are considerable differences between the data of CARTER (1951) and the present data, which are possibly due to the difference in temperature. The silica gel data also differ from those of RAO (1941) but SING and MADELEY (1954) have shown that large variations occur in the water absorbing capacity of silica gel, owing to differences in the conditions of preparation, particularly the pH. In these experiments, normal commercially available grades of desiccant were used.

The results show that the activated alumina is far superior to silica gel, and at 5% water (a convenient level for avoiding too frequent regeneration) will give a relative humidity of about 2.5%. The water content which a

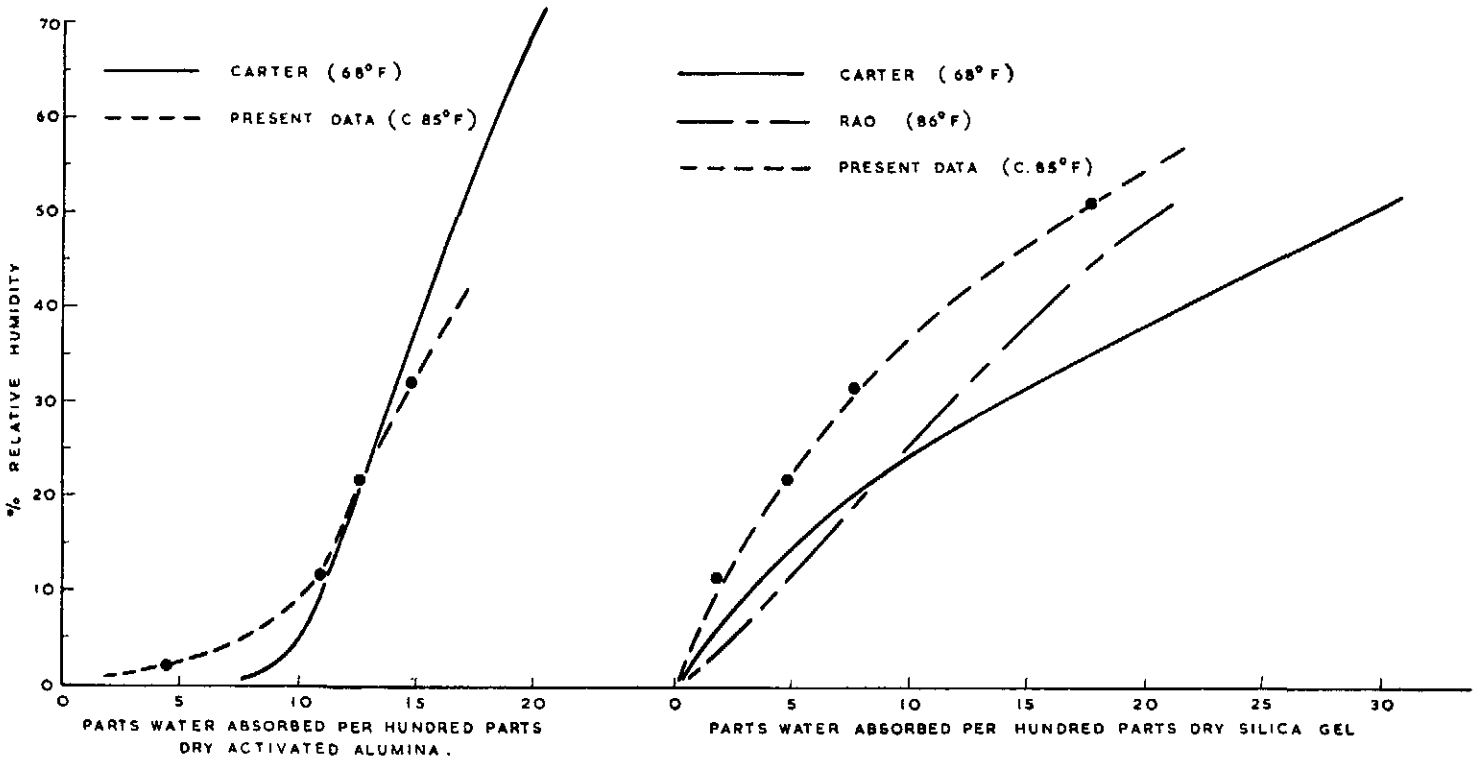

Figure 6. Water absorption of activated alumina and silica gel.

