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## 9—JUTE CELLULOSE AND THE RELATION OF JUTE INCRUSTANTS TO FIBRE AND YARN STRENGTH

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### SUMMARY

When jute is treated with the chemical reagents employed in textile purification and bleaching processes the lignin, hemicellulose and other incrusting substances are attacked and to some extent removed. The greater the extent of this removal the more is the strength of the jute diminished, particularly when the material is tested in the wet state. Removal of substantially all the lignin with retention of hemicellulose, or of hemicellulose with retention of lignin, results in low wet strength although dry strength is but little affected. Treatments that result in the partial removal of both these incrustants usually reduce both dry and wet strength, although in hot alkaline steeps such as are given in peroxide bleaching, where fibre swelling can occur and there is no pronounced removal of incrustants, dry strength may be increased above the original value owing to the closer setting of fibre on fibre that occurs as a consequence of swelling and subsequent drying. Boiling under moderate pressure with sodium hydroxide solution removes substantially all the hemicellulose but little lignin, whilst treatment with acid chlorite solutions and then neutral sulphite liquor removes the lignin but not the hemicellulose.

The incrustants in jute resemble in their behaviour the starch size on a low-twisted sized cotton yarn and while they themselves have little tensile strength they contribute in marked manner to the strength of the jute by cementing together the ultimate cellulose fibre bundles on which the strength fundamentally depends. In oxidation treatments commonly employed in technical bleaching they appear to have a protective action on the cellulose.

Alkaline hypochlorite has mainly an oxidising, and acid hypochlorite a chlorinating action on jute, and use of the latter in conjunction with a pressure boil with sodium hydroxide and a final oxidation bleach with alkaline hypochlorite promotes attack and removal of lignin and fairly readily allows pure white cellulose to be isolated. If the preliminary chlorinating treatment is not given, more drastic boiling at high pressure is necessary and more than one boil may be required. The characteristics of the jute cellulose are such as to suggest that the product can be used satisfactorily for the manufacture of viscose, cellulose compounds and white papers.

The "browning" of bleached jute that occurs when this material is exposed to sunlight can be prevented only when the conditions of purification are such as to ensure that no lignin remains, and it appears that lignin itself is the product responsible for the browning effect.

### INTRODUCTION

Raw jute consists of fibres varying in length from about 4 to 8 feet obtained from the outer sheath of the plant stem by a water retting process. The individual fibre is composed of elementary cellulose units of very short

length (roughly of the order 2 to 5 mm.) surrounded and cemented together by a complex structure of hemicellulose, lignin and other plant substances, and owing to the shortness of these cellulose units it is evident that the strength of the fibre (which as in other natural vegetable fibres depends mainly on the cellulose), must be determined largely by the extent to which the incrustants are capable of cementing them together. It is to be expected therefore, that in comparison with cotton, linen and other bast fibres possessing longer cellulose ultimates, jute will be adversely affected to a more marked extent by any chemical or physical action tending to remove the cementing substances or even to loosen their adhesion to the cellulose.

For textile purposes it is often necessary to improve the appearance, softness and other properties of vegetable fibres by bleaching their coloured impurities and removing gums, resins, fats, waxes and non-cellulose materials that may render less efficient the normal operations of chemical or even mechanical processing, but it is usually undesirable to treat the material in such a manner that its loss of weight becomes considerable, and, above all, strength must be conserved to the maximum extent consistent with the obtaining of the other desired effects. Methods for the purification of cotton and linen have already been fully investigated but hitherto no systematic examination has been made of the conditions under which the common textile purifying treatments may be safely applied to jute, or under which the best compromise between loss of strength or weight and lightening of the shade may be achieved.

In the limit, the whole of the incrustants, amounting to about 40 per cent. of the weight of the jute may be removed, and the residual material then consists only of the extremely short fibres of cellulose or modified cellulose. Such highly purified material is necessary if it is intended as a source of cellulose for the manufacture of rayon, cellulose esters or ethers or white papers, but where the original fibre structure must be retained, and the jute is to be employed in spinning and weaving, only comparatively mild treatments may be given.

In the present work an examination has been made of the behaviour of jute yarn and fibre towards reagents that are commonly employed in the purification and bleaching of vegetable fibres and the effects on yarn strength of the progressive removal of the incrusting substances have been investigated. The conditions have also been explored under which yellowing or browning of bleached jute occurs when the material is exposed to light, and the extent to which purification is necessary if such discoloration is to be prevented has been defined. Finally, observations are recorded dealing with the isolation of cellulose from jute and with the suitability of this cellulose for use in the manufacture of rayon or commercial cellulose compounds.

**The Action of Alkalies, Oxidising and Reducing Agents on Jute**

The common method of purifying and bleaching most vegetable fibres comprises boiling them with dilute alkali metal hydroxide or carbonate solutions, followed by steeping in dilute mineral acid and treatment with hypochlorite, peroxide or other oxidising agents or with reducing agents, and one or more of these operations may be repeated as required.

The object of the "alkali boil" is efficiently to wet out the material, to saponify or emulsify resins, fats and waxes, and to remove them together with pectin and cell-wall polysaccharides that are soluble in hot dilute solutions of alkalis but not in water. Removal of these substances does not impair the strength of cotton or linen yarns because they are composed of relatively long ultimate cellulose units or cell bundles that overlap to an extent that allows a comparatively large proportion of their individual strength to be realised when they are twisted together. With jute, however, owing to the shortness of the cellulose ultimates on which strength

so much depends, conditions of alkali treatments that are satisfactory for cotton and linen may be expected to have harmful effects because the removal of the cementing substances is capable of loosening the fine fibre structure in such a way that cohesion between the very short cellulose ultimates is weakened.

The acid steep serves to eliminate inorganic and other substances that are soluble, whilst the oxidising treatments are usually essential for the production of bleached effects.

Not only the alkali, but also the oxidising treatments are capable of modifying the incrusting substances. For example, Norman[1] states that "Lignin is curiously unstable towards oxidising agents such as hypochlorites, hydrogen peroxide, permanganate and ozone, and if the reaction is allowed to proceed to completion only the simplest degradation products such as the lower fatty acids and dibasic acids, such as oxalic and succinic can be found." Furthermore such agents are capable of attacking hemicelluloses, and even if lignin and hemicelluloses are not actually removed it is to be expected that such chemical modification will adversely affect their cementing action towards the cellulose ultimates and so cause reduction of strength.

**The Action of Hot Alkaline Solutions**

The conditions of hot alkaline treatment normally employed for vegetable fibres vary in severity from boiling under excess pressure with sodium hydroxide solutions to milder steeping at atmospheric pressure in sodium carbonate solutions either alone or containing a small proportion of an oxidising agent such as peroxide.

The effects of such treatments on the loss of weight and strength and on the lignin content of jute yarn are shown in Table I. It is seen that boiling under excess pressure with a 1 per cent. solution of sodium hydroxide for several hours results in a high loss of weight (23 per cent.) actually twice as great as that occasioned by treatment under atmospheric pressure at a temperature near to the boil with either sodium hydroxide or carbonate solutions. Steeping in a sodium carbonate solution at much lower temperature (65°C.) for two hours gives a much smaller loss as would be expected, but if the carbonate liquid contains also sodium peroxide and silicate the loss realised is not greatly different from that obtained with carbonate alone at the boil. The smallest loss of weight is obtained after a steep in hot water alone.

The percentage losses of lignin under these conditions, based on the original jute after taking into account the corresponding losses of weight, are given in column 3. They are shown to diminish with decreasing severity of treatment, but nevertheless are in all cases comparatively small so that although hot alkaline liquids sometimes cause considerable losses of weight they have relatively little effect in removing lignin. When peroxide is present in sodium carbonate solution (No. 6) some oxidation of non-cellulose constituents may occur but this effect is not pronounced because, for example, the loss of lignin is not markedly higher than when peroxide is absent.

The last columns of this table show the effect of the various treatments on the dry- and wet-strength of the yarns. In no case is any very serious loss of dry strength recorded, but on the other hand the strength of the wet yarns is sometimes considerably reduced.

Comparison of the various properties measured reveals some interesting results. It is seen, for example, that the very mild treatment No. 4 causes very little loss of weight or of lignin and has little effect on either the dry or wet strength of the yarn. The most severe treatment however, with sodium hydroxide under pressure has removed 23 per cent. of weight and although the dry strength of the yarn is not seriously affected,

its wet strength is reduced to a very low value. Under these severe conditions the long chain molecules of the cellulose material must remain to a great extent unaffected in the same way as cotton remains relatively unaffected as a result of technical boiling in the kier, but pressure boiling of this kind favours the removal of the shorter chain polysaccharides, and as the lignin content still remains reasonably high the reduction of wet strength must be due largely to the removal of hemicellulose.

Under the milder conditions of open boiling illustrated by treatments 2 and 3, it is seen that dry strength is also satisfactory and that although the wet strength is reduced, the values are higher than those for the pressure boil. This behaviour is consistent with less complete removal, or modification to a lesser degree, of the cementing material than occurs in pressure boiling. When peroxide is used in what would otherwise be a very mild alkali treatment (No. 6), dry strength is also retained but that there is some action on the cementing material is again indicated by the loss of strength on wetting the yarn.

