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# Single bath enzymatic scouring and bleaching process for preparation of absorbent cotton

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This study aims at developing an enzyme based single bath scouring and bleaching process for the preparation of absorbent cotton using short staple cotton fibre with high micronaire. Neutral pectinase and cellulase enzymes individually and also in combination for scouring and hydrogen peroxide for bleaching have been used. The enzymatic process has been optimized to get the desired absorbency of less than 10 s as prescribed by pharmacopeia. An attempt has also been made to explore the possibility of combining the enzyme treatment with peroxide bleaching in a single bath. The result indicates that like conventional scouring and bleaching processes the enzymatic process with a mixture of pectinase and cellulase followed by bleaching produces required qualities for the cotton to be used as absorbent cotton. The absorbent cotton thus produced by the above process shows sinking time of 1.8 s, water holding capacity of 24 grams per gram fibre and less than 0.5% sulphated ash. The weight loss for the single bath scouring and bleaching process is found 18% less as compared to conventional process without any significant difference in the whiteness index and absorbency. The carboxyl content of the treated fibres shows that the formation of oxycellulose is lesser in enzymatic process as compared to conventional treatment. The relative crystallinity index obtained from FTIR spectra shows that the change in proportion of crystalline and amorphous regions is lesser in enzyme treated fibres. The developed process is ecofriendly with savings in energy, time and water along with minimum physical and chemical changes in the fibre.

**Keywords**: Absorbent cotton, Bleaching, Cellulase, Enzymatic scouring, Medical textiles, Pectinase, Single bath pretreatment

## 1 Introduction

Absorbent cotton is a medical product used to absorb body fluids. Apart from medical applications, currently absorbent cotton is also used in various non-medical applications.

Absorbent cotton is the basic raw material for the cotton swab used in ear buds. It is also used for hygiene purposes like wiping pads, sanitary pads, etc. The pharmacopeia of different countries has outlined the type of fibres to be used for the preparation of absorbent cotton. The cotton should be virgin fibre or comber noil obtained during processing of various species of cotton. Short staple cotton with micronaire value more than 5 is considered as best suitable fibre for the preparation of absorbent cotton since it favours the water holding capacity; high micronaire value of the fibre increases the water holding capacity[1]. The pharmacopeia prescribes that the absorbent cotton should be carded, bleached and free from any impurities. The quality of absorbent cotton is determined in terms of different parameters like sinking time, water holding capacity, sulphated ash, etc. As per the pharmacopeia, the absorbency/sinking time should be less than 10 s, water holding capacity should not be less than 23g per gram of fibre and sulphated ash should not be more than 0.5% for absorbent cotton[2]. In order to achieve the above standards, raw cotton has to undergo a series of mechanical and chemical processes. The major chemical processes are scouring and bleaching. Scouring is the process of removing both the natural and the added impurities from cotton fibre. This process is generally done in the kier machine with alkali under high temperature (120°C) and pressure (15 psi). The process has to be done under controlled condition, otherwise the fibre will be damaged due to the formation of oxy- cellulose[3]. Bleaching is the process to remove the residual colour of the fibre and to make it white. Previously, absorbent cotton industries used bleach containing chlorine. However, they shifted to peroxide based bleaching process due to environmental regulations.

Recently, there is a trend to develop environment friendly scouring and bleaching processes using ecofriendly chemicals, less energy and water in place of

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conventional one[4] for cotton fabrics. Alkaline pectinase[5,6], cellulase[7] enzymes based processes are developed as an alternative to alkaline scouring for cotton fabrics. Though the above enzymes are active at lower temperature in the range of 60-65°C, they still require alkaline *p*H through the addition of some quantity of sodium hydroxide and the temperature of the bath need to be increased to 90-95°C to remove the residual wax and to deactivate the enzyme. The above requirements of alkali addition and higher temperature result in making the enzymatic scouring process un-attractive as compared to the conventional process. The enzyme based processes have some further limitations like inability to completely remove non cellulosic impurities due to poor access to the substrates as well as substrate specific nature when used in industrial scale, high cost, temperature and *p*H sensitivity, lesser shelf life, etc. In order to overcome the above limitations, the present study aims to use combination of enzymes such as pectinase and cellulase instead of single enzyme at low temperature and at neutral *p*H conditions. The single bath scouring and bleaching process using sodium hydroxide and hydrogen peroxide was attempted for the preparation of absorbent cotton[8,9]. However, single bath scouring and bleaching process using enzyme as a scouring agent is so far not reported. There are some problems for adoption of such process using enzyme because if enzyme and hydrogen peroxide are taken together, the enzyme might be deactivated due to the oxidation property of hydrogen peroxide. Another problem is the difficulty in maintaining *p*H of the bleaching bath. Based on the above, the study also aims to explore the possibility of developing single stage scouring and bleaching for the preparation of absorbent cotton using combination of above mentioned enzymes at neutral *p*H condition.

