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TRANSACTIONS

4—ON THE EXISTENCE OF A CRITICAL TEMPERATURE FOR MILLING

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Harrison's discovery[1] that the rate of shrinkage of wool fabrics in a milling machine is most rapid at a temperature of 46° to 49° C., when soap is used as the milling agent, was afterwards confirmed[2] by experiments carried out in this laboratory, except that a slightly lower value of 45° C. was obtained for the critical temperature. The validity of these observations has since been questioned by Mercer,[3] who used the method of Creely and LeCompte[4] to determine the shrinkage of 1-metre lengths of 3-ply yarn in buffer solutions at pH values ranging from 4·0 to 11·0 and different temperatures.

According to the theory[2] which was advanced in 1933, the shrinkage of wool fabrics in a milling machine is due to the peculiar mode of migration of the component fibres. Because of their surface scale structure, the fibres tend to migrate in the direction of the root end under the repeated application of pressure, but free migration is impossible in a fabric owing to the restraints imposed by twist in the yarns and their interlacings with one another. Migration is achieved by local extension and contraction of the fibres, and milling shrinkage is promoted by conditions which favour these processes. It is well known that the resistance of wool fibres to extension in water decreases with rise of temperature up to 100° C., but their power of recovery, relative to their resistance to extension, decreases sharply at temperatures above about 35° C., presumably owing to disulphide bond hydrolysis. The precise temperature at which the increasing ease of extension is overwhelmed by a decreasing power of contraction should, of course, depend on the pH of the milling agent and the mechanical conditions of milling, since the reactivity of the disulphide bond in wool is greatly affected by the pH of the environment and the strains induced by fibre extension. Except under conditions where the disulphide bond is stable, there should be an optimum temperature for milling, provided that fibre movement is so restricted that it must occur by alternate extension and contraction of the fibres. Such restraint is normally present in woven fabrics, but was certainly not present in the yarn employed in Mercer's experiments, for he obtained shrinkages as high as 20 per cent. at 20° C. and 45 per cent. at 60° C. by mere shaking for 6 minutes in a buffer solution at pH 9·2. Mercer's experiments could not, therefore, be expected to provide any evidence for or against the existence of a critical temperature for the milling of fabrics in soap solution.

As regards acid milling, the only evidence for an optimum temperature is the unpublished work of Harrison[1], which has been called into question by Schofield[5]. It was thought desirable, therefore, to re-examine the effect of temperature variations on the rate of shrinkage of wool fabrics in acid, as well as alkaline media. The work was carried out with the aid of a model milling machine so as to realise more precise control over experimental conditions than is possible in large-scale work.

EXPERIMENTAL

The apparatus used throughout the investigation was a stainless steel model of the Kilburn rotary milling machine, which was built to economise in fabric as well as for the sake of extra precision in experimental work. A cross-section through the machine, which is 5″ wide, 14½″ long and 13¼″ high, is shown in Fig. 1. The main rollers *A* and *B* each weigh 2·6 kg. and

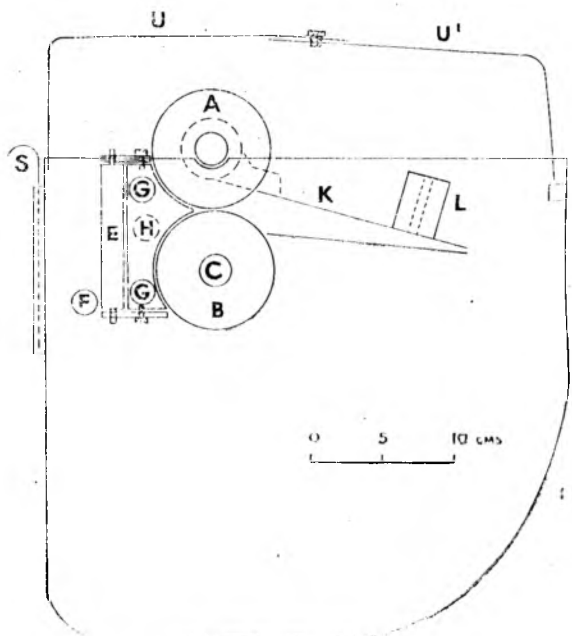

Fig. 1

are 3″ wide and 3″ diameter. In order to give adequate grip on the cloth during milling, the surface of each roller is scratch fluted, and the bottom roller *B*, which is mounted on the main shaft *C*, is driven by a ¼ H.P. electric motor *Z*, running at 1,425 r.p.m., through an 11 : 1 worm reduction unit *Q* and a chain drive, as shown in Fig. 2. The main shaft *C* runs at a calculated speed of 129·5 r.p.m., which was found to be maintained in practice, and the surface speed of the bottom roller was thus 102 feet per minute. The top roller runs at the same speed as the bottom roller, and is driven from the main shaft *C* by means of a chain drive situated on the side of the machine away from the motor. Pressure is applied to the rollers by means of the compression springs *W*, which are held in position by the screws *Y*. The latter can, of course, be adjusted to vary the pressure on the cloth, which is introduced into the machine through the sliding door *S*. After passing

