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An Eco-Friendly Approach Towards Bleaching Process for Whitening of *Kydia calycina* Fibres Instead of Hydrogen Peroxide

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ABSTRACT

This study was conducted to sustain the eco-friendly approach for bleaching of *Kydia calycina* fibres. The *Kydia calycina* fibres were extracted from the young shoots of *Kydia calycina* plant through water retting and then the extracted fibres were scoured with pectinase enzyme. After that, the scoured *Kydia calycina* fibres were bleached with three different methods. Among them, the best bleaching method was selected on the basis of tenacity, elongation, fineness, fibre length, weight loss and whiteness index. The concentration of sodium perborate, concentration of TAED, time and temperature of selected bleaching method were optimised using SAS software. At optimisation stage, tenacity, elongation, fineness and whiteness index of bleached fibres were tested. The results indicated that TAED activated sodium perborate showed better tenacity (3.81 g/d), elongation (2.09 %), fineness (22.92 denier) and maximum whiteness index (68.67) at optimised variables including concentration of sodium hydroxide (5 g/l), concentration of TAED (3 g/l), time (60 min) and temperature (50^{0} C).

Keywords: Bleaching, physical and mechanical properties,
Kydia calycina fibres, TAED, Sodium perborate, eco-friendly bleaching.

INTRODUCTION

The textile wet processing during the entire production chain possesses serious problems related to environmental aspects such as air, water, land and human body. These problems create due to utilization of harmful and large number of chemicals[3]. Pollutants in textile effluents include suspended solids, mineral oils and other organic compounds which are release from different parts of the textile manufacturing processes scouring, desizing, bleaching, mercerizing, dyeing, printing, and other specific treatments[6]. Hence, it can be say that the natural fibre production unit is also one of the most polluting unit because lot of pollutants are generated from the extraction of natural plant fibres to processing of the fibres. This type of pollutants contribute more problems related to the health of human and environment.

In the processing of *Kydia calycina* fibres, scouring and bleaching are main contributors for pollution. So, the eco-friendly processes are needed for scouring and bleaching of the extracted *Kydia calycina* fibres and make it as eco-friendly fibres. Prior to the bleaching, scouring should be used to remove the natural impurities. So that, the bleaching agent may act uniformly on the material free of impediment[7]. The extracted *Kydia calycina* fibres are scoured by pectinase enzyme to remove the pectin content from the fibres. Enzymes are best alternative to the polluting textile processing industries because of non-toxic and environmental friendly nature. Enzyme works under mild conditions (pH, temperature) with low water consumption that results in reduced use of harsh chemicals, time, energy and water savings along with improved product quality[8]. The purpose of bleaching includes the removal of the various natural and added impurities as efficiently as possible with minimum or no damage to the fibre and leaving the fabric in a perfectly white state[12]. The effectiveness of sodium perborate as an eco-friendly bleaching agent was assessed by monitoring the whiteness index, percent loss in fabric weight, tensile strength, carboxyl content of treated sample. The result was shown that the sodium perborate (SPB) was a novel bleaching agent which was used alone without any additives in aspect of ecological, economical and energy conserving[14]. Various chemicals were used in addition to the bleaching agent. These chemicals serve different functions such as to activate the bleaching system, to stabilize or control the rate of activation, to give wetting and detergent action, or to sequester metallic impurities[13]. The non-silicate stabilizer was used in place of silicate without any adverse effect on the uniformity and levelness of dyeing. Bleaching of cotton using TAED activated sodium perborate was good replacement of conventional bleaching process in regard to environment friendly, energy saving and low temperature process. Improved whiteness with minimum fibre damage was obtained by activated tetraacetyl ethylene diamine (TAED) sodium perborate bleaching at low temperature and shorter exposure duration. The process was found to reduce energy cost and effluent load on environment. TAED is colourless, odourless, storage stable, safe and easy to handle. It was established as a non-toxic, non-sensitizing and non-mutagenic product which readily biodegrades to form carbon dioxide, water, ammonia and nitrate[2].

Therefore, research work has been focused on the development and application of eco-friendly approach for bleaching of *Kydia calycina* fibres that enhance the whiteness at low energy consumption. The objective of the study was to compare the various bleaching methods and to select the best one for more whiteness of *Kydia calycina* fibres along with better physical and mechanical properties.