## 2 Materials and Methods

### 2.1 Raw Material

Short staple cotton fibre of Bengal Desi variety with 20.8 mm staple length and 6.1 micronaire value was used in this study. A neutral pectinase enzyme kindly supplied by M/s DuPont (India)Pvt Ltd, India and a commercially available neutral cellulase enzyme purchased from M/s Egenix Biotech Pvt Ltd, Bangalore were used for scouring purpose. Analytical grade of hydrogen peroxide (30 % w/w), sodium hydroxide (NaOH) and sodium silicate were used for bleaching. A non-ionic wetting agent Ultravon RL supplied by M/s Huntsman International (India) Private Limited was used for the study.

### 2.2 Conventional Scouring

Scouring was done by introducing cotton fibre into an autoclave under 15 lbs/sq.inch (psi) pressure and at 120°C using 3% w/w NaOH and 0.1% w/v nonionic wetting agent for 1 h, maintaining material- to- liquor ratio at 1:15. The sample was then washed with hot water followed by cold water.

### 2.3 Conventional Bleaching

The scoured fibre was subjected to bleaching using hydrogen peroxide (3 g/L ), sodium silicate (1.5 g/L ) and sodium hydroxide (1 g/L), 1:20 material-to-liquor ratio at 95°C for 1 h. After the treatment the cotton samples were taken out and washed thoroughly with water and neutralized with acetic acid.

### 2.4 Enzymatic Scouring

The experiments were carried out with raw cotton in an IR beaker dyeing machine, keeping material- to-liquor ratio at 1:20. Ten gram of fibre was used for the experiment. The *p*H of the bath was adjusted at 6.8-7. The scouring process was carried out for 30 min at 65°C. After that, the temperature of the bath was gradually raised to 80°C and continued for 10 min to deactivate the enzyme. Finally, the scoured fibre was washed thoroughly with water. The concentration of pectinase and cellulase enzyme mixture to be taken was optimized in terms of sinking time of scoured cotton. For this purpose, three combinations of pectinase and cellulase enzymes in the different ratio as presented in Table 1 along with individual enzyme concentration of 0.25%, 0.5%, 0.75% and 1% each were used.

After enzyme treatment, the scoured materials were subjected to conventional bleaching treatment in separate bath (two bath enzyme scouring and bleaching) before testing the sinking time. The minimum concentration of enzyme or combination of enzymes that fulfills the criteria of below 10 s sinking time was taken as optimized one.

Table 1—Details of experiment with different ratio of enzymes

| S.No | Pectinase enzyme, % | Cellulase enzyme, % |
|---|---|---|
| 1 | 0.25 | 0.25 |
| 2 | 0.25 | 0.50 |
| 3 | 0.25 | 0.75 |
| 4 | 0.50 | 0.25 |
| 5 | 0.50 | 0.50 |
| 6 | 0.50 | 0.75 |
| 7 | 0.75 | 0.25 |
| 8 | 0.75 | 0.50 |
| 9 | 0.75 | 0.75 |

**2.5 Single Bath Enzymatic Scouring and Bleaching Process**

In this process, the enzyme scouring was done using the enzyme mixture with optimized concentration of pectinase and cellulase enzymes in a beaker dyeing machine keeping material- to- liquor ratio at 1:20. The *p*H of the bath was adjusted at 6.8- 6.9. The scouring process was done for 30 min at 60-65°C. After that the bath was allowed to cool to 40- 45°C. Then the bleaching chemicals were added in the same bath. The concentrations of hydrogen peroxide, sodium hydroxide and sodium silicate were kept at 3% (owm), 1 g/L and 1.5 g/L respectively. The temperature of the bath was then raised to 95°C and the bleaching was done for 45 min. Finally, the fibre was washed thoroughly and dried.