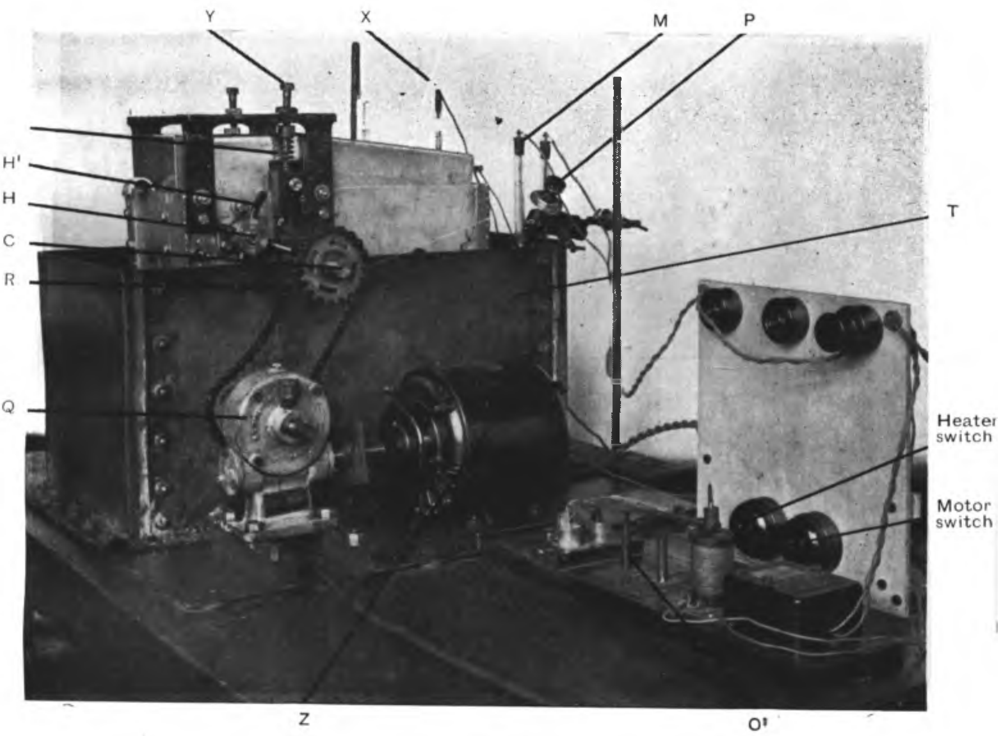

Fig. 2

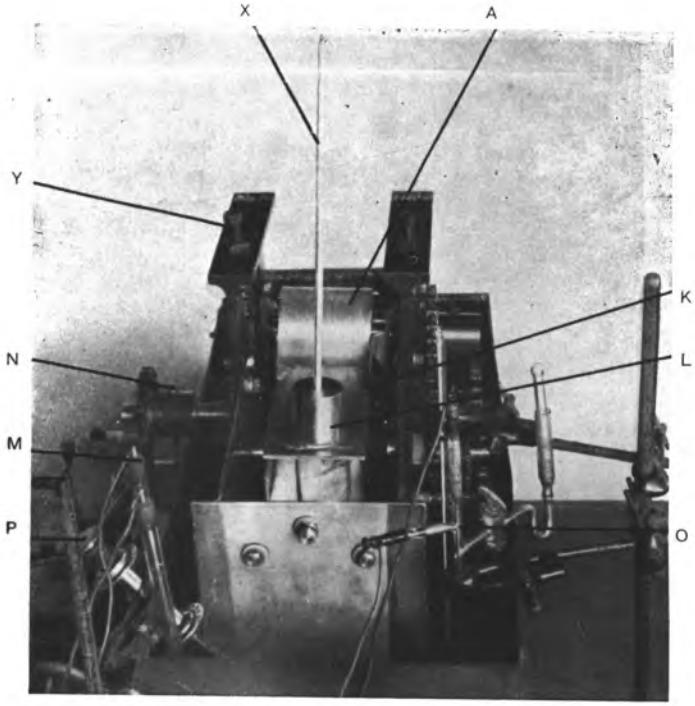

Fig. 3

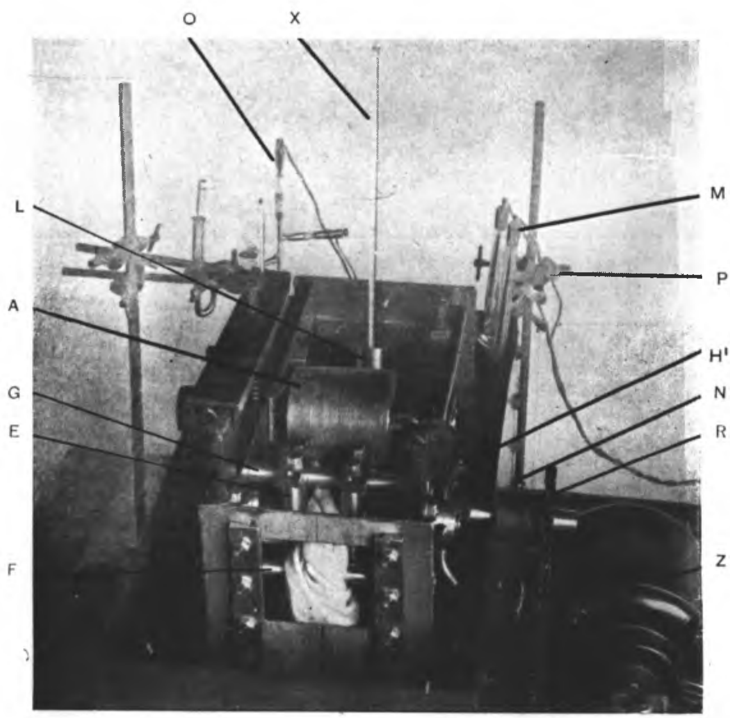

Fig. 4

through the rollers, the cloth enters the spout, which is provided with a hinged lid, permanently weighted, as shown in Fig. 3, with the stainless steel cylinder *L*. Through the latter is drilled an axial hole, $\frac{3}{8}''$ in diameter, which reaches almost to the inner face of the hinged lid, for the purpose of accommodating a thermometer *X* to record the temperature of the cloth during milling. Despite its simplicity, this device was found to give accurate values for the temperature of the cloth, far more accurate than could be obtained by stopping the machine and inserting a thermometer into the folds of the cloth in the spout. Width-ways compression of the cloth is effected by means of the vertical rollers *E*, which run freely in bearings carried by the horizontal shafts *G* and *G'*, as shown in Fig. 4. The distance apart of the vertical rollers can be adjusted by turning the hand wheels *H'* to operate the two worms *H* (11 turns per inch). Before entering the vertical rollers, the cloth passes over a freely-running guide roller *F*, but there is no draft board. In the event of serious resistance being encountered by the cloth during milling, tearing is prevented by the fact that the sleeve holding the toothed wheel *R*, through which the machine is driven, is attached to the shaft *C* by means of a screw *N*. The latter acts as a friction clutch and slips on the shaft whenever the cloth is not free to travel through the main rollers.

So that the temperature of the cloth can be adjusted to and controlled at different values, the milling machine is mounted by means of four brass brackets inside a water thermostat *T*, which is $10\frac{1}{2}''$ wide, 22″ long, and 11″ high. When full up to the lower edge of the sliding door *S*, the tank contains about $3\frac{1}{2}$ gallons of water, which is circulated by means of the stirrer *P*. Temperature control is maintained by the thermoregulator *O*, which operates the immersion heater *M*, having an output of 800 watts, through the relay *O'*. The upper part of the machine, outside the thermostat, is closed by two easily removable lids *U* and *U'*. The latter are clothed with felt to minimise heat losses and serve the additional purpose of reducing the amount of water lost by evaporation from the cloth during milling.