MATERIAL AND METHODS

The young shoots of *Kydia calycina* plant were collected from the Agro-forestry Research Center, Govind Ballabh Pant University of Agriculture and Technology, Pantnagar. The various chemicals and enzyme such as pectinase enzyme, hydrogen peroxide, non-ionic wetting agent (Lissapol D), sodium silicate, stabilizer NS, sodium hydroxide, sodium carbonate, sodium perborate and TAED were used in present study.

Extraction and scouring of fibres

Kydia calycina fibres were extracted from young shoots of plant through water retting and were scoured with pectinase. The scoured *Kydia calycina* fibres had maximum tenacity and elongation and fineness than extracted *Kydia calycina* fibres. The result of statistical analysis between extracted and scoured *Kydia calycina* fibres showed non-significant difference at five percent level of significant in the tenacity and elongation whereas, significant difference was found in the fineness. But, the physical properties of scoured fibres were found maximum than extracted fibres. Hence, it can be concluded that the scoured *Kydia calycina* fibres were better than extracted fibres[4].

Bleaching of fibres

Three different bleaching methods were used to bleach the scoured *Kydia calycina* fibres. The best bleaching method was selected on the basis of their tenacity, elongation, fineness, fibre length, weight loss and whiteness index.

In method-I, the bleaching solution was prepared by using hydrogen peroxide bleaching agent (2.5 ml/l), stabilizer NS (1.7 g/l) and non-ionic wetting agent (0.5 g/l). One gram fibre sample was taken in a beaker and bleached with prepared solution at 95^0 C for 60 min. The material liquor ratio was kept as 1:20. Caustic soda (1.5 g/l) was used for maintaining the pH of the bath around 10 to 11. After bleaching, the fibres were washed in cold water and neutralised with acetic acid (2 ml/l). The neutralised fibre samples were washed thoroughly by cold water again and dried in air[2].

In Method-II, one gram scoured fibres were treated with 1.0 % concentration of hydrogen peroxide (35 % v/v) bleaching agent along with wetting agent (0.5g/lit of Lissapol D) and sodium silicate (6.0 g/l) stabilizer. Sodium Carbonate (1.5 g/l) was used to maintain the pH around 10. The temperature of bleaching bath was 80^0C for 60 min. The material liquor ratio was kept 1:20. The bleached fibres were washed successively with cold and hot water respectively and then, dried at room temperature[11].

In Method-III, the solution was prepared by combination of sodium perborate bleaching agent (15 g/l) and bleach activator TAED (Tetra Acetyl Ethylene Diamine). TAED (1g/l) was added into bleach bath for performing the bleaching process at lower temperature and alkalinity. The temperature of bath was maintained at 50^0 C for 60 min. Material liquor ratio was taken as 1:10. One gram scoured fibres were bleached through prepared solution and washed under tap water[10].

Physical and mechanical testing of fibres

The tenacity, elongation, fineness, fibre length and weight loss were determined. The tenacity, elongation and fineness of the *Kydia calycina* fibres were tested using **Fafegraph-M**.

Length of the *Kydia calycina* fibres was analyzed by the single fibre measurement method[1].

The weight loss was determined using following formula[5]:

$$\text{Weight loss (\%)} = \frac{W_1 - W_2}{W_1} \times 100$$

The hunter scale based whiteness index of *Kydia calycina* fibres was measured by using '**Dual Beam Colorscan Spectrophotometer**'. This instrument was based on the principle of reflectance. The whiteness index was calculated by formula given below.

$$\text{Whiteness index} = \frac{L}{100}(L - 5.72b)$$

Values of colour coordinates were computer calculated from reflectance data which reflects:
L^*= lightness/ darkness axis (L^*=0 for black and L^*=100 for white)
a^*= red-green axis (+ a^* for red and -$a^*$for green)
b^* = yellow-blue axis (+ $b^*$for yellow and -b* for blue)

Statistical analysis

Data were collected from the various experiments and statistically analyzed by using SAS software. Mean, standard error and co-efficient of variance were calculated to describe the variables. One way analysis of variance (ANOVA) was used to analyze the significant different in the data of each bleaching variables by using *Duncun post hoc test* at 5 % level of significance. The higher the *p*-value means lower the significance. So, we can say that *p*d" 0.05 means that the difference is significant.