**2.6 Test Methods**

***2.6.1 Fibre Properties***

The fibre properties like staple length, uniformity ratio, strength and micronaire value (MIC) of control and treated fibres were tested by using Uster High Volume Instrument (HVI)-900 systems.

***2.6.2 Weight Loss***

The weight loss of the samples was calculated by measuring the dry weight of fibre before and after the treatment. The percentage weight loss was calculated using the following equation:

$$\% \text{ weight loss} = [(w_1 - w_2)/w_1] \times 100$$

where $w_1$ is the dry weight before treatment; and $w_2$, the dry weight after treatment.

***2.6.3 Sinking Time and Water Holding Capacity***

A pre-weighed amount of absorbent cotton was taken in the standard dimension basket. Then it was dropped in the water column of standard dimensions. The time taken by the basket containing absorbent cotton to completely sink to the bottom of the water column was measured using stop watch and noted as sinking time. As per the standard, the sinking time should be less than 10 s. The same test was used for measuring water holding capacity. The basket containing absorbent cotton used for the sinking test was taken immediately after the test and held for 30 s for draining excess water. The test specimen was then placed in a previously tared beaker and weighed accurately. The difference between the initial dry weight and final weight of the wet absorbent cotton was noted and water retained per gram of cotton was calculated and reported as water holding capacity. As per the standard the water holding capacity should not be less than 23g.

***2.6.4 Sulphated Ash***

Two gram of absorbent cotton was weighed accurately in a pre-weighed silica crucible. At the same time 1g of cotton sample was weighed for determination of moisture in a muffle furnace. Cotton fibre was then ignited gently till it was thoroughly charred. After that it was moistened with sulphuric acid. The crucible was then heated gently till white fumes no longer evolved. It was then ignited at about 800 °C until all the black particles disappeared. The residue was again moistened with sulphuric acid and reignited. After that the crucible was allowed to cool in the desiccator and accurately weighed. The difference in the initial dry weight and final weight was used to calculate the sulphated ash percentage.

***2.6.5 Whiteness Index***

The treated fibres were opened in the trash analyzer machine before determining the whiteness index. The whiteness index of the fibres was determined by measuring CIE 2000 whiteness index using Premier Spectrascan 5100 reflectance Spectrophotometer as per following equation;

$$\text{Whiteness index}_{CIE} = Y + 800\,(x_n - x) + 1700\,(y_n - y)$$

where $Y$ is the Y tristimulas value of the sample; $x$, $y$ are the x, y chromaticity coordinates of the sample; and $x_n$, $y_n$ are the chromaticity coordinates of the perfect diffuser for the CIE 1964 standard colorimetric observer.

***2.6.6 FTIR Spectra***

The physico-chemical changes occurred in the cotton fibres after the conventional as well as enzymatic treatments were analyzed by Shimadzu IR-Prestige 21 FTIR spectrophotometer in the range of 4000-400 $cm^{-1}$ using DRS (Diffused reflectance spectrophotometer) assembly. To get the reflectance spectra, cotton fibres were powdered in a grinding mill and mixed with KBr (potassium bromide). The spectra were obtained for each sample at a resolution of 2 $cm^{-I}$ with 64 scans. The relative crystallinity index of the samples was determined by measuring ratio of absorbance at 1428 $cm^{-1}$ and at 900 $cm^{-1}$ $(A_{1428}/ A_{900})$[10]. This ratio indicates the amount of crystalline and amorphous regions in the cotton fibre.

#### *2.6.7 Carboxyl Content*

The carboxyl contents of the untreated and treated fibres were determined as per ASTM D1926-00. Briefly, the cotton was treated with 0.0002 M methylene blue solution at *p*H 8 for a period of 18 h. The decrease in methylene blue concentration in the bath was then measured using UV visible spectrophotometer and related to carboxyl content as millimoles of carboxyl group per 100 g of cellulose. The higher carboxyl content implies higher oxycellulose formation due to treatment which provides the information about the damage to the cotton fibre due to scouring and bleaching.