Preliminary experiments showed there was usually some difference between the temperature of the cloth and that of the thermostat, although the cloth temperature became steady after a few minutes' milling. The cloth used in this work was made from 64s merino noil and had the following characteristics:—

Warp: 28s Yorkshire skeins, with 10 turns/inch twist; 41 ends/inch.

Weft: 28s Yorkshire skeins, with 10 turns/inch twist; 36 picks/inch.

Weave: Plain. *Weight:* 7 ozs./square yard.

Lengths of cloth, measuring 81″ × $15\frac{1}{2}''$ and weighing about 190 grams air-dry, were wetted out in water, centrifuged, and then milled with 120 ccs. of a 5 per cent. soap solution. Temperature readings were taken every 10 minutes during milling and the results are shown in Table I.

Table I

Temperature of thermostat (° C.)	Temperature (° C.) of Cloth after milling for			
	10	20	30	40 minutes.
24·5	25·0	26·3	26·3	26·3
32·2	28·5	30·5	31·5	31·5
39·5	35·0	35·2	35·2	35·2
45·0	38·0	39·2	39·3	39·3
55·2	44·5	45·0	45·2	45·2

Owing to the frictional heat developed in the spout, the temperature of the cloth is higher than that of the thermostat at low temperatures; whereas the opposite is the case at high temperatures, owing to the heat lost through the upper part of the milling machine which is outside the water thermostat. Although the temperature of the cloth can in all cases be controlled and fixed by means of the water thermostat, the delay of 10 to 20 minutes in reaching temperature equilibrium would be serious in milling experiments which normally occupy no more than 40 minutes. To obviate this delay, the temperature of the exposed parts of the milling machine is raised by blowing steam into the apparatus, condensed water being removed with a dry duster immediately before the cloth to be milled is introduced into the machine. In this way, very precise control over the temperature of the cloth was realised in the following study of the effect of temperature on the rate of shrinkage of wool fabrics during milling.