TABLE 10. WATER ABSORPTION AT c. 85°F

Percentage of dry desiccant

Relative humidity	Water absorption	
	Silica gel	Activated alumina
1.8%	—	4.5%
12.0%	1.9%	10.9%
22.0%	4.9%	12.6%
32.0%	7.7%	14.8%
52.0%	17.7%	—

rubber sheet will have at this humidity can be estimated from the data in Table 9 on the assumption that the water absorption is linearly related with relative humidity in the low humidity region. That this assumption is valid, has been shown by the work of LOWRY AND KOHMAN (1927) and approximately confirmed by the data in Table 8 in Section 3.

The relative humidity in equilibrium with silica gel containing 6.1% water is 26% (Figure 6), which gives a water content of the rubber of 0.08 to 0.09% (Table 9). At 2.5% R.H. (activated alumina containing 5% water), the water content of the rubber would be less than 0.01%.

Oven Drying and Drying over Activated Alumina

Although the use of activated alumina reduces the water content of sheet to a suitably low value at room temperature, it is possible that at higher temperatures and a similar humidity the sheet might liberate more water. If this were the case it would be preferable to use a higher temperature for determining the dry weight of a sample. The position is complicated however by the loss of volatile material other than water. CHAMBERS (1951) showed that oven drying in the United Kingdom at about 160°F causes a loss of volatile material other than water of about 0.05% for smoked sheet, and WOOD (1952) observed that approximately 0.1% of additional moisture and/or volatile matter is removed by heating rubber at 185°F after it has reached equilibrium over calcium chloride at room temperature.

The maximum temperature of drying envisaged was 200°F and this temperature was therefore selected as potentially the most useful temperature for oven drying of the samples, since the water removal would be greater than or equal to the removal obtained in the main drying investigation.

A comparison was therefore made of oven drying at 200°F/4%R.H. and drying over freshly dried activated alumina.

Experimental procedures. Oven drying was carried out in a constant-temperature chamber with forced circulation. A special weighing device enabled weights of the sheets to be measured in a compartment at the same temperature and humidity as the air used for drying. Corrections were applied for buoyancy effects caused by the density of air surrounding a sheet being different from that of the ambient air.

Eight sheets were prepared by the normal method and dried at 100°F and 60% relative humidity. Half of the sheets were then brought to equilibrium over activated alumina containing less than 1% of water and half brought to equilibrium at 200°F and the loss in weight measured. All samples were then returned to equilibrium at 100°F and 60% relative humidity and the reabsorption of moisture measured.

Results. The results given in Table 11 indicate that the oven drying at 200°F causes a greater loss in weight than drying over

TABLE 11. PERCENTAGE LOSS AND RECOVERY OF WEIGHT IN RUBBER SAMPLES

Activated alumina		Oven drying at 200°F	
% lost	% regained	% lost	% regained
0.27	0.28	0.63	0.30
0.20	0.28	0.79	0.37
0.23	0.29	0.79	0.33
0.24	0.24	0.61	0.32

activated alumina. The recovery of weight in the latter case is similar in magnitude to the loss (i.e. the moisture absorption is reversible), but the high temperature treatment produces a loss which is only partly reversible.

There are two possible explanations of this. The additional loss could be mainly volatile matter and about 0.05% water (the difference between the percentage weights recovered) or the additional loss could be entirely water, and the lower recovery of weight could be explained by a reduction in the water absorbing properties of the rubber caused by the high temperature treatment.

In any event the distinct possibility that there is a loss of about 0.4% of volatile material other than water renders oven drying at 200°F a doubtful method of water content determination.

Conclusions

The method selected for the determination of water content of air dried raw rubber sheets is the weighing of samples before and after equilibrating with an atmosphere in contact with activated alumina containing not more than 5% water. This brings the weight of the sample to within about 0.01% of the dry weight, which can be defined as the weight it would attain when vacuum dried at 0.01 mm mercury and 85°F.

The value of moisture content obtained is somewhat arbitrary since it is possible that a sheet dried over activated alumina might lose more water if its temperature were raised to say 200°F. The latter procedure is not desirable since there is a possibility that volatile material other than water would be removed at the same time.