The effect of a hot water steep (No. 5) is very mild indeed and here the breaking loads should be noted because they are higher than those for the untreated yarn. These higher values may be ascribed in part, though probably not entirely, to mechanical removal of "batching" oil that otherwise acts as a lubricant and promotes fibre slippage. A similar increase is obtained in fact if the oil, fat and wax present is removed from the yarn by extraction with an organic solvent. For example, a hank of the yarn was re-wound, tied twice at opposite points to retain twist, and divided into two portions, one of which was extracted three times with trichlorethylene while the other remained untreated. The loss of weight on extraction was 5 per cent. so that actually more soluble matter was removed than by the hot water steep No. 5. Breaking load measurements were made after conditioning both portions and the results were:

| | | | |
|---|---|---|---|
| Dry untreated | 8·5 lb. | Dry extracted | 9·6 lb. |
| Wet ,, | 8·1 ,, | Wet ,, | 8·5 ,, |

From the low value for loss of weight (2 per cent.) it is obvious that the hot water treatment has no significant action on hemicellulose or lignin and the wet strength of the treated yarn is high, although not equal to the dry strength.

**Table I**

**Effect of Hot Alkali Treatment on the Loss of Weight, Lignin Content and Strength of Jute Yarn**

| Treatment | % Lignin | | % Loss of weight | B.L. lbs. | |
|---|---|---|---|---|---|
| | In residual material | Loss on original jute | | Dry | Wet |
| Untreated ... ... ... ... ... | 13·8 | Nil | Nil | 8·1 | 7·1 |
| Scour. | | | | | |
| 1. Pressure boil, 1% NaOH at 20 lb. excess pressure for 6 hours ... ... ... | 13·6 | 3·3 | 23·0 | 7·4 | 3·3 |
| 2. Open boil, 1% NaOH at 90° C. for 6 hours... | — | — | 11·0 | 7·2 | 4·5 |
| 3. Open boil, 10% $Na_2CO_3$ on weight for 2 hours ... ... ... ... ... | 12·9 | 2·2 | 11·0 | 7·4 | 4·4 |
| 4. Hot steep 7·5% $Na_2CO_3$ on weight at 65° C. for 2 hours ... ... ... ... | 13·3 | 1·0 | 4·3 | 7·8 | 6·4 |
| 5. Water steep, 70° C. for 2 hours ... ... | 13·9 | Nil | 2·0 | 9·0 | 7·9 |
| 6. Oxidising steep, 10 g/l. $Na_2CO_3$+1 g/l $Na_2O_2$ + 8·5 g/l sodium silicate at 65° C. for 2 hours ... ... ... ... | 13·1 | 1·9 | 9·8 | 8·0 | 4·7 |

(Wt. Ratio of liquor to jute in all cases approximately 8 : 1.)

It follows from the results recorded that important loss of strength occurs only when there is significant removal of incrustants, and even then this loss is realised to a marked extent only when the yarns are tested in the wet state. Wetting an otherwise untreated yarn causes a loss of strength of the order 10 per cent. due probably to the softening effect of water on the individual fibres or their incrustants and to the lubricating action that promotes fibre slippage. Reference to the effect of the non-cellulose impurities on strength is made again in a later section.

**The Action of Oxidising Agents**

*Hypochlorite Solutions.*

It is obvious from the chemical structure of the jute fibre and from the variation that occurs in the properties of hypochlorite solutions with change in acidity or alkalinity in the neighbourhood of the neutral point, that very different effects are to be expected according to the *p*H of the solutions with which the jute is treated.

The importance of *p*H in the rate of oxidation of cellulose by hypochlorite solutions was abundantly demonstrated several years ago by Clibbens and Ridge[2] who showed the extreme activity of neutral or nearly neutral solutions. Since, however, the jute fibre is composed of lignin and hemicellulose as well as cellulose, and since lignin is very sensitive to both oxidation and chlorination, whilst the polysaccharides other than cellulose are also attacked by hypochlorite it is clear that if undue modification of the structure is to be prevented conditions must be chosen so that both the cellulose and the non-cellulose constituents are affected as little as possible.

It is also fairly common practice in purifying and bleaching other vegetable materials to steep them in a hot solution of an alkali or in an alkaline peroxide liquid after a hypochlorite treatment. Here the effect of the alkaline solutions is to dissolve and remove lignin that has been modified by chlorination and also to remove hemicellulose, and the greater the degree of chlorination and of oxidising attack of these substances the greater is the effect of alkali in promoting their dissolution. The extent to which such liquids have a disintegrating action on jute therefore also depends on the precise conditions of the previous treatment with hypochlorite and for this reason also it is important to ensure that the conditions of use of the hypochlorite solutions are such as to minimise any undesirable effects of subsequent wet processes.

**The Effect of *p*H on the Course of the Reactions between Hypochlorite Solutions and Jute**

It is already well known that under certain conditions of *p*H hypochlorite solutions can exert a chlorinating, and under others, an oxidising action, but no quantitative expression of the extent to which these effects are obtained has yet been given. From what is known of the effects of such solutions on cotton and linen cellulose, oxidation of the cellulose and polysaccharide constituents of jute can no doubt occur, though at varied rates, throughout the *p*H range, but, for a given concentration of hypochlorite, chlorination of the lignin or other impurities should be controlled largely by the extent to which hypochlorous acid is present, and should therefore take place to an appreciable extent only when the *p*H of the liquor is such as to permit of the existence of this acid in significant proportion, namely within the range about *p*H 7 to 2[3].

If hypochlorite acts solely as an oxidising agent, the total salt concentration, measured as chloride, of a solution used in bleaching should not change greatly, as the reaction should be in its simplest terms,

$$NaOCl \longrightarrow NaCl + O$$

$$\text{or } HOCl \longrightarrow HCL + O$$

the chlorides remaining in the solution.

When chlorination occurs, chlorine is removed from the solution into the material and the total salt concentration as chloride must fall. For simplicity this effect may be represented thus,

$$NaOCl + H\text{–}R \longrightarrow NaOH + Cl\text{–}R$$
$$\text{or } HOCl + H\text{–}R \longrightarrow HOH + Cl\text{–}R$$

A considerable fall in the total salt concentration as chloride may therefore be taken to indicate chlorination even though oxidation may also occur during the bleaching.

From experiments in which the change in salt concentration was followed when jute yarn was treated under standard conditions, it has been estimated that between *p*H 11·5 and 9 the fall is approximately one-fifth of the change in hypochlorite concentration and therefore in this range the hypochlorite functions as an oxidising and not to any important extent as a chlorinating agent. Under neutral conditions the corresponding fall was about 46 per cent. and in acid solution (*p*H about 5) approximately 85 per cent. of the change in hypochlorite concentration. In neutral solutions therefore the hypochlorite functions both as an oxidising and a chlorinating agent, whilst under acid conditions the effect is essentially one of chlorination.

**The Action on Yarns of Hypochlorite Liquors of Different *p*H.**

In order to examine the effects of hypochlorite liquors of different *p*H values, hanks of the standard yarn were prepared, wetted out in a cold 0·25 per cent. solution of Calsolene Oil and hydroextracted. They were then entered separately into hypochlorite solutions of the concentration and *p*H specified below, using a liquor/yarn ratio of 15 : 1, and treated for the stated time. Afterwards they were rinsed, antichlored in a 1 per cent. solution of sodium bisulphite, washed thoroughly and dried.

The liquors employed were prepared by suitable dilution of a concentrated stock solution of bleaching powder or sodium hypochlorite, and were buffered to *p*H 11 by addition of sodium carbonate, *p*H 7 by addition of sodium bicarbonate, and *p*H 4·2 by addition of aluminium sulphate[3]. The concentrations of available chlorine were nominally 3, 5 and 7 g. per litre at the start and treatment was continued for respectively 15, 30 and 60 minutes at room temperature (about 17 to 19°C.)

Table II shows the fall of concentration of available chlorine in the liquors during the treatments.

**Table II**

| | Available Chlorine g/litre | | | |
|---|---|---|---|---|
| | Start | After 15 minutes | After 30 minutes | After 60 minutes |
| Alkaline Hypochlorite ... | 3·05 | 1·85 | 1·53 | 1·25 |
| | 5·10 | 3·33 | 2·56 | 1·81 |
| | 7·15 | 4·97 | 4·40 | 3·32 |
| Neutral Hypochlorite *p*H 7 | 3·03 | 1·06 | 0·71 | 0·28 |
| | 5·06 | 2·27 | 1·31 | 0·64 |
| | 7·01 | 2·48 | 1·49 | 0·92 |
| Acid Hypochlorite *p*H 4·2 | 2·95 | 1·10 | 1·06 | 0·85 |
| | 5·11 | 2·95 | 2·35 | 1·50 |
| | 7·20 | 4·40 | 4·01 | 3·70 |

From these figures it is evident that the consumption of available chlorine in a given time is least for the alkaline liquors, greatest for neutral ones and intermediate for the acid solutions, although the difference between the consumptions under acid and alkaline conditions is not very pronounced.

In the next Table, III, are recorded the percentage consumptions of chlorine based on the weight of yarn, and it is shown that with the neutral

solutions the percentages are at least 50 per cent. higher than for the other solutions. It is notable in fact that with a neutral liquor of initially 7 g. of available chlorine per litre the jute yarn is seen to have consumed 7 per cent. of its weight of chlorine in 15 minutes and over 9 per cent. in 60 minutes. For comparison it may be stated that the proportion of available chlorine consumed in 30 minutes by brown linen yarns from a neutral hypochlorite solution of original concentration 5 g. per litre, is only about 3 to 3·5 per cent., but it must be remembered that the lignin content of jute (12-14 per cent.) is very much higher than that of linen (3-6 per cent.[19]) and that much of the available chlorine is probably used up in chlorinating and oxidising this substance.