The all-wool flannel used throughout the investigation was a commercial fabric with the following characteristics :—

Warp : 20s Yorkshire skeins with 14·5 turns/inch twist ; 34 ends/inch.

Weft : 20s Yorkshire skeins with 10·0 turns/inch twist ; 31 picks/inch.

Weave : Plain. *Weight :* 4·2 ozs./square yard.

Alcohol-soluble matter, determined by Soxhlet extraction : 1·5 per cent. on the dry weight.

Three sets of experiments were carried out, using soap, borax and sulphuric acid, respectively, as the milling agents, and as there were slight differences of procedure in each of these cases, it is convenient to give them separate consideration.

(1) Soap Milling

The general procedure adopted in carrying out a milling experiment was as follows. A strip of cloth, 200 cms. long and 38 cms. wide, was cut off the main length of flannel and weighed. About 40 cms. away from the ends of the strip, two rectangles, each 30 cms. long and 20 cms. wide, were marked out with coloured cotton. The fabric was then immersed in 2·5 litres of water overnight, centrifuged for two minutes, and, after measuring the areas of the rectangles, re-weighed. In the meantime, the temperature of the thermostat was adjusted to give the desired temperature of milling, in accordance with the data of Table I, and the exposed parts of the machine were warmed up with steam as decribed above. As soon as condensed water had been removed, the cloth was introduced and the ends sewn together with strong sewing cotton. The machine was then started and 100 ccs. of a 5 per cent. soap solution, warmed up to the temperature of the experiment, were added gradually to the cloth as it passed over the guide roller. The soap used in making up the stock solution for milling was a potash soap which contained 30·2 per cent. of water and had been prepared by direct neutralisation of fatty acids with caustic potash. The fatty acids had an iodine value of 38·7, a setting point of 46·2° C., an apparent molecular weight of 275 and a palmitic/stearic ratio of 52 : 48. Milling was allowed to proceed for 30 minutes in the enclosed machine with the front rollers 4·2 cms. apart, and temperature readings were taken after 5, 10, 20 and 30 minutes. No exact definition of the pressure applied to the cloth by the spring-loaded horizontal rollers can be given, because it varied with the state of folding of each part of the cloth as it passed between the rollers. With cloth between the rollers, the springs were compressed, and since each had a sensitivity of 15·5 kg./cm., it is evident that a large, unknown, but standardized pressure (as between one experiment and another) was applied to the cloth during milling. Although the machine was totally enclosed, it was not air-tight, and there was some loss of water from the cloth by evaporation, especially at high temperatures. In the light of preliminary experiments, it was found possible to correct the loss by making two equal additions of water through the slit in the machine lid, which accommodates the thermometer, after

10 and 20 minutes' milling. At temperatures above 50° C., some difficulty was encountered in maintaining the desired temperature of the cloth, and a small jet of steam was therefore blown into the machine at intervals by raising the sliding door, care being taken that the steam did not impinge directly on the cloth. The condensed water arising from this process compensated partially for the water lost by evaporation, and the amounts of added water were therefore smaller than would otherwise have been necessary. At the end of the milling period, the cloth was removed from the machine and weighed in order to determine the extent of the gain or loss of water during milling. From the data given in Table II, it is evident that, except at very high temperatures, the devices adopted to compensate for loss of water by evaporation were very successful.

Table II

Temperature (° C.)	Weight of Cloth (g.) Air-dry	After Centrifuging	After Soaping	After Milling	Change in Weight during Milling (g.)	Additions during Milling: Water (ccs)	Steam	Percentage Shrinkage in Length	Width	Area
15·6	131	168	268	263	− 5	–	–	20·0	12·3	29·9
16·0	130	168	268	259	− 9	–	–	20·5	10·8	29·0
24·4	130	170	270	265	− 5	6	–	25·2	13·9	35·6
24·5	132	171	271	267	− 4	6	–	24·2	13·8	34·6
31·5	128	166	266	251	−15	10	–	30·5	22·6	46·2
34·9	132	169	269	265	− 4	16	–	29·6	22·9	45·7
38·5	133	170	270	260	−10	18	–	32·4	23·5	47·8
47·0	130	164	264	262	− 2	28	–	29·9	21·8	45·2
51·4	130	164	264	258	− 6	10	+	32·0	23·5	46·9
52·1	131	170	270	256	−14	–	+	30·4	21·4	45·5
56·3	132	171	271	256	− 15	–	+	32·5	21·0	46·8
58·3	127	166	266	295	+19	–	+	34·2	21·0	48·2