The results also present a new factor to be taken into account when the effect of high temperature drying is considered. The sheets oven-dried at 200°F were held at this temperature for 10 days before equilibrium was reached. The period during which the loss of water-absorbing properties or of volatile material occurred is not known, but if either loss occurred during the initial period required to bring the sheets to an acceptable commercial level of dryness, one effect of high temperature drying would be to reduce the yield of product; this requires further investigation.

5. EFFECT OF DRYING CONDITIONS ON BLISTER FORMATION

Possibly the greatest limitation to the use of high temperatures, under the present market system of grading rubber, is the formation of blisters in the sheet. The mode of formation is not fully understood, but it is convenient to record here observations which give some indication of the mechanisms involved.

The Nature of the Gas and its Source

In these experiments a simple Orsat apparatus was used for determining oxygen and carbon dioxide contents of the gas samples. The volumetric accuracy of the apparatus was checked by determining the oxygen content of air, when dry and when saturated with water vapour. Oil was used in the measuring system instead of water. The value obtained for wet air was 19.7% oxygen, and for dry air 20.6% (theoretically 21%).

Blisters produced by drying sheets at 200°F were clipped under water and the gas collected. The analysis of the gas was 0.6% carbon dioxide, 19.5% oxygen and 79.9% of residual gas. The quantity of gas collected on occasions, however, amounted to as much as 0.3 c.c. per gram of serum originally present in the machined sheet, which is many times more than the solubility of air in water, 0.015 c.c. per gram at 86°F (COMEY AND HAHN

1921); thus the possibility appears to be ruled out that the blisters were caused merely by the release on heating of dissolved air from the serum.

The possibility of air diffusing in to replace the gas which originally formed the bubbles was examined. Blisters were blown in partially dried sheet by injecting carbon dioxide with a hypodermic syringe. The sheets were dried for two days at 120°F and the blisters clipped under potassium hydroxide solution. The presence of residual gas indicated the presence of air in the blisters.

In order to work with larger quantities of gas a sealed bag was made from sheets of machined coagulum. Using a hypodermic needle attached to a carbon dioxide cylinder, the bag was alternately inflated and deflated several times to expel the air, and then filled. The hypodermic needle was withdrawn and the point of entry sealed. The walls of the bag were translucent after three days at 120°F and the contained gas gave an analysis of carbon dioxide 37%, oxygen 14%, residual 49%, an analysis approximating closely to a mixture of air (63%) and carbon dioxide (37%). Since it was clear that air had passed through the walls of the rubber bag, even with a surface/volume ratio far smaller than any blister and a much lower temperature than that used in blistering experiments, it was concluded that further analyses of the gas from blisters in dried sheets were of little value.

One other result of this investigation is that gas recovered by heating two-day-old sheet serum was found to contain 66% carbon dioxide, which suggests that this gas may be largely responsible for blister formation: the solubility of carbon dioxide in water is about 0.66 c.c. per c.c. of water (KAYE AND LABY, 1918), which is much more in line with the observed volume of blisters.

Methods of Reducing or Eliminating Blister Formation

In an initial experiment a sample sheet from a coagulum prepared at pH 4.0 and machined to M.R. 0.032, and then steamed at atmospheric pressure, did not blister when subsequently dried at 200°F although there were several tiny blemishes on the surface.

This suggested that in the wet state the coagulum was sufficiently porous to allow the escape of the greater part of the gas during steaming. Plentiful blisters occurred with sheet that was not steamed, and it was noticed that these appeared during the first hour or two of drying.

Table 12 which gives the results of a further trial clearly demonstrates the effect of sheet thickness. The coagula were prepared at pH 4.0 and all machined to M.R. 0.032.

TABLE 12. EFFECT OF THICKNESS ON BLISTER FORMATION

Latex depth in.	Sheet thickness mm	Occurrence of blisters
1.5	2.7	Many
1.0	1.8	Few
0.5	0.8	None
0.16	0.3	None

The combined effect of thickness and humidity of drying is evident from Figure 7 which shows sheets prepared from coagula at latex depths of 0.87 in., 1.2 in., and 1.5 in. and dried at 200° F and relative humidities of 25%, 40%, 55% and 70%.