**Table III**

ACTION OF HYPOCHLORITE SOLUTIONS ON JUTE YARN

| Nominal concentration of Av. Cl. in Liquor | 3 g/l. | | | 5 g/l. | | | 7 g/l. | | |
|---|---|---|---|---|---|---|---|---|---|
| Time of Treatment | % of Av. Cl. on Jute | B.L. Lb. Dry | Wet | % of Av. Cl. on Jute | B.L. Lb. Dry | Wet | % of Av. Cl. on Jute | B.L. Lb. Dry | Wet |
| Untreated ... | Nil | 8·1 | 7·1 | (Grand mean values) | | | — | — | — |
| ALKALINE HYPOCHLORITE | | | | | | | | | |
| 15 mins. ... | 1·8 | 7·8 | — | 2·7 | 7·5 | 7·6 | 3·3 | 7·8 | 7·4 |
| 30 ,, ... | 2·3 | 7·7 | — | 3·8 | 7·8 | 6·9 | 4·1 | 8·3 | 7·3 |
| 60 ,, ... | 2·7 | 7·9 | — | 4·9 | 7·9 | — | 5·8 | 7·9 | 6·9 |
| NEUTRAL HYPOCHLORITE | | | | | | | | | |
| 15 mins. ... | 3·0 | 7·8 | 7·5 | 4·2 | 6·7 | 6·1 | 6·8 | 6·1 | 5·8 |
| 30 ,, ... | 3·5 | 7·8 | 7·3 | 5·6 | 7·0 | 6·4 | 8·3 | 6·4 | 5·8 |
| 60 ,, ... | 4·1 | 8·3 | 7·9 | 6·7 | 6·5 | — | 9·2 | — | 5·8 |
| ACID HYPOCHLORITE | | | | | | | | | |
| 15 mins. ... | 2·8 | 6·8 | — | 3·2 | 3·6 | — | 4·2 | 4·6 | — |
| 30 ,, ... | 2·8 | 6·6 | — | 4·1 | 3·2 | — | 4·8 | 5·0 | — |
| 60 ,, ... | 3·2 | 6·9 | — | 5·4 | 2·7 | — | 5·3 | 1·5 | — |

In the same Table III, are shown the mean breaking loads of the yarn treated under the three conditions of *p*H. After the alkaline treatments the strengths of the yarns are good for all concentrations employed, whilst for the neutral conditions they are good for the lowest concentrations (3 g/l) of available chlorine but the loss is greater with increasing concentration. It will be observed that the percentage consumption of available chlorine by the jute is roughly the same for the treatments with neutral hypochlorite of 3 g. per litre concentration as for the corresponding ones with alkaline hypochlorite of 5 g/l concentration, and that the yarn strengths for these two conditions are approximately the same.

A marked fall of strength is to be noted for the treatments with the acid hypochlorite liquors, particularly at the high concentrations, and it is clear therefore that whereas under practical bleaching conditions alkaline solutions may be used without detriment to strength, and neutral liquors are safe in relatively low concentration, the use of acid solutions should be avoided. It is shown later that the undesirable effects of acid hypochlorite are even more pronounced if the treatment is followed by a steep in hot alkaline solutions.

Apart from chlorine consumption and yarn strength, the colour of the treated jute also varies to a marked extent with change of *p*H of the hypochlorite. Acid solutions lighten the shade to some extent but also impart a yellowish brown tone, neutral liquors give a slightly better colour but the shade is

only lightish brown and the bleaching effect must be considered poor even for the most severe treatment for one hour with a solution of 7 g. of available chlorine per litre. A very marked difference is shown however for the treatments in alkaline solutions where a much lighter shade corresponding to a " half white " is given. The improvement with increasing concentration of available chlorine from 3 to 7 g. per litre is only slight and the bleaching effect is good even at the lowest concentration.

No very close approach to a white can be obtained by means of hypochlorite alone, even with alkaline solutions, and the jute must receive additional treatment if better bleaching is to be obtained.

**Change of pH of Alkaline Hypochlorite Liquors and its Effect on the Properties of the Yarn**

When jute is treated with alkaline hypochlorite liquors, a fall of *p*H takes place unless precautions are taken by the addition of buffer salts to maintain the alkalinity, and the colour, weight loss and strength of the bleached material may vary according to the *p*H conditions governing the course of the treatment. For example, if the *p*H falls from a high value to near to the neutral point the colour of the bleached yarn may be better or worse than that of a sample treated under conditions such that no great change of *p*H is possible. In order to examine these effects, sample bundles of the jute were wetted out and treated in hypochlorite solutions all of the same original concentration (5 g/l) of available chlorine for 30 to 60 minutes at 15 : 1 liquor ratio but under different conditions of *p*H control. They were afterwards antichlored, washed, dried, compared for colour and otherwise tested.

The experimental conditions and the results are recorded in Table IV.

**Table IV**

| Treatment | Time | *p*H. | | % loss of weight | B.L. Lb. | | Colour |
|---|---|---|---|---|---|---|---|
| | | Start | End | | Dry | Wet | |
| 1. Bleach Powder. Unbuffered. | 30 mins. at 16-17° C. | 11·1 | 8·1 | 6·2 | 8·0 | — | Slightly worse than 2, 3 and 4. |
| 2. NaOCl. Unbuffered. | ,, | 11·1 | 9·0 | 4·9 | 7·5 | 7·1 | All half white colour. No real differences of shade among them. |
| 3. NaOCl. +0·5 g/l. $Na_2CO_3$. | ,, | 11·2 | 9·2 | 5·0 | 7·9 | 6·5 | |
| 4. NaOCl. +2 g/l. $Na_2CO_3$ | ,, | 11·3 | 9·9 | 6·1 | 8·3 | 7·1 | — |
| 5. NaOCl. Unbuffered. | 60 mins. at 20° C. | 11·1 | 8·8 | 7·25 | 8·3 | 7·2 | Better colour than 1 to 4 above. |
| 6. NaOCl. +10 g/l. $Na_2CO_3$. | ,, | 11·5 | 10·3 | 8·6 | 7·2 | 6·8 | Noticeably better than 5. |
| Original untreated yarn | | | | ... | 8·1 | 7·1 | |

From the results it is evident that the fall of *p*H is greatest for the unbuffered bleaching powder solution, which contains only a small reserve of alkali determined largely by the solubility of lime and the temporary hardness of the water used in preparing the solution. With the sodium hypochlorite solutions, which contain a greater reserve of alkali, the fall is relatively less, but even the addition of 10 g. of sodium carbonate per litre fails to maintain the *p*H at its original value. This effect is to be expected because the buffering effect of sodium carbonate is not particularly good in the neighbourhood of *p*H 11·5. Nevertheless in trial 6, where this quantity was added the *p*H did not fall below 10, and the colour of the bleached jute was better than that in any of the other experiments. The effect shown in experiments 5 and 6 of prolonging the time, and probably also of increasing

slightly the temperature of the treatment, in producing better bleaching should be noted. With longer time there is also greater uniformity of colour throughout the material, although at the same time there appears to be a small increase in the loss of weight.

**Hot Alkaline and Peroxide Bleaching Treatments following Treatment with Hypochlorite**

As already stated it is common practice in purifying certain vegetable materials (for example linen) to follow a hypochlorite treatment by a steep in a hot solution of sodium carbonate for the purpose of removing coloured and other impurities rendered soluble in alkali as a result of their modification by the hypochlorite. It is also known, particularly in linen bleaching, that a treatment in hot peroxide solution following a hypochlorite bleach is capable of giving a good white without significant chemical degradation of the material[4, 5]. The effects of such after-treatments on jute are shown by the results in Table V.

**Table V**

**Breaking Loads (Dry)**

(Mean B.L. of Original Untreated Yarn 8·1 lb.)

| Concentration of Av. Cl. | Time, Alkaline | Hypochlorite | | |
|---|---|---|---|---|
| | | Alkaline. | Neutral | Acid |
| Hypochlorite treatment only. | (mins.) | (lbs.) | (lbs.) | (lbs.) |
| 3 g/l. | 15 | 7·8 | 7·8 | 6·8 |
| | 30 | 7·7 | 7·8 | 6·6 |
| | 60 | 7·9 | 8·3 | 6·9 |
| 5 g/l. | 15 | 7·5 | 6·7 | 3·6 |
| | 30 | 7·8 | 7·0 | 3·2 |
| | 60 | 7·9 | 6·5 | 2·7 |
| 7 g/l. | 15 | 7·8 | 6·1 | 4·6 |
| | 30 | 8·2 | 6·4 | 5·0 |
| | 60 | 7·9 | — | 1·5 |
| Hypochlorite followed by hot Sodium Carbonate. | | | | |
| 3 g/l. | 15 | 7·1 | 7·0 | 7·6 |
| | 30 | 7·5 | 8·7 | 5·6 |
| | 60 | 7·5 | 7·25 | 5·4 |
| 5 g/l. | 15 | 7·6 | 7·3 | 5·1 |
| | 30 | 7·2 | 6·7 | — |
| | 60 | 7·2 | 4·5 | — |
| 7 g/l. | 15 | 7·6 | 5·8 | Very low |
| | 30 | 8·0 | 6·5 | (results |
| | 60 | 7·8 | 6·6 | unreliable) |
| Hypochlorite followed by Peroxide. | | | | |
| 3 g/l. | 15 | 6·2 | 7·6 | 7·1 |
| | 30 | 6·7 | 8·0 | 6·3 |
| | 60 | 7·2 | 6·9 | 7·0 |
| 5 g/l. | 15 | 7·4 | 5·8 | 6·1 |
| | 30 | 6·7 | 5·6 | 5·2 |
| | 60 | 7·1 | 5·6 | 4·5 |
| 7 g/l. | 15 | 7·1 | 6·1 | — |
| | 30 | 7·9 | 5·9 | 4·6 |
| | 60 | 7·4 | 6·6 | 3·9 |

Samples of the jute yarns bleached with alkaline neutral and acid hypochlorite as already described were treated further as follows, using again a liquor/yarn ratio of 15 : 1.