As soon as the cloth had been weighed, the dimensions of the marked rectangles were re-measured, and the shrinkages calculated. The results are given in Table II, illustrated by Fig. 5, where the shrinkage-temperature

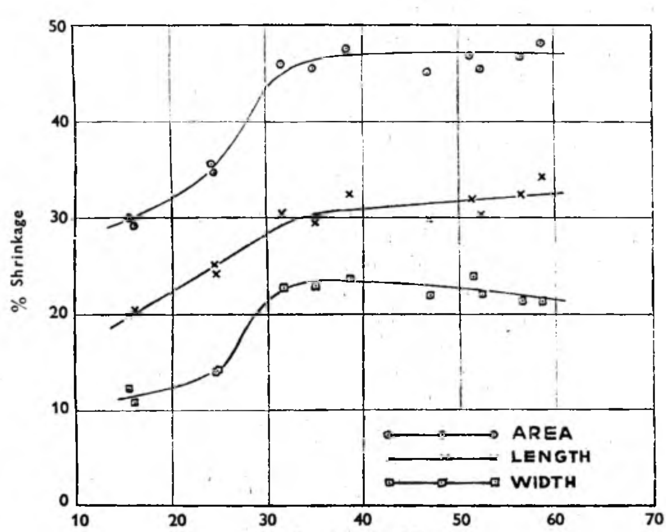

Fig. 5

curves show that the critical temperature for milling with the potash soap in the model milling machine is 35° C. At temperatures higher than this critical value, the width shrinkage decreases with rise of temperature and the area shrinkage remains approximately constant on account of the slight increase in length shrinkage. Length shrinkage is abnormally high in relation to width shrinkage because of the wide separation of the vertical rollers; when their distance apart is reduced to 2·5 cms., width shrinkage preponderates, but the wider separation of 4·2 cms. was used so that all experiments could be carried out under the same mechanical conditions, without the extent of shrinkage in acid milling being so great as to invalidate the results at high temperatures. Since the length shrinkage increases slightly with rise of temperature above 35° C., the number of passages of the cloth through the machine and, therefore, the amount of work carried out on the cloth, must also increase with rise of temperature. In consequence, although the area shrinkage is almost constant at temperatures between 35° and 58° C., the area shrinkage *for the same energy input* decreases with rise of temperature above 35° C. Even if this factor is neglected, however, the constancy of area shrinkage is sufficient to confirm earlier argument regarding the importance of the elastic properties of the wool fibre in relation to milling shrinkage. The ease of extension (deformation) of wool fibres in water increases steadily with rise of temperature up to at least 100° C., and so should the rate of shrinkage if ease of deformation of wool fibres is essential for milling. Conversely, the power of recovery of deformed fibres, relative to the ease of deformation, first increases and then, above about 35° C., decreases with rise of temperature in water.[2] If the shrinkage of wool fabrics is due to fibre migration by alternate extension and contraction, in the manner already described, there should be a critical temperature for milling at some temperature at and above which an increased ease of deformation if off-set by a reduced power of recovery from deformation. In the preceding series of experiments with potash soap as the milling agent, it is evident that a fairly exact balance between the two opposing factors is struck at temperatures between 35° and 58° C., in accordance with the general theory of milling developed in a previous paper.[2]

In the earlier experiments which were carried out to confirm Harrison's discovery[1] of a critical temperature for soap milling, shrinkage was found to take place most rapidly at 45° C., compared with the preceding value of 35° C. The discrepancy is no doubt due to differences between the large-scale and model milling machines. In particular, the mechanical action of the model milling machine is far more severe than that of the large scale machine, and in the large-scale experiments there was no precise compensation for water lost by evaporation during milling.

(2) Alkali Milling

The physical properties of the soap solution used as milling agent in the preceding experiments must, of course, alter with change of temperature. In order to show that the critical temperature for milling is not determined by a change in the state of the soap solution, further experiments were carried out with a 2 per cent. solution of borax ($Na_2B_4O_7 \cdot 10H_2O$) as the milling agent. The general procedure was the same as in the preceding experiments, with the following exceptions. Each strip of cloth was soaked overnight in 2·5 litres of the borax solution instead of in water. After the cloth had been centrifuged, measured, and weighed, it was introduced into the machine and sufficient of the solution in which the cloth had been soaked (final pH 9·17) was added, after being warmed to the temperature of the experiment, to bring the total weight of the cloth up to twice its air-dry weight. The time of milling was 40 minutes and temperature readings were taken every ten minutes. Loss of water by evaporation was made good by adding three equal amounts after 10, 20 and 30 minutes' milling. The results obtained in this series of experiments are given in Table III, illustrated by Fig. 6.