Conclusions

Although there may be considerable difficulties in preventing the formation of the type of blister encountered in these experiments at high temperatures, it appears to be technically possible to overcome them.

The mechanism of formation is not completely clear but a tentative explanation for the appearance of blisters during the first few hours of drying and the effect of humidity and thickness is as follows.

The sheet immediately before drying contains considerable quantities of dissolved gas and/or substances which decompose to yield a gas. The gas can be liberated before drying, without forming blisters, by heating the sheet while the surface is still wet to a temperature

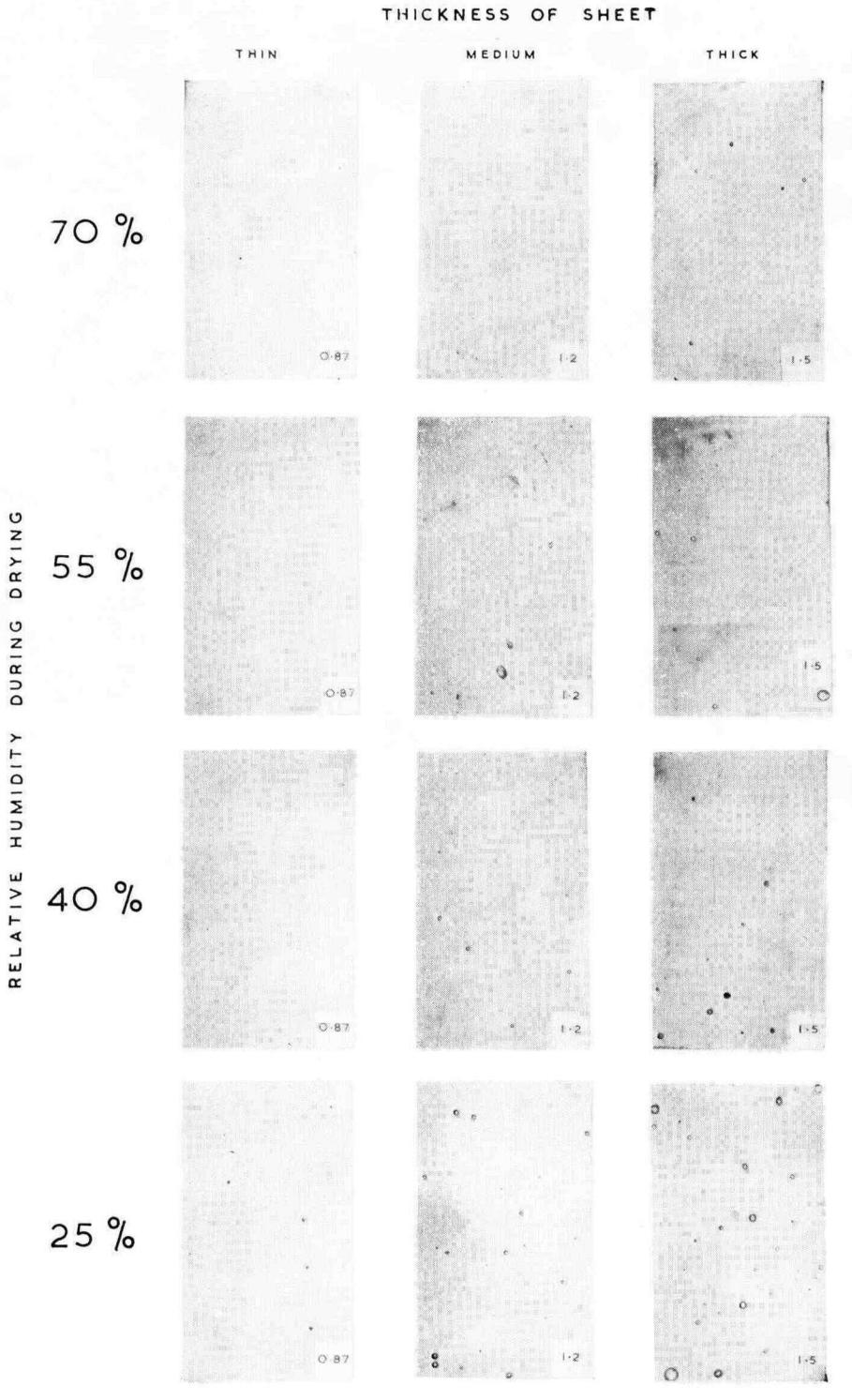

Figure 7. Blister formation at 200°F.

equal to or greater than the temperature of drying; steaming is an example of this.