RESULTS AND DISCUSSION

Selection of bleaching method

The tenacity, elongation, fineness, fibre length, weight loss and whiteness index of all bleached samples of *Kydia calycina* fibres were tested and shown in Table 1.

Table 1 indicates that the *Kydia calycina* fibres bleached with Method-III were had maximum tenacity (3.89±0.04 g/d), elongation (2.43±0.02 %), fibre length (6.66±0.20 cm) and whiteness index (62.98±0.04) as compared to other fibres bleached with Method-I and Method-II. However, this fibre sample exhibited minimum weight loss (8.82±0.29 %) and coarser fibres (24.54±0.24 denier). From this table, the *p*-value of one-way analysis of variance showed the significant difference in fineness, weight loss and whiteness index separately and non-significance differences were found in the tenacity, elongation and fibre length of *Kydia calycina* fibres at 5 % level of significance. According to highest tenacity, elongation, fibre length, whiteness index and minimum weight loss, the Method-III was selected for bleaching of *Kydia calycina* fibres. The reason for better properties of fibres may be the efficient removal of colouring matter and other related impurities from the fibres with less reduction of fibre properties i.e. tenacity, elongation, fibre length, weight loss as compared to other methods. Removal of colouring matter is based on lignin content of fibres and lignin is difficult to bleach than other impurities. And, the sodium perborate becomes more effective than hydrogen peroxide for removing lignin along with colouring matter from

Table 1: Properties of *Kydia calycina* fibres bleached with different methods

S. No.	Bleaching methods	Tenacity, g/d (Mean ± S.E.)	Elongation, %(Mean ± S.E.)	Fineness, denier (Mean ± S.E.)	Fibre length, cm (Mean ± S.E.)	Weight loss, % (Mean ± S.E.)	Whiteness index (Mean ± S.E.)
1	Method I	$3.82^{a} \pm 0.04(3.64)$	$2.40^{ab} \pm 0.02(2.34)$	$23.72^{a} \pm 0.36(4.77)$	$6.59^{a} \pm 0.24(25.80)$	$10.02^{a} \pm 0.50(15.84)$	$54.80^{c} \pm 0.03(0.20)$
2	Method II	$3.78^{a} \pm 0.03(2.76)$	$2.38^{b} \pm 0.01(1.28)$	$22.65^{b} \pm 0.39(5.47)$	$6.60^{a} \pm 0.24(25.22)$	$10.38^{a} \pm 0.35(10.68)$	$55.33^{b} \pm 0.04(0.23)$
3	Method III*	$3.89^{a} \pm 0.04(3.34)$	$2.43^{a} \pm 0.02(2.07)$	$24.54^{a} \pm 0.24(3.05)$	$6.66^{a} \pm 0.20(21.02)$	$8.82^{b} \pm 0.29(10.22)$	$62.98^{a} \pm 0.04(0.19)$
Sig (p-value)	0.1422	0.0753	0.0019	0.9738	0.0222	0.0001	

- Value in parentheses indicate CV (%)
- If the p-value less than 0.05 then significant difference present among mean of all groups within the property
- Data followed by same letter within column are not statistically different according to Duncun post hoc test(p>0.05) at 5 % level of significance
- Star (*) denotes selected scouring method

the fibres as per given results. Another finding also supported this result that TAED activated sodium perborate rapidly generates peracetic acid which improves the bleaching efficiency of fibres due to extra alkalinity of prepared solution. As results for this improved efficiency, the better properties were observed and less fibre damage occurred at lower temperatures[9]. The bleaching of cotton materials by sodium perborate is better than that of hydrogen peroxide bleached material. Since, this process is carried out at lower temperature and for shorter exposure duration along with an acceptable degree of whiteness and minimum cellulose degradation[10]. Further it has been found that sodium perborate works as an ecological, economical and energy conserving bleaching agent[14].

Optimisation of selected bleaching variables

After selecting the bleaching method i.e. Method-III, the variables such as concentration of sodium perborate, concentration of TAED, time and temperature were optimised for obtaining more whiteness of *Kydia calycina* fibres with better physical and mechanical properties. Based on reviews, the ranges for each bleaching variables i.e. concentration of sodium perborate, concentration of TAED, time and temperature were taken.