Table III

Temperature (° C.)	Weight of Cloth (g.) Air-dry	After Centrifuging and adding Borax Solution	After Milling	Change in Weight during Milling	Additions during Milling Water	Steam	Percentage Shrinkage in Length	Width	Area
				(g.)	(ccs)				
15·0	135	270	257	−13	–	–	17·9	9·4	25·4
23·0	131	262	253	− 9	5	–	22·1	12·6	33·2
26·8	135	270	263	− 7	8	–	24·2	16·7	37·0
34·7	135	270	265	− 5	12	–	27·5	21·6	43·4
38·3	138	276	266	−10	15	–	28·4	23·8	45·2
41·7	129	258	246	−12	18	–	29·2	20·7	43·8
43·8	132	264	260	− 4	27	–	28·5	18·8	41·8
45·3	131	262	255	− 7	27	–	29·4	19·4	43·1
47·7	132	264	256	− 8	27	–	28·8	21·0	44·5
55·3	129	258	250	− 8	3	+	31·7	20·7	45·6
57·6	137	274	273	− 1	24	+	30·7	19·1	43·7
60·8	138	276	313	+37	15	+	31·2	20·3	45·2

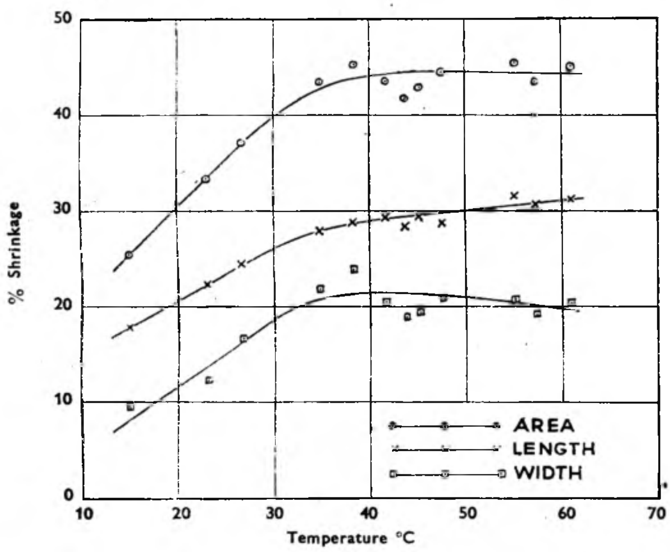

Fig. 6

In general form, the shrinkage curves are remarkably similar to those of Fig. 5 for soap milling, and the existence of a critical temperature for soap milling is clearly not determined by the properties of the soap used.

One peculiarity of the results obtained in soap milling and alkali milling is that the critical temperature (35° and 37° C., respectively) should be so close to the value of 35° C., the temperature above which wool fibres show increasing reluctance to recover from extension (deformation) in water. The fibres were not milled in water, however, but in alkaline media, and the most probable explanation of the difficulty is that the balance between an increasing ease of deformation and a decreasing power of recovery is struck at lower temperatures as the pH of the medium increases. This would, in fact, be expected from first principles because the power of recovery of wool fibres from extension is governed to a large extent by the number of intact cross-linkages between the peptide chains, and the disulphide bonds are readily hydrolysed by alkali, especially when they are strained and the temperature is high. The hysteresis between extension and contraction of wool fibres in buffer solutions at various pH values was therefore determined at temperatures ranging from 25° to 65° C.

For this purpose, the root ends of Cotswold wool fibres, which had been purified by extraction with alcohol and ether in a Soxhlet apparatus, were attached by means of sealing wax or dental cement to light glass hooks in the usual manner. Each fibre, which was about 5 cms. long, was then immersed in the appropriate buffer solution for two hours at 22·2° C. before being stretched in another lot of the same buffer solution at the desired temperature. Extension was restricted to 30 per cent. and the fibres were taken through a cycle of extension and contraction by means of the Cambridge recording fibre extensometer, as modified to meet the requirements of this laboratory. The area of each hysteresis loop was calculated and expressed as a percentage of the area between the load-extension curve and the axis of extension. A summary of the results is given in Table IV, which includes the *p*H values of the buffer solutions at 22·2° C.

At *p*H 8 and above, the percentage hysteresis increases steadily with rise of temperature above 25° C., showing that in milling with soap or borax solution, there will be no difficulty in the reduced power of recovery from deformation compensating for the increased ease of deformation at 35° C. and above, as indicated by the preceding milling experiments. At