With drying at low R.H. the wet bulb temperature—and thus the sheet temperature at the start of drying—is low, and gas remains dissolved in the serum. When the water content of the sheet has been reduced to about 15% there is a sudden rise in temperature which corresponds with the surface of the sheet drying out (e.g. Curve A of Figure 2). The rising temperature progressively increases the rate of expulsion of gas from solution and, at the same time, a relatively impermeable skin of dry rubber is formed on the surface so that conditions are provided for the development of blisters.

If a higher humidity is used the sheet temperature is higher (e.g., Curve B of Figure 2) and the sheet remains at this temperature in the wet conditions so that some gas is liberated without blistering. When the temperature rises at the first critical point there is less gas remaining to be liberated and blistering is reduced. The progressive decrease in blistering with increasing humidity is therefore associated with the progressive increase of wet bulb temperature.

The effect of thickness on blister formation is due to the lower gas concentration per unit area and to the increased ease with which gas can diffuse out from the thin sheet.

It is appreciated that blisters can arise by other mechanisms and these are at present under investigation.

6. EFFECT OF DRYING CONDITIONS ON TECHNOLOGICAL PROPERTIES

It was recognised at the outset that high temperatures would cause some softening of the rubber and that the quantitative effect would need to be determined. A casual observation had, however, also shown that a sheet immersed in hot water for measurement of water absorption became very much more tacky than a similar sheet kept at the same temperature in the dry state. It was thus thought that raising the humidity of drying could affect the degradation of the rubber quite apart from increasing the time during which the sheet is held at the drying temperature.

It was also considered that thickness of the sheet would have an effect on the rate of oxidation owing to the greater surface/volume ratio of such a sheet.

Effect of Elevated Temperature and Water Content on Raw Rubber Properties

It was considered that as the presence of water could affect the mechanism of the degradation of rubber it would be important to test the effect of conditions more extreme than those likely to be encountered in drying, so that any differences would be accentuated.

Experimental procedures. Six coagula were prepared at 1½ in. latex depth and pH 4.0 and machined to M.R. 0.032. Two sample sheets were cut from each and assigned randomly to the following three drying and storage treatments.

1	2	3
70 hrs at 140°F, 15% R.H.	24 hrs at 200°F, 5% R.H.	24 hrs at 200°F, 100% R.H.
	46 hrs at 140°F, 15% R.H.	46 hrs at 140°F, 15% R.H.

The sheets at 100% R.H. were suspended horizontally over water in a vessel having a water-sealed outlet which kept the system at approximately atmospheric pressure.

The Mooney Viscosity and Williams Plasticity of the dry sheets were measured in triplicate after one week at room temperature, after 40 days at room temperature, and after 40 days followed by 24 hours ageing at 212°F (100°C).

TABLE 13. MOONEY VISCOSITY

Treatment after drying and before testing	Drying treatment 1	2	3
7 days at c.85°F	58	82	56
40 days at c.85°F	61	78	50
40 days at c.85°F plus oven ageing	68	39	27

TABLE 14. WILLIAMS PLASTICITY (D_{10}), MM. EFFECT OF DRYING CONDITIONS

Treatment after drying and before testing	Drying treatment 1	2	3
9 days at c.85°F	3.9	3.3	2.5
40 days at c.85°F	3.7	3.3	2.5
40 days at c.85°F plus oven ageing	3.5	2.5	2.2

Discussion of results. Increasing the temperature of drying from 140°F to 200°F causes an increase in the Mooney Viscosity (Table 13) of the rubber before oven ageing, in a manner similar to that found by WOOD (1952). When the high temperature treatment is at 100% R.H., either the hardening effect is inhibited or degradation is accelerated.

Ageing the samples after drying gives the following picture: storage hardening occurs for rubber dried at 140°F, whereas for rubber treated at 200°F storage hardening is outweighed by degradation, the process of degradation being accelerated by the maintenance of the rubber in the wet state during the period at 200°F.