(*a*) Steeped in 0·5 per cent. solution of sodium carbonate at 60-65°C. for one hour.

or (*b*) Bleached for two hours, at 65°C. in a solution containing sodium peroxide, sodium bicarbonate, and sodium silicate.

Results of tests on the peroxide liquor were,

| | Conc. of Peroxide g/l $Na_2O_2$ | Alkalinity (Normality) |
|---|---|---|
| Start ... ... ... ... ... | 2·14 | 0·18 N. |
| After 30 minutes ... ... ... | 1·95 | 0·18 N. |
| After 2 hours ... ... ... ... | 1·30 | 0·10 N. |

Although the breaking load results are somewhat irregular, it is possible to draw the following significant conclusions, namely,-

(1) The effects of either a steep in hot sodium carbonate or in dilute alkaline peroxide are small if in the first place the yarn has been treated with alkaline hypochlorite, and this statement obtains for all the concentrations of the hypochlorite solutions examined and for treatments up to one hour's duration.

(2) After a neutral hypochlorite treatment, the losses of strength are relatively small for the lowest concentration (3 g/l) of available chlorine, but for higher concentrations both the sodium carbonate and peroxide solutions cause appreciable further losses of strength.

(3) After acid hypochlorite both the sodium carbonate and the peroxide treatments reduce strength very considerably particularly where the higher concentrations of available chlorine and the longer times are concerned. Thus the effect of hot sodium carbonate following a treatment for 1 hour with an acid hypochlorite liquor of 7 g. of available chlorine per litre is so severe that strength measurements can be made only with difficulty.

It follows therefore that whereas after alkaline hypochlorite hot alkaline treatments may be given without serious effect on strength, they must be avoided when an acid solution has been used, but are permissible after neutral hypochlorite provided that the concentration of available chlorine in this solution does not greatly exceed 3 g/l at the stated liquor/yarn ratio.

The sodium carbonate steep following treatment with alkaline hypochlorite causes a further slight improvement in colour with a change from a yellowish to a greyer tone. After neutral hypochlorite it gives no significant improvement but again a slight alteration in tone, whilst after an acid solution the colour, which was already bad, is if anything worse.

A mild peroxide treatment under the conditions stated improves considerably the appearance of yarn previously bleached with alkaline hypochlorite, and after this treatment there is some approach to white.

After neutral hypochlorite there is also some improvement, although under the conditions employed the colour is not as good as that given by the above alkaline sequence, but after acid hypochlorite the effects of the peroxide are still very poor and the yarn still remains brown in colour.

**The Effects of an Oxidising Steep and of Subsequent Hypochlorite and Peroxide Treatments.**

The presence of a small proportion of peroxide in a sodium carbonate steeping liquor has a marked effect on the colour of jute and enables better shades to be obtained after subsequent treatments with hypochlorite and peroxide. The effects obtained by combining such a treatment with other bleaching operations are illustrated by the results of the following trials.

A batch of jute yarn was steeped for two hours at 65°C. in a solution containing per litre 9·5 g. of sodium carbonate, 8·5 g. of sodium silicate and 1 g. of sodium peroxide. The colour obtained was light fawn, considerably lighter than when the liquor contains no peroxide. Portions of this material were treated with neutral or alkaline hyoochlorite solutions of the concentrations and for the times stated in Table VI, and portions of these samples again were subsequently immersed for one hour at 65°C. in a solution containing per litre 8·5 g. of silicate, 3 g. of sodium peroxide, and the equivalent of sodium bicarbonate (see section on Further Experimental Details).

For comparison also, a further batch of yarn that had received no steep, was treated first with alkaline hypochlorite and then with peroxide under conditions already used for the previous steeped yarns (see No. 9, Table VI).

The liquor/yarn ratios throughout were approximately 15 : 1 and handling was avoided as far as possible until the yarns were dry.

The results obtained are collected in the follcwing Table VI.

**Table VI**

| Treatment | % Loss of Wt. | B.L. | | Colour |
|---|---|---|---|---|
| | | Dry | Wet | |
| 1. Original yarn ... ... ... ... | — | 8·1 | 7·1 | |
| 2. Oxidising steep at 15 : 1 ratio for 2 hours at 65°C. | 6·0 | 8·6 | 6·7 | Slightly lighter than 1. |
| 3. Oxidising steep, Neutral hypochlorite 2 g. Av.Cl/1 20 minutes | 7·4 | 8·4 | 5·6 | Lighter than 2. |
| 4. Oxidising steep, alkaline hypochlorite 2 g. Av. Cl/1, 20 minutes | 6·9 | 8·4 | 6·0 | Lighter than 3 (fair bleach). |
| 5. Oxidising steep, alkaline hypochlorite 3 g. Av. Cl/1, 30 minutes | 6·5 | 9·8 | 6·7 | Lighter than 4. |
| 6. As (3)+peroxide bleach 3 g/l $Na_2O_2$, 6·6 g/l $NaHCO_3$, and 8·5 g/l of Silicate | 9·8 | 8·8 | 1·9 | Considerably better than 5—good bleach. |
| 7. As 4 then peroxide bleach as 6 ... | 7·0 | 8·9 | 4·1 | Slightly better and more lustrous than 6. |
| 8. As 5 then peroxide bleach as 6 ... | 8·0 | 9·5 | 4·2 | Very slightly better than 7. |
| 9. No steep, alkaline hypochlorite 3 g. Av. Cl/1, for 30 minutes then peroxide bleach as 6 | — | 9·5 | 5·2 | Intermediate in colour between 6 and 7. |
| 1A, No. 1, after extraction with Trichlorethylene | | 8·6 | | |
| 2A, No. 2, do. | | 8·8 | | |
| 5A, No. 5, do. | | 9·5 | | |

It will be observed that for all the conditions of treatment the losses of weight of the yarns are low, the highest value being 9·8 per cent. for sample No, 6, given the neutral hypochlorite treatment before peroxide, but with this solution chlorination as well as oxidation can occur as already shown. The strengths of the dry yarns must also be regarded as good throughout. It is noticeable that they are in all cases higher than that of the original yarn itself, and with one exception, they tend to be higher after the peroxide stage than after the hypochlorite treatment.

At the end of the table, values are given for certain of the yarns that were extracted with trichlorethylene for the purpose of removing oil that might have acted as a lubricant. It is seen that the breaking load of the untreated yarn No. 1 is raised to some extent as a result of this extraction (from 8·1 to 8·6), but not to a value as high as those for the peroxide bleached yarns. In other words, the increase of strength cannot be ascribed merely to removal of lubricant. On suitably staining the yarns, all fat and wax was seen to be removed from those that had had the final peroxide bleach but not from

those that had received only an alkaline steep or a steep and hypochlorite treatment. It was also noticeable that the peroxide bleached yarns were more compact than the untreated or steeped yarns, and it would seem therefore that the increase of strength observed is to be attributed to an increase in the internal friction in the yarns between the fibres themselves, occasioned by both the removal of lubricant and by closer setting of fibre on fibre after the swelling in the hot solutions and subsequent drying.

When single fibres or short lengths of yarn from unbleached and bleached material were immersed in water it was seen that whilst the former remained almost motionless, the latter showed a lively untwisting effect closely resembling the liveliness of a viscose crepe yarn under the same conditions of treatment. This effect may be taken as evidence that the jute material had been previously dried whilst in a strained, or more closely packed, state. An increase in the twist of the yarn caused by shrinkage of the treated hanks can also increase strength and contribute to this liveliness, but here although the shrinkage was actually greatest for the yarns that had received two hot treatments and showed the highest dry strength (Nos. 6, 7 and 8 of Table VI) the amount of such shrinkage (about 2 per cent.) was very small and its effect in increasing twist could therefore also have been only small.

The values for Nos. 2A and 5A in Table VI are not significantly different from those for the corresponding treated but unextracted yarns 2 and 5, so that here the removal of any residual material soluble in the organic solvent has had no effect in further increasing the strength.

The wet strengths in Table VI show a reduction for each of the successive stages of the wet processing, and this reduction is greatest for the yarns that were submitted to all three stages, namely steep, hypochlorite and peroxide bleach. Even under the careful conditions employed in the trials now summarised the loss of wet strength is considerable and it thus appears that such loss is an unavoidable accompaniment of the bleaching processes. The greater loss attending the peroxide stage is probably due in part to the fact that modification of lignin and hemicellulose by the action of the hypochlorite liquors yields products that are soluble in the hot alkaline peroxide solutions and that damage already latent in the partly bleached jute is realised only after the final hot alkaline treatment. Thus when yarn from samples 3 and 5 of Table VI was further treated for two hours at 65°C. in one case (*a*) with a peroxide liquor 0·2N in excess alkali and in the other (*b*) with 0·2N. alkali in the absence of peroxide the following wet strengths were obtained.

| | Wet B.L. lb. | | |
|---|---|---|---|
| | | then (a) Peroxide 0·2N in Alkali | or (b) 0·2N Alkali |
| *Sample* 3. Alkali Steep and Neutral hypochlorite | 5·6 | 1·3 | 2·5 |
| *Sample* 5. Alkali Steep and Alkaline hypochlorite | 6·7 | 3·25 | 3·7 |

From these values it is obvious that treatment with the hot dilute alkali alone reduces the wet strength very considerably although the loss is greater if peroxide is also present. Although the mechanism may not be the same, it may be recalled that latent damage in overbleached rayon and other cellulose textiles is fully realised only after treatment of the material in hot alkaline liquids.[6]

The comments in the last column of Table VI indicate the degree of bleaching to be obtained by the different treatments listed. The colours

of samples 6, 7, 8 and 9 that had received a final peroxide treatment were pale cream and must be considered to represent good bleaching for jute yarns.