Concentration of sodium perborate

Ten concentrations from 1 to 19 g/l by keeping the difference of 2 g/l were used to optimise the concentration of sodium perborate for bleaching of the *Kydia calycina* fibres. The properties i.e. tenacity, elongation, fineness and whiteness index were tested. The results of the fibres are given in Table 2

It is evident from Table 2 that with increasing concentration of sodium perborate, the tenacity and elongation of *Kydia calycina* fibres were decreased. At this increasing order of concentration, the fineness of fibre sample was also increased whereas the whiteness index was firstly increased and then decreased. Due to maximum whiteness index of the bleached fibres, 5 g/l concentration of sodium perborate was selected.

Concentration of TAED

The scoured *Kydia calycina* fibres were bleached with ten different concentrations of TAED

Table 2: Properties of *Kydia calycina* fibres bleached at different concentrations of sodium perborate

S. No.	Concentrations of sodium perborate, g/l	Tenacity, g/d (Mean ± S.E.)	Elongation, % (Mean ± S.E.)	Fineness, denier (Mean ± S.E.)	Whiteness index (Mean ± S.E.)
1	1	$4.61^{a} \pm 0.04(2.74)$	$3.19^{a} \pm 0.14(14.01)$	$25.00^{a} \pm 0.09(1.15)$	$63.42^{e} \pm 0.10(0.49)$
2	3	$4.46^{ba} \pm 0.10(6.76)$	$3.04^{ab} \pm 0.06(6.60)$	$24.77^{ab} \pm 0.11(1.44)$	$65.67^{d} \pm 0.07(0.36)$
3	5	$4.32^{bc} \pm 0.08(5.84)$	$2.83^{bc} \pm 0.03(3.85)$	$24.24^{bc} \pm 0.17(2.16)$	$69.43^{a} \pm 0.06(0.28)$
4	7	$4.28^{bc} \pm 0.12(9.20)$	$2.70^{dc} \pm 0.11(13.31)$	$23.85^{dc} \pm 0.17(2.27)$	$68.46^{b} \pm 0.09(0.42)$
5	9	$4.15^{dc} \pm 0.12(9.00)$	$2.60^{dce} \pm 0.08(9.35)$	$23.72^{dc} \pm 0.29(3.91)$	$67.47^{c} \pm 0.07(0.33)$
6	11	$4.01^{de} \pm 0.14(11.03)$	$2.52^{de} \pm 0.10(12.08)$	$23.36^{de} \pm 0.22(2.97)$	$65.49^{d} \pm 0.07(0.35)$
7	13	$3.85^{ef} \pm 0.10(7.77)$	$2.43^{fe} \pm 0.14(17.71)$	$22.73^{ef} \pm 0.22(3.11)$	$63.54^{e} \pm 0.09(0.44)$
8	15	$3.78^{ef} \pm 0.03(2.76)$	$2.38^{fe} \pm 0.01(1.28)$	$22.65^{f} \pm 0.39(5.47)$	$62.36^{f} \pm 0.32(1.64)$
9	17	$3.61^{fg} \pm 0.02(1.70$	$2.19^{gf} \pm 0.03(3.93)$	$21.56^{g} \pm 0.22(3.28)$	$60.39^{g} \pm 0.08(0.40)$
10	19	$3.50^{g} \pm 0.01(1.21)$	$2.05^{g} \pm 0.10(15.39)$	$21.10^{g} \pm 0.20(3.06)$	$59.40^{h} \pm 0.08(0.44)$
Sig (*p*-value)		0.0001	0.0001	0.0001	0.0001

- Value in parentheses indicate CV (%)
- If the p-value less than 0.05 then significant difference present among mean of all groups within the property
- Data followed by same letter within column are not statistically different according to Duncun post hoc test($p>0.05$) at 5 % level of significance
- Star (*) denotes selected range of levels

Table 3: Properties of *Kydia calycina* fibres bleached at different concentrations of TAED