The data in Table 14 show that degradation is more severe at 200°F than at 140°F and that again the presence of water during treatment at 200°F accelerates the degradation.

Recent work by SEKHAR (1958) has contributed to the elucidation of some of the mechanisms causing these effects. The main conclusion relating to drying is that at high temperatures the presence of water accelerates degradation.

Effect of Thickness, Temperature and Humidity

Since the presence of water was found to affect the degradation of the rubber it appeared likely that high humidities of drying would affect the properties more than low humidities. Experiments were therefore made to find the effect of the most severe condition of drying likely to be encountered (200°F/70% R.H.).

Experimental procedures. 16 coagula were prepared at pH 4.0 and machined to M.R. 0.032. Latex depths of 0.87 in., 1.2 in. and 1.5 in. were used. 4 sheets of each thickness were dried at 200°F/70% R.H. and 4 medium sheets were dried at 140°F/15% R.H. as a control.

Sheets were removed when translucent and stored for one week under laboratory conditions. The raw rubber was tested in duplicate for Williams Plasticity and Recovery before and after oven ageing for 24 and 48 hours. Samples were also compounded according to the A.C.S.1. specification and 6 discs of each compounded sample were vulcanised for 40 minutes at 284°F (140°C). Three discs were tested before ageing and three after 48 hours ageing in an oxygen bomb at 158°F (70°C) and 300 p.s.i.g. (B.S. 903: Part A19: 1956).

Results. The results are presented in Tables 15, 16 and 17.

Discussion of results. Before ageing both Williams Plasticity and Recovery are lower for the treated sheets than for the control, but the thin sheets, which have been subjected to the drying conditions for a shorter time, are less affected than the thick ones

TABLE 15. WILLIAMS PLASTICITY (D_{10}) MM. RAW RUBBER. EFFECT OF THICKNESS AND DRYING CONDITIONS

Sheet	Drying	Unaged	Aged 24 hours	Aged 48 hours
Thick	200°F/70% R.H.	3.2	2.8	2.7
Medium	200°F/70% R.H.	3.5	2.8	2.8
Thin	200°F/70% R.H.	3.6	2.8	2.6
Medium (control)	140°F/15% R.H.	4.1	3.8	3.8

TABLE 16. WILLIAMS RECOVERY, MM. RAW RUBBER

Sheet	Drying	Unaged	Aged 24 hours	Aged 48 hours
Thick	200°F/70% R.H.	3.7	3.5	3.3
Medium	200°F/70% R.H.	4.2	3.6	3.4
Thin	200°F/70% R.H.	4.3	3.5	3.2
Medium (control)	140°F/15% R.H.	5.3	5.8	6.0

TABLE 17. VARIOUS PROPERTIES OF VULCANISED RUBBER BEFORE AND AFTER AGEING

Properties after ageing shown in brackets

Sheet	Drying	Tensile strength kg/cm²	Elongation at break %	Modulus at 660 elongation kg/cm²	Hardness °BSI	Resilience at 70°C (158°F) %
Thick	200°F/70% R.H.	141 (159)	887 (777)	48 (87)	35.2 (38.2)	81.4 (84.9)
Medium	200°F/70% R.H.	135 (159)	883 (780)	48 (89)	34.3 (37.8)	81.0 (85.3)
Thin	200°F/70% R.H.	132 (156)	875 (769)	48 (87)	34.2 (37.2)	80.2 (85.1)
Medium (control)	140°F/15% R.H.	140 (144)	885 (757)	48 (89)	35.6 (37.9)	81.2 (85.1)

(Tables 15 and 16). The ageing properties of the sheets dried at high temperature are inferior; the recovery (Table 16) shows that storage hardening has occurred with the control but degradation has overshadowed this in the treatment sheets. No effect of thickness is established in the aged samples.

Table 17 shows that the drying treatment has no great effect on the properties of the vulcanised rubber. There is however some indication that reducing the thickness may adversely affect the properties, but further confirmation of this is required.