Although the three-stage processes in which alkaline hypochlorite was used gave perhaps slightly better results than when a neutral solution was employed in otherwise the same sequence, the differences in colour, strength and loss of weight were not pronounced, so that either sequence may be used satisfactorily under the conditions stated. When no steep was given (Sample 9, Table VI), the bleaching effects of alkaline hypochlorite followed by peroxide were also good.

**The Importance of Liquor/Yarn Ratio**

In other experiments similar conditions were employed but the ratio of liquor to yarn in all the operations was twice that used for the yarns treated as in Table VI. The conditions for the oxidising steep and the peroxide bleach were the same as before and the neutral and alkaline hypochlorite solutions of 3 g. of Av. Cl. per litre were used for 30 minutes. The results of tests on the yarns are given in Table VII.

**Table VII**

| Treatment | Loss of Weight | B.L. Dry | lb. Wet | Colour |
|---|---|---|---|---|
| 1. Oxidising steep only | | 8·0 | 4·7 | Light brown |
| 2. Oxidising steep, Alk. hypochlorite, Antichlor. | 11·4 | 7·0 | 4·5 | Good colour (yellow) |
| 3. Oxidising steep, Alk. hypochlorite, Peroxide | 12·1 | 8·0 | 3·4 | Good white |
| 4. Oxidising steep, Neutral hypochlorite, Antichlor. | 12·5 | 8·0 | 3·0 | Light brown |
| 5. Oxidising steep, Neutral hypochlorite, Peroxide | 25·0 | 4·4 | Too weak to measure | Almost pure white, slightly better than (3) |

With this higher ratio the losses of weight are seen to be greater than for the 15 : 1 ratio, and for the sample No. 5 treated with neutral hypochlorite in the intermediate stage the very high value of 25 per cent. was realised. The "dry strengths" are satisfactory except for No. 5 again, for which a serious loss is shown, whilst in the wet state this yarn was extremely weak, owing to the excessive removal of incrustants.

The colour of the yarn No. 2 steeped and treated with alkaline hypochlorite was good, with a yellowish shade, whereas the corresponding yarn No. 4 treated with the neutral solution, was only light brown. After the final peroxide bleach however this order was reversed, the better white being obtained with the sample treated with neutral hypochlorite. This particular yarn was almost pure white.

The corresponding "alkaline hypochlorite" sample No. 3 was only slighter darker in colour than No. 5, and had retained strength satisfactorily, and the inference to be drawn is that if strength is to be conserved, alkaline hypochlorite solutions are to be preferred to neutral ones, in the sequence oxidising steep, hypochlorite, peroxide bleach, when high liquor/yarn ratios are employed.

In the bleaching of cotton materials it is known that good results can be obtained by peroxide treatments without an intermediate stage. The usual two-stage bleach comprises first a steep in a hot solution of sodium carbonate containing a relatively small proportion of peroxide and then a second treatment in an alkaline bath containing more peroxide. Experiments illustrating this method are described below.

Jute yarn was steeped for two hours at 65°C. in a solution containing per litre, 8·5 g. of sodium silicate 1·0 g. of sodium peroxide, and 2·2 g. of sodium bicarbonate.

It was washed with water, the batch was divided and the various equal portions were treated for two hours at 65°C. in solutions of the same initial concentration of peroxide (about 3 g/l as $Na_2O_2$) but of different alkalinity.

Tests on these solutions and on the yarns gave the following results:

**Table VIII**

| Time | Peroxide (g/l $Na_2O_2$) | Alkalinity | | B.L. lb. Dry | B.L. lb. Wet |
|---|---|---|---|---|---|
| | | | Steep only | 6·2 | 3·9 |
| 1. Start | 2·88 | | | | |
| 2 hours | 1·05 | 0·21 N. | Steep and peroxide | 5·8 | 3·4 |
| 2. Start | 2·88 | | | | |
| 2 hours | 1·23 | 0·15 N. | ,, | 6·2 | 3·4 |
| 3. Start | 2·84 | | | | |
| 2 hours | 1·33 | 0·11 N. | ,, | 6·4 | 4·1 |
| 4. Start | 2·88 | | | | |
| 2 hours | 2·13 | 0·05 N. | ,, | 5·6 | 3·8 |

It is evident that after the hot steep the peroxide treatments have little further effect on either dry or wet strength but the relatively low values for the latter indicate that the incrustants are modified to a considerable extent. The fall of concentration of peroxide is seen to be greater the higher the concentration of alkali but the variation in shade of the yarns was only small and the material treated with the solution of highest akalinity was only slightly whiter than that treated with the solution of lowest alkalinity.

**Chlorine Peroxide and Sodium Chlorite**

Until recently little interest has been shown in chlorine peroxide or chlorite solutions, and these substances have been considered more as interesting rare chemicals than as technically useful reagents. Their oxidising and bleaching properties have been known for some years and on the laboratory scale they have been used for a few purposes. For example, Sarkar[7] has mentioned the complete removal of lignin from jute by treatment of the material with chlorine peroxide in the presence of moisture and Speakman[8] has shown that the same product is capable of imparting a non-shrink finish to wool when used in carbon tetrachloride solution. In America, however, attention has been directed towards the technical use of chlorite solutions for textile bleaching purposes and claims have been made in patent specifications and in technical literature that this product is unique among textile bleaching agents because it enables bleaching to be done in acid solution at elevated temperature with no significant attack of cellulose material even if metals are present that commonly are capable of catalytic action.[9]

In the present investigation it has been considered of interest to examine the effects of these reagents and to compare their behaviour with that of some of the other substances employed. The trials made and the conclusions drawn from the results are discussed below.

A small hank of jute yarn previously moistened with water was exposed to excess of chlorine peroxide gas for four hours at 0°, to 7°C. A change in colour to yellowish brown occurred fairly quickly but no significant further lightening of the shade was apparent after 2½ hours. In order to find the extent to which lignin had been modified, the sample was extracted for 1½ hours with an alkaline sulphite solution containing 10 g. of the sodium salt per litre. The loss of weight after this extraction was 16·5 per cent. whilst the lignin content was reduced from 13·8 per cent. to 6·1 per cent. The dry strength, originally 8 lb., was 5·0 lb. and the wet strength too low to be measured.

When chlorine peroxide is passed into water to saturation, a *p*H below 3 is obtained. Such a solution bleaches only slowly at ordinary tempera-

ture, whilst if the temperature is raised a considerable loss of gas occurs. On the other hand good bleaching is realised if the solution is buffered to *p*H 5 and the jute is treated near to the boil. The results of experiments made under different conditions with solvent extracted, but otherwise untreated jute yarn are given in Table IX.

**Table IX**

| No. | Treatment | Temp. | Time hrs. | % Loss of wt. | % Lignin Content of residual material | Colour | Strength Dry lb. | Strength Wet lb. |
|---|---|---|---|---|---|---|---|---|
| | Original yarn | — | — | — | 13·8 | — | 8·1 | 7·1 |
| 1. | $ClO_2$ 6g/l at *p*H 3-2, Liq. ratio 20 : 1 | 0°C. to 6°C. | 70 | 3·1 | 7·25 | Golden Yellow | 7·8 | 6·1 |
| 2. | $ClO_2$ 5°4g/l, *p*H 5·1, Liq. ratio 15 : 1 | 85°C. | 1 | 3·2 | 10·2 | Fair white | 7·9 | 6·9 |
| 3. | As 2 but at | 16°C. | 24 | 3·0 | 11·5 | Golden Yellow | 8·3 | 7·9 |
| 4. | No. 1 extracted twice with 2% neutral sulphite liquor at | 85° to 90°C. | 1 | 9·5 | 1·7 | Pale fawn | 8·4 | 1·2 |
| | (Total loss of weight and lignin content of (4) based on original yarn) | | | 12·3 | 1·5 | | | |

It is seen that at *p*H 5 and 85 C. a fair bleach with low loss of weight may be obtained in one hour, whereas at lower *p*H and low temperature only a golden yellow colour is reached after many hours.

The effect of treating sample No. 1 with a neutral sulphite liquor to remove lignin after the $ClO_2$ treatment is interesting because the total loss of weight after both treatments (12·3 per cent.) is the same as the loss of lignin (13·8—1·5 per cent.) which indicates that substantially no hemicellulose, but only lignin, is thus removed.

The close agreement shown is of course to some extent accidental because the methods employed for determining both lignin and loss of weight are not highly accurate. Moreover the precise significance of the results of lignin determinations is unknown and the material actually weighed as lignin is probably a chemically modified substance rather than true lignin as it exists in the original fibre. Nevertheless the approximate agreement between these values has been substantiated in other experiments and the methods of treatment may be of some analytical importance. It has been observed that when alkaline instead of neutral sulphite liquors are employed higher losses of weight are obtained.

**Sodium Chlorite**

From other work on textile materials it is known that chlorite solutions bleach best at a *p*H of about 5 and under boiling or nearly boiling conditions. These conditions were accordingly used in examining the behaviour of chlorite solutions towards jute. A concentrated solution of sodium chlorite previously prepared in the laboratory was suitably diluted with acetic acid-sodium acetate buffer liquid, adjusted to give an initial *p*H of 5·2, and samples of jute, previously wetted out in a 0·5 per cent. solution of Calsolene Oil were treated as in Table X. In order to give a direct comparison between $ClO_2$ and chlorite, further experiments were made in which equivalent solutions of these reagents were buffered to the same *p*H (5·1) and samples of jute yarn were treated with them under identical conditions of temperature, time and liquor ratio. The results of these experiments are also included in Table X (see Nos. 5 and 6).