Table IV

Buffer	*p*H	Percentage Hysteresis at				
		25° C.	35° C.	45° C.	55° C.	65° C.
HCl + KCl	1·03	35·9	29·7	28·3	22·9	22·7
HCl + KCl	2·06	43·2	38·2	33·7	31·5	29·2
Na_2HPO_4 + Citric Acid ...	3·10	50·8	44·6	43·5	38·6	37·7
,, ,, ,, ...	4·12	55·2	48·0	47·5	43·7	44·6
,, ,, ,, ...	5·08	57·7	50·4	51·6	47·8	50·7
,, ,, ,, ...	5·87	57·5	52·3	53·7	54·1	64·1
,, ,, ,, ...	7·00	58·0	55·4	65·4	72·6	77·5
,, ,, ,, ...	8·10	58·7	63·8	75·4	86·1	88·3
Na_2HPO_4 + KH_2PO_4 ...	7·96	58·0	59·6	74·0	84·6	89·0
NaOH + H_3BO_3 + NaCl ...	9·24	62·3	74·5	83·8	90·3	95·1
NaOH + H_3BO_3	9·98	69·0	80·8	91·3	93·7	98·4
NaOH + Na_2HPO_4	11·29	79·4	89·1	95·5	97·5	98·0
,, ,, ...	12·06	84·4	94·5	97·2	97·6	95·8
Glycine + NaOH + NaCl ...	12·83	73·5	85·8	84·9	83·8	88·4

*p*H 3 and below, however, the percentage hysteresis *decreases* steadily with rise of temperature above 25° C. and there seems to be no possibility of a critical temperature for milling in acid media. The only evidence that there is a critical temperature for acid milling is the unpublished work of Harrison, though some indication that it might exist is contained in Schofield's data for the felting of loose wool with acid[6]. Aral[7] attempted to confirm Harrison's results in 1939 by milling cloth with sulphuric acid in the Kilburn rotary milling machine at different temperatures. The cloth used was made from 64s merino noil, the 10 skeins yarn being woven in 2/2 twill weave with a setting of 22 ends and picks per inch. After the cloth had been scoured, it was dried in absence of tension and cut up into strips 32 yards long and 30 inches wide. Two areas measuring 36″ × 20″ were marked out in coloured cotton on each strip, which was weighed before immersion in cold water for 24 hours. The cloth was then centrifuged, weighed, and immersed in 10 times its weight of water to which concentrated sulphuric acid had been added. The amount of sulphuric acid used was 4 per cent. on the air-dry weight of the wool and the *p*H value of the solution was measured before and after the cloth had been immersed for 24 hours at room temperature. After the cloth had been centrifuged and re-weighed, the areas of the rectangles were re-measured. The cloth was then fed into the milling machine and the ends sewn up, the temperature of the machine having previously been raised to the desired value by blowing in steam from a perforated pipe fitted underneath the floor board. In all experiments, the weight on the lid of the spout and the distance apart of the vertical rollers were kept constant, and the same volumes of the sulphuric acid solution with which the cloth had attained equilibrium during soaking were added as milling agent after the same times of milling. At regular intervals during each experiment, the temperature of the cloth was recorded by stopping the machine momentarily and inserting a thermometer into the folds of the cloth where it emerged from the spout. In addition, the machine was stopped at intervals of about 15 minutes so that the areas of the marked rectangles could be redetermined by making three measurements of length and four of width on each of the two areas. Shrinkages were expressed as percentages of the areas of the rectangles after the cloth had been immersed in the sulphuric acid solution, and the results are summarised in Table V, illustrated by Fig. 7.

Table V

Temperature (° C.)	Initial *p*H	Final *p*H	Time of Milling (minutes)	Percentage Shrinkage in Area
35·2	1·26	2·22	17	22·8
			36	44·3
			55	55·7
			72	60·6
			89	63·4
45·3	1·30	2·22	17	25·0
			34	45·5
			51	56·4
			68	62·0
			85	64·9
			102	67·0
54·3	1·28	2·29	17	26·6
			34	48·7
			45	56·6
			54	60·8
			71	65·1
			88	68·0
64·2	1·25	2.20	17	22·8
			34	42·7
			52	59·6
			69	65·6
			86	69·2
			103	71·6

It is evident that the critical temperature for acid milling is certainly not below 54·3° C., and although the curve for milling at 64·2° C. is below that for 54·3° C. up to 54 minutes' milling, the highest degree of shrinkage is ultimately obtained at the higher temperature. The results are remarkably precise for large-scale work of this kind, but no attempt was made to correct for loss of water by evaporation and it was feared that the results for high temperatures might be invalidated from this cause. Further experiments were therefore carried out in the model milling machine.