Conclusions

Temperature, humidity, and sheet thickness affect the properties of the raw rubber after drying; in particular the presence of water in the sheet accelerates degradation. Drying at 200°F/70% R.H. causes considerably more degradation than drying at 140°F/15% R.H. but the properties of the vulcanised rubbers are little different. The degradation and the lowering of the resistance to ageing of the raw rubber is of a significant extent from a practical point of view and means of prevention would probably be needed if such drying conditions were used: either revision of the processing of the rubber to give higher retention of natural antioxidants or the deliberate addition of synthetic antioxidants, e.g., polyamines (SEKHAR 1958).

GENERAL SUMMARY AND CONCLUSIONS

The objective of the work reported in the six sections above has been to obtain data on which to base a plan for a more detailed investigation of drying.

Factors have been considered which include the source of the latex, coagulation, machining, drying conditions, and the effect of these drying conditions on the yield of the product and its visual and technological properties. Methods have been devised firstly for the minimisation of variability in factors affecting the drying characteristics of sheet dried under

a constant set of temperature, humidity, and air speed conditions and secondly for the measurement of properties likely to affect drying. The presence of three phases of drying, which often occur with other materials and were shown by the work of Piddlesden to be valid for the drying of rubber, has been confirmed and studied in greater detail; these studies have revealed further complexities in the mechanisms involved in rubber drying. In the first phase it has been shown that under certain drying regimes syneresis can contribute materially to the loss of water, and in the third phase the raising of the humidity of the air can retard the drying rate to an extent not previously realised. The finding that the change in equilibrium water content associated with this retardation is small (Sections 2 and 3), has emphasised the need for accurate water content measurements if any correlation between equilibrium water content and drying rate is to be established: a convenient method of determining the dry weight of samples has therefore been developed. Means for the accurate determination of the wet weight of a sample during drying, not reported here, have also been devised and will be described in a further paper. Data have been obtained which point to high temperatures being more economic (Section 2: Conclusions) and the limitations set by visual and technological defects have been examined; it has been shown that high humidity treatments reduce blister formation but accelerate degradation.

In the course of these investigations further insight has been obtained into the mechanisms involved in:

(a) coagulum behaviour during machining and subsequent syneresis
(b) the changes of the dimensions of a sheet during drying and
(c) the formation of blisters and degradation at high temperatures.

An overall picture of the way in which the results impinge on the economics of drier design is as follows. The marked effect of humidity makes it clear that there is an optimum humidity of operation, at which the benefits of reduced fuel consumption at high humidities are balanced against the disadvantage of reduced output.

The balance is, however, altered if use is made of high humidity to reduce the tendency to blister formation and thus permit the use of higher temperatures. If the method were practicable up to very high temperatures, say 200°F, the limit might well be set by degradation of the rubber which is itself influenced by the humidity treatment it receives and the concentration of antioxidants in the rubber; thus a new set of factors would then determine the balance.

Another method of reducing blister formation—and so allowing high temperatures to be used—is to reduce the thickness of the sheet. From a general economic point of view, thin sheet is preferable because reducing sheet thickness gives a greater reduction of drying time than of the weight of rubber per unit of drier volume; the output from a given size of drier would therefore be increased.

The introduction of the idea of an optimum thickness, however, takes one into a field where not only the drying operation itself must be considered, but also on one side the processes required to produce the wet sheet, and on the other the handling of the dry sheet to get into a form suitable for shipment. With the present methods of manufacture, although drying might be cheaper with thin sheet, the cost of preparation and packing would be greater. The present sheet thickness of about 0.1 in. is the outcome of many years of practical experience and development and is probably very near to the optimum.

If however some of the limitations were removed by the introduction of novel methods of coagulation, machining and packing, in which the balance of costs was different, then the optimum thickness might well be different from that in current use. If continuity of operation, and mechanisation, were associated with these changes the ratio of capital to operating charges would probably increase, and means of reducing drying times (i.e., reducing size of drying plant for a given output) would assume a new importance.

Considerable attention has been devoted by the various research institutes in the East to the possibilities of new coagulation and preparative techniques but it is clear that before the economics of a unified process can be assessed, further more detailed information is required on the effect of drying conditions on drying rates and quality. The present paper supplies data essential to the planning of the main programme designed to provide this information. The experimental part of this programme has already been carried out, but the analysis and assessment of the observations are not yet complete. It is hoped to publish the results of this further work in 1960.

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