**Table X**

| | Treatment | Temp. | Time | % Loss of wt. | % Lignin content | Colour | Strength Dry lb. | Strength Wet lb. |
|---|---|---|---|---|---|---|---|---|
| 1. | $NaClO_2$ 13 g/l, *p*H 5·2 | Boil | 45 min. | 9·4 | 9·7 | Creamy white | 7·3 | 5·4 |
| 2. | (Loss of chlorite during treatment 63 %) | | | | | | | |
| | As (1) then extracted with 1% NaOH | 95°C. | 2 hrs. | 28·0 | 5·0 | White | Less than 3 lb. | Nil |
| 3. | As (1) then extracted with 2% sulphite *p*H 7·2 | 90°C. | 2 hrs. | 1·2 | 7·7 | Fair white slightly better than 1 | 7·1 | 4·3 |
| 4. | $NaClO_2$, 7·2g/l at *p*H 5·1 | 85°C. | 2 hrs. | 6·4 | 10·8 | Fairly good bleach, Cream | 7·7 | 6·4 |
| | (Loss of Chlorite during treatment 89 % | | | | | | | |
| | *Direct comparison of $ClO_2$ and Chlorite at same pH* | | | | | | | |
| 5. | $NaClO_2$ 4·8g/l *p*H 5·1 | 85°C. | 1 hr. | 4·3 | 14·5 | Fair bleach, Cream | 7·9 | 6·7 |
| 6. | $ClO_2$, 2·8g/l at *p*H 5·1 | 85°C. | 1 hr. | 4·6 | 14·2 | Equal to that of 5 | 8·1 | 6·6 |

From experiments 1 and 4 it is seen that good colours may be obtained without serious loss of weight and strength by bleaching with hot chlorite solutions at *p*H 5 for comparatively short times.

Experiments 5 and 6 show as would be expected that chlorine peroxide and sodium chlorite solutions of equivalent concentration give identical effects under the same conditions stated. The results of other experiments in Tables IX and X are referred to in the subsequent section.

**Non-cellulose Impurities and the Strength of Jute Yarns**

The experiments with chlorine peroxide and chlorite solutions enable certain conclusions to be drawn regarding the importance of the non-cellulose impurities in contributing to the strength of jute yarns. Thus experiment 4 of Table IX shows that a yarn from which substantially all the lignin has been removed, but which retains practically all its hemicellulose, has a very low wet strength although its dry strength is still high. In experiment No. 1 of Table I, it is shown on the other hand that a yarn that retains most of its lignin but loses its hemicellulose (as shown by the high weight loss of 23 per cent.) also has a low wet strength although again its dry strength is relatively high. Again if No. 3 of Table X is compared with No. 4 of Table IX it is seen that the total losses of weight in the two cases are roughly the same (10 to 12 per cent.) but the latter material which has lost almost all its lignin has a very low wet strength, whilst the former with a much higher lignin content has also a higher wet strength.

If both lignin and hemicellulose are removed completely by careful treatment of jute yarn with acid hypochlorite and then boiling caustic soda under conditions that have but little effect on the cellulose (see Table XI No. 10), the material is reduced to a fibrous mass having no strength at all as yarn. Hence, in order to retain dry strength it is necessary to have present at least part of the non-cellulose incrustants to act as a cementing material between the cellulose ultimates, and apparently it does not matter greatly whether lignin or hemicellulose predominates.

If high wet strength is to be maintained, a considerable proportion of both these incrustants must be present, and as shown by other results in the various tables the greater the extent to which both are retained the higher is the wet strength.

It is a matter of common knowledge that jute yarns become weaker on wetting with water, and in this respect they may be compared with sized soft-twisted cotton yarns which in the dry state have a tensile strength considerably in excess of that of the corresponding unsized yarn. Since the strength of the size film is low, and this film by itself contributes little to the strength of the individual cotton hair, its effect is to increase by its adhesive properties the internal friction existing in the yarn between adjacent and overlapping cotton hairs, thus permitting a greater proportion of their individual strength to be realised than is possible when slippage can occur. In their work on the effect of humidity on cotton yarn, Pierce and Stephenson[10] showed that under equilibrium conditions above 80 per cent. R.H. starch size rapidly loses its strength and resilience and the strength of the sized yarn falls with further increase of humidity until at 100 per cent, corresponding to saturation of the material it becomes identical with that of the original unsized yarn. Furthermore the extensibility of a dry sized cotton yarn of low twist is much less than that of the corresponding unsized, but with increase of humidity and consequent softening of the size film the extensibility of the sized material increases (in some cases by as much as 30 per cent.) until at its maximum it is identical with that of the unsized yarn. The higher strength of the sized cotton yarn is due to the fact that slippage of the comparatively short cotton hairs, which in unsized yarns is a very important factor in determining tensile strength, is prevented.

In jute yarns the fibres are normally very long compared with cotton hairs, so that fibre slippage is relatively unimportant. On this account for present purposes of comparison a jute yarn may be considered to be composed essentially of the very short cellulose ultimates resembling the hairs of the cotton yarn, surrounded by the hemicellulose, lignin and other incrustants that may be compared with the starch size. As with starch, these incrustants of themselves have relatively little strength. Actually it might be considered more apposite to compare a jute fibre with a cotton yarn, but since the jute fibres in the yarns are very long so that slippage and yarn structure can be ignored, the behaviour of the yarn is governed essentially by that of the individual fibre, and the yarn to yarn comparison is therefore permissible for purposes of illustration.

Barker[11] has recently stated that the torsional rigidity of the wet jute fibre is only one-fifth to one-sixth that of the corresponding dry fibre, and Macmillan[20] has shown that maximum swelling is attained when the moisture regain has reached about 32 per cent. and that then the fibre is in a completely plastic condition. Furthermore, mean extensibility results on dry and wet unbleached jute yarn observed during the course of the present work gave the values, dry 2·05 per cent. and wet 3·3 per cent. and on the same yarn bleached, dry 2·8 per cent. and wet 3·5 per cent. thus revealing a marked increase on wetting the yarn. Hence the effects of water on the starch size and on the jute incrustants are similar.

Brown, Mann and Peirce[12] have shown that an increase in the equilibrium humidity from about 70 per cent. to 100 per cent. has only a small effect on the strength of cotton hairs, although the effect actually observed is one of increase and not decrease of strength, and by analogy it may be argued that no diminution of tensile strength of the cellulose ultimates of jute is produced by wetting the fibres. From these observations it may be postulated that the decrease of strength shown on wetting yarns is due not to weakening of the cellulose ultimates but to a swelling and softening action of the water on the non-cellulose incrustants in the same way as that of a sized cotton yarn is due to softening and loss of resilience of the starch size.

**The Yellowing of Bleached Jute on Exposure to Light**

A well-known disadvantage of partly bleached jute is that on exposure to sunlight it becomes discoloured in a very pronounced manner to a yellow-

ish or brown shade. A similar effect is shown by newspaper, which, since it contains a high proportion of ground wood also has a relatively high percentage of ligneous and other non-cellulose impurities. Pure cellulose as typified by cotton carefully bleached to a low fluidity and copper number is resistant to the discolouring effects of light, and since it has been shown earlier in this paper that bleaching, unless of a very drastic nature, fails to remove much of the non-cellulose impurities of jute it is reasonable to assume that the yellowing of this fibre is connected with its residual non-cellulose constituents and not with the cellulose itself.

In order to examine the factors contributing to the discoloration of jute, the materials described in Table XI were prepared and exposed under the same conditions for five hours to the light from a high intensity arc. The intensity of the light on the samples was about 200,000 foot-candles, that is, approximately 20 times the intensity of noon-summer sunlight, whilst the time allowed was roughly equivalent to 75 hours exposure to the standard C.P.A. lamp. All the samples were cooled during the exposure by means of a blast of cold, filtered humidified air.

The first seven samples form a series in which the lignin content of the jute yarn decreases progressively from 13 per cent. to about 2 per cent. and the removal of lignin was effected by various methods comprising mild alkaline steeps, hypochlorite, chlorite and reducing treatments. No. 10 represents jute fibre from which lignin and hemicellulose had been removed as completely as possible by means of acid hypochlorite and sodium hydroxide solution, whilst No. 11 was a cutting of jute-cotton fabric boiled under pressure and bleached with hypochlorite by the usual cotton process. Nos. 14 and 15 were samples of linen yarn bleached to respectively 3/4 and 4/4 white. They still contained some hemicellulose although their lignin contents, as indicated by qualitative tests, were nil. No. 16 was a fully bleached Egyptian cotton yarn of low fluidity and may be regarded as wholly pure cellulose.

From the last column of Table XI it is seen that samples 1 to 7 all yellowed to marked degree, whilst from visual examination of the exposed portions it was impossible to differentiate among them. In other words there was no decrease in the intensity of the yellowing with decreasing lignin content, and No. 7 with a very low lignin content was as deeply yellowed as the rest.

As expected, the cotton and linen yarns remained unchanged, even though the latter contained residual hemicellulose. The "delignified jute fibre" and the jute-cotton cloth were both unaltered, and while the result was anticipated for the fibre samples in view of the severity of their treatment and their high losses of weight, it was rather surprising to find that the cloth sample did not yellow. Subsequent examination of the jute weft revealed however that it had been bleached before being woven into the cloth and this pretreatment combined with the "cotton bleach" was evidently sufficient to remove impurities that would otherwise cause yellowing.

From the results recorded it is seen that ordinary bleaching treatments, even if rather severe, fail to eliminate completely the constituents responsible for the yellowing, and that yellowing occurs when lignin is present even though the residual proportion is very small. Thus No. 8 still showed slight discoloration yet its lignin content was only about 0·1 per cent.