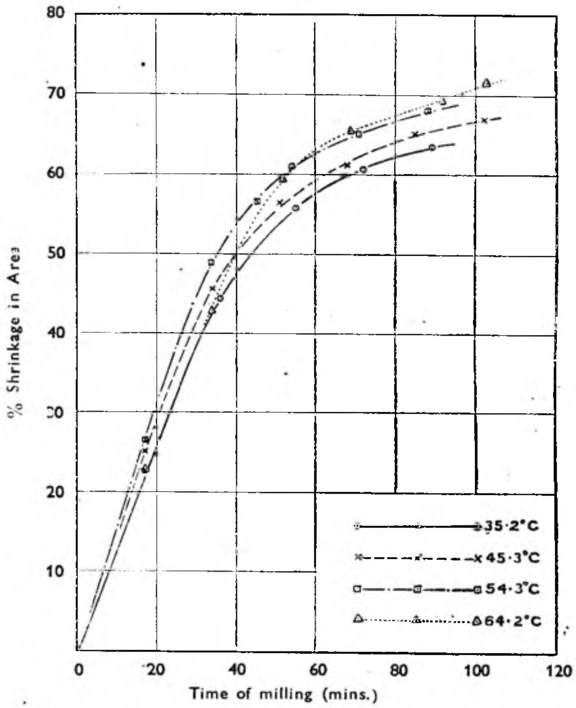

Fig. 7

(3) Acid Milling

The general conditions of the experiments were similar to those adopted in the case of soap milling (p. T44). Each length of cloth was soaked overnight in 2·5 litres of a 0·4 per cent. (v/v) solution* of sulphuric acid in water, centrifuged, measured, weighed and then introduced into the milling machine. After the ends of the cloth had been sewn up, the machine was started and sufficient of the solution in which the cloth had been soaked was added to bring the total weight of the cloth up to twice its air-dry weight. Milling was stopped after 30 minutes, and the results of the experiments are given in Table VI.

Table VI

Temperature (° C.)	Final *p*H	Weight of Cloth (g.): Air-dry	Weight of Cloth (g.): After Centrifuging and adding Acid Solution	Weight of Cloth (g.): After Milling	Change in Weight during Milling	Additions during Milling: Water	Additions during Milling: Steam	Percentage Shrinkage in: Length	Percentage Shrinkage in: Width	Percentage Shrinkage in: Area
					(g.)	(ccs.)				
17·2	1·34	142	284	270	− 14	—	—	27·4	16·0	39·0
19·1	1·36	132	264	249	− 15	—	—	29·6	18·4	42·4
24·6	1·32	133	266	260	− 6	6	—	34·0	23·1	49·3
29·0	1·31	129	258	251	− 7	8	—	40·8	29·3	58·1
34·7	1·30	132	264	260	− 4	14	—	39·2	33·2	59·4
39·5	1·28	134	268	267	− 1	20	—	40·2	36·8	62·0
42·0	1·33	130	260	251	− 9	20	—	42·2	34·8	62·2
46·8	1·30	133	266	266	0	26	—	42·7	41·4	66·4
58·0	1·33	130	260	258	+ 2	—	+	45·8	47·2	71·3

* The large volume of solution, besides ensuring complete wetting-out, gave a final *p*H close to unity, where all salt linkages between the peptide chains of wool are broken.

From the shrinkage-temperature curves of Fig. 8, it is evident that, whether area-shrinkage, length-shrinkage or width-shrinkage is considered, the critical temperature for acid milling in the model milling machine is not below 58° C. This result is in conflict with Harrison's, but agrees with the theory of milling[2], because the hysteresis between extension and contraction of wool fibres decreases steadily with rise of temperature in acid media.

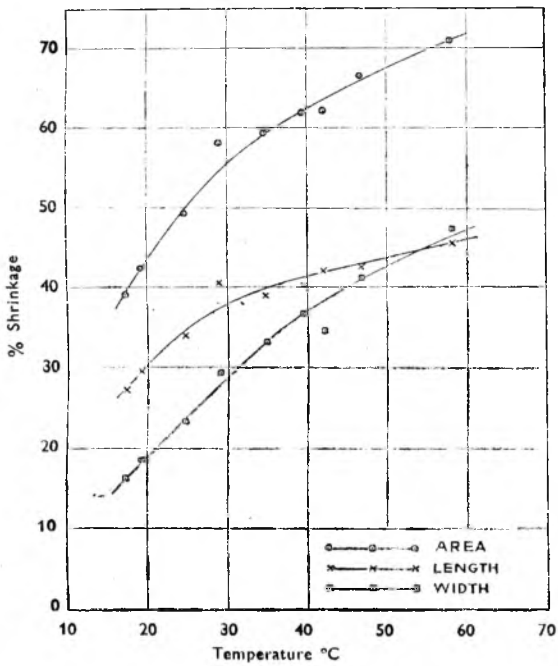

Fig. 8

SUMMARY

Using solutions of soap, borax and sulphuric acid as milling agents, the rate of shrinkage of an all-wool woven fabric in a model milling machine has been determined at different temperatures up to 60° C. The optimum temperature for milling with solutions of soap and borax was found to be 35° to 37° C., but no evidence could be obtained for the existence of a critical temperature for acid milling. These results lend further support to an earlier theory of milling[2], for whereas the hysteresis between extension and contraction of wool fibres in alkaline media increases, the hysteresis in acid media decreases steadily with rise of temperature above 25° C.

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