S. No.	Concentrations of TAED , g/l	Tenacity, g/d (Mean ± S.E.)	Elongation, % (Mean ± S.E.)	Fineness, denier (Mean ± S.E.)	Whiteness index (Mean ± S.E.)
1	1	3.86^{a} ± 0.06(4.75)	2.37^{a} ± 0.10(13.68)	22.04^{a} ± 0.74(10.61)	62.36^{j} ± 0.32(1.64)
2	2	3.64^{b} ± 0.03(2.12)	2.05^{b} ± 0.04(6.18)	20.80^{b} ± 0.27(4.04)	63.41^{i} ± 0.10(0.48)
3	3	3.60^{bc} ± 0.03(2.24)	2.04^{b} ± 0.04(5.35)	20.10^{cb} ± 0.30(4.64)	79.40^{a} ± 0.08(0.33)
4	4	3.60^{bc} ± 0.03(2.20)	1.96^{cb} ± 0.10(16.57)	19.82^{c} ± 0.32(5.12)	77.47^{b} ± 0.07(0.29)
5	5	3.53^{cbd} ± 0.06(5.43)	1.89^{cbd} ± 0.07(11.03)	19.40^{cd} ± 0.22(3.57)	75.49^{c} ± 0.07(0.31)
6	6	3.49^{cd} ± 0.02(1.55)	1.82^{cd} ± 0.04(7.40)	19.23^{cd} ± 0.20(3.28)	73.54^{d} ± 0.09(0.38)
7	7	3.41^{ed} ± 0.10(8.99)	1.75^{ed} ± 0.03(5.86)	18.78^{d} ± 0.11(1.80)	70.39^{e} ± 0.08(0.34)
8	8	3.41^{ed} ± 0.02(1.89)	1.64^{fe} ± 0.02(4.58)	17.71^{e} ± 0.16(2.77)	69.44^{f} ± 0.06(0.28)
9	9	3.31^{fe} ± 0.03(2.51)	1.62^{fe} ± 0.03(5.06)	17.04^{fe} ± 0.10(1.87)	68.46^{g} ± 0.09(0.42)
10	10	3.20^{f} ± 0.01(1.26)	1.50^{f} ± 0.03(6.34)	16.54^{f} ± 0.20(3.77)	65.67^{h} ± 0.07(0.36)
Sig (*p*-value)		0.0001	0.0001	0.0001	0.0001

• Value in parentheses indicate CV (%)

•If the p-value less than 0.05 then significant difference present among mean of all groups within the property

•Data followed by same letter within column are not statistically different according to Duncun post hoc test(p>0.05) at 5 % level of significance

•`Star (*) denotes selected range of levels i.e. 1%, 2%, 3%, 4%, 5%, 6%, 7%, 8%, 9% and 10%. In these experiments, the selected concentration of sodium perborate i.e. 5 g/l was kept. The properties including tenacity, elongation, fineness and whiteness index were tested and the data of fibres are given in Table 3.

It can be observed from Table 3 that the tenacity and elongation of *Kydia calycina* fibres were decreased with increasing concentrations of TAED while the fineness was increased. The whiteness index of all bleached fibre samples was increasing up to 3 g/l concentration and then it was decreased. Due to maximum whiteness index of bleached fibres, 3 g/l concentration of TAED was selected for further experiments.

Bleaching time

The scoured *Kydia calycina* fibres were bleached for five different times i.e. 20, 40, 60, 80 and 100 min separately. The physical properties including tenacity, elongation, fineness and whiteness index were tested. The results of fibres are reported in Table 4

It is clearly indicated from Table 4 that tenacity and elongation of *Kydia calycina* fibres were decreased with increasing time period whereas the fineness was increased. The whiteness index of all bleached fibre samples was increased up to 60 min and then it was decreased. The treatment time for bleaching of fibres i.e. 60 min were selected as optimum time for bleaching because of higher whiteness index.

Bleaching temperature

Five different bleaching temperatures such as 40^0, 50^0, 60^0, 70^0 and 80^0 C were used to optimise the bleaching temperature for *Kydia calycina* fibres. The tenacity, elongation, fineness and whiteness index of bleached fibres were tested and results are shown in Table 5

As can be observed from Table 5, the tenacity and elongation of *Kydia calycina* fibres were decreased with increasing the temperature of bleaching whereas the fineness and whiteness index of fibres was firstly increased up to 70^0 C afterwards it was decreased. It was also observed that the non-significant differences were found among each property of all bleached fibres. So, the temperature for bleaching of *Kydia calycina* fibres i.e. 50^0 C was taken as optimum temperature.