Sample No. 13 represents hemicellulose isolated from bleached wood pulp and washed thoroughly to ensure freedom from chemicals. It contained no lignin. Although it may perhaps differ from the hemicellulose present in jute it is significant that it remained unchanged on exposure and from this result and from that on the linen yarns Nos. 14 and 15 which also contained hemicellulose, it appears that this substance does not contribute to the yellowing. The only samples of jute that remained unaltered

**Table XI**

| No. | Material | Treatment | %Lignin Content | %Loss of wt. | Results of Exposure |
|---|---|---|---|---|---|
| 1. | Jute Yarn | Oxidising steep | 13·1 | 6 | Marked yellowing |
| 2. | ,, ,, | Oxidising steep, neutral hypochlorite | 11·4 | 7·5 | ,, |
| 3. | ,, ,, | Chlorite 13 G/l at *p*H 5, 45 min. at boil | 9·7 | 9 | ,, |
| 4. | ,, ,, | Oxidising steep, Alkaline hypochlorite, Peroxide and hydrosulphite treatments | 9·5 | 9 | ,, |
| 5. | ,, ,, | Oxidising steep, neutral hypochlorite, peroxide | 7·9 | 10 | ,, |
| 6. | ,, ,, | Press. boil 1% NaOH, NaOCl. 4g. Av.Cl/l. | 2·3 | | Slightly yellow |
| 7. | ,, ,, | $ClO_2$ solution, 70 hrs. at ord. temperature. Extracted twice with hot (neutral) sulphite liquor | 2·0 | 12 | Marked yellowing |
| 8. | ,, ,, | Acid hypochlorite (5g. Av. Cl/l) 1% sodium sulphite hot (Twice) followed by 1% NaOH pressure boil | about 0·1 | | Very slightly yellow, hardly noticeable |
| 9. | ,, ,, | Chlorine peroxide and sulphite then press. boil 1% NaOH | Nil | | No discolouration |
| 10. | Jute fibre | Delignified with Cl and NaOH | Not detectable | 43 | ,, |
| 11. | Jute-cotton cloth | Boiled with NaOH under pressure and bleached with hypochlorite | ,, | — | ,, |
| 12. | Bleached hessian cloth | Previously browned by exposure to light and then re-bleached with hypochlorite | — | — | Brown re-developed |
| 13. | Hemicellulose isolated from bleached wood pulp. | | Nil | — | No discolouration |
| 14. | Linen yarn | Bleached to 3/4 white | Not detectable | abt. 10% | ,, |
| 15. | Linen yarn | Bleached to 4/4 white | ,, | — | ,, |
| 16. | Egyptian cotton yarn | Bleached to full white | ,, | 6 | ,, |
| 17. | Retted Flax fibre | Unbleached | — | — | Colour lightened |
| 18. | Unretted flax fibre | Unbleached | — | — | ,, |
| 19. | Coir fibre | Bleached with peroxide | — | — | Yellow |
| 20. | Sisal fibre | Untreated | — | — | ,, |
| 21. | Cellulose isolated from sisal | | Not detectable | | No discolouration |

were those in which lignin could not be detected and it is concluded that yellowing on exposure to light can be avoided only if the conditions of purification are severe enough to ensure that all lignin has been eliminated.

A necessary accompaniment of such severe treatment is that the jute is reduced essentially to pure cellulose of very short ultimate fibre length and consequently the strength of the treated yarn is very low indeed.

Sample No. 12 was a hessian cloth that had been bleached with peroxide to a very pale cream and then browned by exposure to light. The original cream colour was restored by bleaching the fabric in a dilute solution of sodium hypochlorite and the dry material was afterwards re-exposed under conditions similar to those used for the other samples. It is seen that the discoloration reappeared after this exposure and in some measure the effect therefore appears to be reversible.

In contrast to jute, unbleached retted and unretted flax fibres become lighter on exposure, and this effect is perhaps to be expected in view of the bleaching that occurs on exposure of linen yarns or fabrics to light in the

old established practice of "grassing." Unbleached linen has a lignin content estimated at 3 to 6 per cent[19] and if lignin is the primary cause of yellowing it might be thought that raw flax fibre would become discoloured, but here it is conceivable that both yellowing and bleaching occur and that the latter effect predominates. The chemical treatments normally given to linen materials that are subsequently "grassed" are probably sufficient either to eliminate lignin or to reduce it to a very small proportion and with such materials the exposure effect is presumably one of bleaching only.

Coir, which in its normal state is stated to contain about 30 per cent. of lignin, yellows badly even after a peroxide treatment, whilst sisal fibre with a content of about 6 per cent. also yellows. Lignin-free cellulose isolated from sisal by treatment with acid hypochlorite and hot sodium hydroxide solution, however, shows no discoloration.

With the exception of linen (where perhaps the effect may be masked) yellowing occurs with all the fibres examined that contain lignin and it is probable therefore that lignin itself is the constituent of the bast fibres that is directly responsible for the discoloration in the presence of light.

**Jute Cellulose**

Fairly pure cellulose may be isolated from jute by methods not involving boiling under pressure with alkali, for example by treating it with chlorine peroxide and then extracting with an alkaline sulphite solution, or by using first an acid hypochlorite treatment and then a hot extraction with alkali or alkaline sulphite followed by bleaching with alkaline hypochlorite or peroxide, but it is advisable to use conditions of pressure boiling with sodium hydroxide if the best effects are to be secured.[18]

Difficulty in obtaining a uniformly pure white cellulose lies mainly in the resistance to attack afforded by the dark root ends of the fibre and by residual fragments of bark. For this reason a chlorinating treatment with acid hypochlorite before boiling the material under pressure has advantages because then the lignin is attacked and disintegrated and the incrustants are more readily removed. If the preliminary acid hypochlorite stage is not employed, and the material contains much bark and dark coloured fibre it is generally impossible to obtain a clean white product by giving a single boil at 20 to 30 lb. pressure and then bleaching with alkaline hypochlorite. Generally more than one boil must be given although better results are obtained by cutting up the jute and by boiling at high pressure (55 lb./sq. in or over) preferably in a rotary vessel.

Since the incrustants in jute consume alkali in considerable proportion it is necessary to ensure that sufficient is present at the beginning of the boil. For example, batches of red and grey Tossa fibre were treated at 25 : 1 liquor ratio for one hour at *p*H 4 with hypochlorite solution containing 7 g. of Av.Cl/l, boiled at 20 lb. excess pressure for five hours in a 1 per cent. solution of sodium hydroxide, washed, bleached for one hour with alkaline hypochlorite (5g. of Av.Cl/l) and again washed. The product obtained was only creamy white in colour and still contained dark fibre and bark whilst examination of the liquor at the end of the boil showed that the sodium hydroxide was almosy completely consumed and that the initial concentration was therefore too low.

By using the same sequence, but with a 3 per cent. solution of sodium hydroxide, good effects were secured and approximately one third of the

**Table XII**

| Material | Total loss of wt. | % Lignin | % -cellulose | % Ash | Fluidity (0·5% soln. |
|---|---|---|---|---|---|
| Grey Tossa | 43 | — | 94 | 1·03 | 9·5 |
| Red ,, | 43 | — | 92 | 0·79 | 10·9 |
| ,, ,, (Cut up) | 43·3 | Not detected | 94 | 0·48 | 7·7 |
| with additional bisulphite extraction | 44·4 | ,, | 96 | 0·41 | 9·1 |

alkali remained at the end of the boil. Results obtained by this treatment are given below, and it is shown also that a steep in hot bisulphite solution after the acid hypochlorite stage and before the boil is not necessary for the purpose of removing more of the incrustants.

The effects of using more drastic conditions of boiling are shown by the following experiments in which no preliminary acid hypochlorite treatment was given. Here the material employed was an all-jute sacking fabric cut up into roughly 3-inch squares.

| Treatment | % Lignin | Fluidity 0·5% soln. | % Loss of wt. | Colour |
|---|---|---|---|---|
| Boil 1% sodium hydroxide 3 hours at 28 lb. pressure Alk. Hypochlorite 5g. Av.Cl/l 2½ hours | 4·4 | 18·6 | about 35 | Light brown |
| As above but 3% solution of sodium hydroxide at 55 lb. pressure | 1·4 | 15·4 | 50 | Fair white |

It is seen that with the more severe conditions the lignin content of the residual material is lower, whilst the loss of weight is higher. On the other hand, a fair white instead of only a brown colour is obtained.

From the various figures recorded above it is seen that the loss of weight to be expected in obtaining pure cellulose from raw jute is of the order 40 to 50 per cent., but the α-cellulose content of the product is high. Values for ash contents are reasonably low and could no doubt be diminished by careful attention to rinsing after the steep in dilute acid that is customary in technical practice after bleaching cellulosic fibres.

The fluidity values for the material boiled after an acid hypochlorite treatment show that the processes employed did not result in undue attack of the cellulose, and if the values are converted to viscosities in centipoises of 1 per cent. solution direct comparison may be made with values recorded by Hebbs[13] for wood pulps suitable for the manufacture of viscose rayon, thus :—

| | Cuprammonium Viscosities (1% Soln.) | | | | |
|---|---|---|---|---|---|
| Jute Cellulose from Tossa fibre | 24 | 37·2 | 42·7 | 55·0 | 58·9 | 77·6 |
| | Low Viscosity | | Medium Viscosity | | High Viscosity | |
| Wood Pulp from Mill A ... ... | 17·5 | | 21·3 | | 27·6 | |
| ,, ,, ,, ,, C ... ... | 10·1 | | 12·0 | | 14·9 | |

The viscosities of the cellulose products isolated from jute are higher than those for the commercial wood pulps, and judging by this property and the high alpha-cellulose and relatively low ash contents there should be no great difficulty in obtaining jute cellulose suitable for viscose rayon, white papers, and perhaps some kinds of cellulose esters or ethers (see also [18]).

Methods similar to those mentioned above may be used for the isolation of cellulose from other lignified fibres. For example Sisal yields a white material of good quality if the original fibre is boiled for five hours at 55 lb. excess pressure with a 3 per cent. solution of sodium hydroxide at 8 : 1 liquor ratio and is then bleached with alkaline hypochlorite of 5 g. of Av.Cl/l for about two hours. Coir however, in view of its very high lignin content, requires in addition to these treatments a second boil in 1 per cent. sodium hydroxide solution and a final bleach in a more dilute (3g. of Av.Cl/l) alkaline hypochlorite solution.

## FURTHER EXPERIMENTAL DETAILS

### Material

Unless otherwise stated, the materials employed in the experimental work described were 8 lb. warp yarns of fairly dark colour. They were chosen as being of medium bleaching quality and representative of average rather than very good grades.

**Preparation of Solutions**

For maintaining hypochlorite solutions substantially at the neutral point *p*H 7, sodium bicarbonate was added in the proportion 5 to 10 g/l, depending on the concentration of available chlorine in the liquor. For acid solutions the usual acetate buffers were employed or for *p*H values of approximately 4·1 aluminium sulphate was added to dilute hypochlorite in the proportion about 8 g/l[13].

Except for the conditions of varying alkalinity referred to in Table VIII, the peroxide solutions were always approximately 0·20N in excess alkali and (unless otherwise stated) of a peroxide concentration equivalent to about 2-3 g. of sodium peroxide per litre. For these conditions the required amount of sodium peroxide is dissolved in water containing the chemically equivalent proportion of sodium bicarbonate. (39 $Na_2O_2$ = 84 $NaHCO_3$, i.e. $NaHCO_3/Na_2O_2 = 2{\cdot}2$ approximately), and 8·5 g. of sodium silicate (I.C.I. grade J.81) per litre is added. Neutralisation of the sodium peroxide to the carbonate stage in this way enables advantage to be taken of its alkalinity. In some cases hydrogen peroxide was employed with the necessary higher concentration of added sodium carbonate.

Solutions for the oxidising steep were prepared similarly from sodium peroxide, bicarbonate and silicate, or with hydrogen peroxide, and differed from the corresponding bleach liquors only in their concentration of peroxide (1g. of $Na_2O_2$ instead of about 2·9 g/l).

In order to increase the stability of the peroxide solutions, magnesium sulphate in the proportion 0·25 g/l was usually added.

Solutions of chlorine peroxide were prepared by the method described by Doree[14] using potassium chlorate, oxalic acid and sulphuric acid, whilst chlorite solutions were subsequently obtained from them in known manner.

**Testing**

*Lignin Content*

For the determination of lignin content the sulphuric acid method was employed essentially as described by Shorger[15]. The cut-up material was first extracted with an alcohol-benzene solution and 2 g. samples were treated with 72 per cent. sulphuric acid for 18 hours to isolate the lignin. Additional preliminary extractions first with hot water and then with 1 per cent. hydrochloric acid as recommended by Phillips and Goss[16] were not given. Since insufficient information is so far available about the constitution of lignin itself and the effects upon this substance of the various methods of isolation and purification that have hitherto been proposed, all methods of determination must be considered to be to some extent arbitrary and liable to error. The method now used, however, is analytically convenient and at least allows for progressive removal of lignin to be followed in such a way as to show how the content is affected by the various treatments given.

If the bulky flocculent precipitate of lignin obtained after the extraction of the jute material with sulphuric acid in this method of estimation is treated with dilute (N/25) sodium hypochlorite solution the lignin is readily attacked and dissolved, and it is usually found that a small residue of white fibrous cellulose remains. With both unbleached and moderately well bleached jute the amount is approximately 0·5 per cent. on the weight of the original untreated yarn.

*Loss of Weight*

In assessing loss of weight, the samples were carefully conditioned before and after the chemical treatment in an atmosphere of 64-65 per cent. R.H., and at 18-19°C., and were then weighed on a sensitive balance. Check weighings after further conditioning were made when necessary to ensure that equilibrium had been reached.

*Assessment of Chemical Damage*

In assessing damage to the jute yarns resulting from the chemical treatments given, reliance has been placed on determinations of tensile strength.

Chemical methods such as the fluidity in cuprammonium, solubility number or copper number, give no simple relations for jute that enable the degree of chemical degradation to be determined because these tests apply substantially to cellulose or modified cellulose. Even well bleached jute, however, contains, besides its cellulose, a considerable proportion of hemicellulose and lignin, and these impurities interfere in the tests or in their interpretation. If all the non-cellulose impurities are carefully removed, the methods may be employed in the usual way for assessing the damage to the cellulose itself, but where the exact proportion of cellulose in any sample of jute taken for examination is unknown, separate determinations of this proportion are necessary before the test results can be interpreted in the light of their known behaviour with cellulose itself.

Boiling with caustic soda solution, which is recommended[6] before determination on cotton and other cellulose materials, does not enable satisfactory fluidity results to be obtained with jute because (as shown in Table I) it fails to remove the lignin, and not only does this lignin "dilute" the cellulose to an unknown extent but it remains insoluble in the cuprammonium liquid and may interfere with the normal determinations of viscosity. The lignin and other non-cellulose impurities already possess reducing properties so that in the copper number determination a high value may be due to residual non-cellulose impurities or to chemical attack of the cellulose itself or to both, and the effect on the cellulose cannot be accurately assessed until that due to the impurities is known or has been avoided.

In attempts to determine whether bleaching treatments cause undue attack of the cellulose, the following additional experiments were made.

Samples of unbleached and bleached jute yarn, previously cut up into short lengths, were treated at 50 : 1 liquor ratio for 30 minutes in a hypochlorite solution of 5 g. Av.Cl/1 at *p*H 4·5, filtered, washed and antichlored. They were then boiled under reflux with 2 per cent. sodium hydroxide solution, acidified and washed. The resulting white fibre was dried to constant weight in a desiccator over sulphuric acid and used for fluidity measurements on 0·5 per cent. solutions in B.C.I.R.A. standard cuprammonium. The following results were obtained :

| Exp. No. | Sample of Cellulose from | % Loss of wt. | Mean Fluidity |
|---|---|---|---|
| 1. | Untreated Jute ... ... ... ... ... | 43 | 11·8 |
| 2. | Jute yarn bleached by the sequence oxidising steep, 3g Av.Cl/l alkaline hypochlorite, peroxide | 37 | 12·3 |
| 3. | As 2, but with 2g. Av.Cl/l neutral instead of alkaline hypochlorite | 42 | 12·8 |

From these values it is evident that the bleaching treatments given, which correspond to those already described in an earlier section, have only a slight effect in increasing the fluidity, and damage to the jute cellulose itself resulting from such bleaching is, therefore, small. Hence in bleaching processes the losses of strength that are encountered appear to be due much more to attack of the non-cellulose constituents of the jute than to attack of the cellulose, and the incrustants can be regarded as having a protective action towards the cellulose whilst they themselves are degraded.

From preliminary experiments it seems likely that more satisfactory separation of cellulose from the other constituents of jute can be made by attacking the lignin with a solution of chlorine peroxide and then removing the modified lignin and the hemicellulose by sulphite and alkaline treatments. By this method less chemical damage to the cellulose occurs than when acid hypochlorite is employed. After such treatment the cuprammonium fluidity of jute cellulose has been estimated to be about 5 to 6 units but further work on the method is necessary before reliance can be placed on such results.

*Breaking Loads*

Strength tests were made on a standard Goodbrand Single Thread Tester after conditioning the yarns at 65 per cent. R.H. and 18 to 19°C. The length of the specimens between grips was not less than 10 inches. In some cases the cut-skein method[17] was used for preparation of the specimens actually employed in testing, the skeins being taken from hanks chosen at random from the main batch of yarns. One portion, or more, of the cut skeins was reserved as original untreated yarn, the remaining portions were submitted to the chemical treatment, and all sets were afterwards tested. For the main bulk of yarn used, grand means for the dry and wet strengths of the original unprocessed yarn were obtained from the results of hundreds of breaks.

In determining the dry strength of the original yarns, the test specimens were first wetted thoroughly with water to remove latent strain and to render the yarns more strictly comparable with the corresponding chemically treated ones, then dried and conditioned. Wet strengths were determined in the usual way by wetting the threads completely in water and keeping them wet during the testing.

Owing to the high variability of jute yarns, which would necessitate an inordinate amount of testing if mean values of great accuracy were to be obtained, no precise significance attaches to small differences between values in the Tables, and the recording of standard deviations has been considered unnecessary since only general and obvious deductions have been made regarding the effects on strength of the various treatments given.

As an example, however, standard deviations have been calculated for the yarns from one experiment in which, of two test bundles obtained by the cut skein method from one and the same re-wound hank of unbleached jute yarn, one bundle was wetted and redried before testing whilst the second remained untreated. The results were as follows :—

| | Untreated | Wetted and redried |
|---|---|---|
| Mean of 100 breaks ... ... | 7·70 lb. | 7·88 lb. |
| Standard Deviation ... ... | 1·07 ,, | 1·13 ,, |
| Standard Error ... ... ... | 0·107 ,, | 0·113 ,, |

Taking differences greater than twice the standard error of the means as being statistically significant, it is found that the difference between the mean breaking loads is in this case not significant.

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