CONCLUSION

As compared to other two methods i.e. Method-I (hydrogen peroxide + NS stabilizer) and Method-II (hydrogen peroxide + sodium silicate stabilizer), TAED activated sodium perborate i.e. Method-III was selected on the basis of highest tenacity, elongation, fibre length, whiteness index

Table 4: Properties of *Kydia calycina* fibres bleached at different times

S. No.	Bleaching times, min	Tenacity, g/d (Mean ± S.E.)	Elongation, % (Mean ± S.E.)	Fineness, denier (Mean ± S.E.)	Whiteness index (Mean ± S.E.)
1	20	4.30^a ± 0.08(6.03)	3.59^a ± 0.02(1.76)	24.79^a ± 0.30(3.78)	60.51^e ± 0.09(0.48)
2	40	4.20^a ± 0.08(6.36)	3.25^b ± 0.08(7.57)	23.54^b ± 0.19(2.52)	69.57^b ± 0.09(0.43)
3	60	3.92^b ± 0.04(3.29)	2.98^c ± 0.05(5.64)	22.60^{cb} ± 0.50(6.98)	70.44^a ± 0.14(0.64)
4	80	3.82^b ± 0.05(4.67)	2.60^d ± 0.04(4.90)	22.32^{cd} ± 0.31(4.32)	68.03^c ± 0.17(0.77)
5	100	3.64^c ± 0.02(1.56)	2.42^e ± 0.07(8.82)	21.38^b ± 0.34(5.10)	64.77^d ± 0.14(0.67)
Sig (*p*-value)		0.0001	0.0001	0.0001	0.0001

•*Value in parentheses indicate CV (%)*

•*If the p-value less than 0.05 then significant difference present among mean of all groups within the property*

•*Data followed by same letter within column are not statistically different according to Duncun post hoc test(p>0.05) at 5 % level of significance*

•*Star (*) denotes selected range of levels*

Table 5: Properties of *Kydia calycina* fibres bleached at different temperatures

S. No.	Bleaching temperatures, ^{0}C	Tenacity, g/d (Mean ± S.E.)	Elongation, % (Mean ± S.E.)	Fineness, denier (Mean ± S.E.)	Whiteness index (Mean ± S.E.)
1	40^{0}	$3.81^{a} \pm 0.15(12.29)$	$2.17^{a} \pm 0.03(4.40)$	$23.15^{a} \pm 0.29(3.94)$	$68.01^{b} \pm 0.47(2.17)$
2	50^{0}	$3.81^{a} \pm 0.02(1.90)$	$2.09^{a} \pm 0.28(41.95)$	$22.92^{a} \pm 0.38(5.29)$	$68.67^{ab} \pm 0.27(1.23)$
3	60^{0}	$3.79^{a} \pm 0.06(5.08)$	$2.08^{a} \pm 0.10(15.31)$	$22.53^{a} \pm 0.52(7.26)$	$69.08^{a} \pm 0.18(0.82)$
4	70^{0}	$3.68^{a} \pm 0.02(1.55)$	$2.05^{a} \pm 0.05(7.81)$	$22.17^{a} \pm 0.58(8.25)$	$69.15^{a} \pm 0.25(1.16)$
5	80^{0}	$3.65^{a} \pm 0.02(1.56)$	$1.97^{a} \pm 0.18(28.25)$	$21.62^{a} \pm 0.63(9.26)$	$68.77^{ab} \pm 0.08(0.37)$
Sig (*p*-value)		0.3788	0.9292	0.2151	0.0462

•Value in parentheses indicate CV (%)
•If the p-value less than 0.05 then significant difference present among mean of all groups within the property
•Data followed by same letter within column are not statistically different according to Duncun post hoc test($p>0.05$) at 5 % level of significance
•Star (*) denotes selected range of levels

and minimum weight loss. This method also becomes more environment friendly than other two methods. So, the optimisation of variables for selected bleaching method was carried out and the results indicated better tenacity (3.81 g/d), elongation (2.09 %), fineness (22.92 denier) and maximum whiteness index (68.67) at the concentration of sodium hydroxide (5 g/l), concentration of TAED (3 g/l), time (60 min) and temperature (50^0 C). Therefore, these bleaching conditions are optimum conditions for final bleaching of *Kydia calycina* fibres.

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