## 3 Results and Discussion

### 3.1 Optimization of Enzymatic Process

The results show that neutral pectinase and neutral cellulase enzyme when applied individually could not produce absorbency required as per pharmacopeia in the cotton fibre in the all four concentrations, namely 0.25%, 0.5%, 0.75% and 1%. The absorbency of such treated cotton fibres in terms of sinking time is found more than 10 s. The results of binary enzyme combination of pectinase and cellulase enzymes show that the 0.5:0.5%, 0.5:0.75%, 0.75:0.5% concentrations fulfill the sinking time requirement of absorbent cotton. From the results, it is inferred that the addition of 0.5% cellulase enzyme to the pectinase enzyme produces required absorbency for the cotton fibre to be used for absorbent purposes. Hence, due to economic reasons, the pectinase and cellulase enzyme of 0.5:0.5% combination is taken as optimized concentration for the preparation of absorbent cotton. The addition of cellulase to pectinase improves the removal of non-cellulosic impurities present in the fibre. The cellulase enzyme tends to absorb on the primary wall of cotton and facilitates the speedy removal of non-cellulosic impurities[11]. Similar results have also been observed in the previous studies on cotton fabric. It has been reported that the combination of cellulase with pectinase produces higher absorbency than individual enzymes when applied on the fabric[12,13]. However, no such process has been reported about the application of combination of pectinase and cellulase for absorbent cotton preparation.

### 3.2 Single Bath Scouring and Bleaching

Based on the excellent results obtained from the neutral *p*H scouring and conventional bleaching using hydrogen peroxide, the possibility of developing single bath scouring and bleaching is explored in this study. To the best of our knowledge no such process has been reported for the preparation of absorbent cotton. The development of such process for absorbent cotton preparation would reduce the process time, energy, water and chemical usage. The enzyme deactivation can be prevented if the hydrogen peroxide and other bleaching aids are added to the bath after the completion of enzyme scouring. The temperature of the bath can be reduced to 40-45°C from 65°C and the bleaching chemicals can be added without any *p*H problem since enzyme scouring is done at neutral *p*H. Based on the above, in this study, a single stage scouring and bleaching process has been developed in which initially the bath is set with pectinase, cellulase and wetting agent at neutral *p*H and the scouring is completed at 65°C. After that the temperature of the bath is reduced followed by addition of bleaching chemicals such as hydrogen peroxide, sodium hydroxide and sodium silicate.

### 3.3 Effect of Treatment on Fibre Properties

The results of fibre properties of raw and treated fibres are shown in Table 2. It is observed that conventional as well as enzymatic scouring has slightly reduced the fibre length and bleaching has further reduced it. There is almost no difference found in fibre length in one bath or two bath enzymatic scouring and bleaching treatments. This reduction might be caused by the shrinkage due to immersion in water during these purification treatments. Micronaire has also been reduced slightly which may be due to

Table 2—Fibre properties of raw and treated fibres

| Treatment | 2.5% Span length, mm | Micronaire | Fibre strength g/tex | Elongation % | Uniformity ratio |
|---|---|---|---|---|---|
| Raw cotton | 20.7 | 6.1 | 16.8 | 4.5 | 50 |
| Conventional scoured cotton | 19.9 | 5.6 | 18.2 | 4.9 | 51 |
| Conventional bleached cotton | 18.8 | 5.8 | 17.9 | 5.4 | 51 |
| Enzyme scoured cotton | 20.3 | 5.8 | 17.3 | 5.0 | 52 |
| Two bath enzyme scoured + bleached cotton | 20.0 | 5.7 | 17.6 | 4.8 | 50 |
| Single bath enzyme scoured + bleached cotton | 19.9 | 5.8 | 17.3 | 5.2 | 52 |

the removal of impurities present on fibre during the scouring process. Strength values have slightly increased after both enzymatic and conventional scouring though the increase is lower for enzymatic scouring. Fibre shrinkage and loss of short fibres during the scouring process may be responsible for this increase. The lower value of strength observed for enzymatic process is contrary to the earlier study[6] which could be due to the slight degrading effect of the cellulase component of the enzyme mixture[14]. There is a slight improvement observed in percentage elongation as well which again may be due to the fibre shrinkage. A slight increase observed in uniformity ratio may have been caused by the loss of some short fibres during processing.

#### 3.4 Effect of Treatments on Performance Properties

Various performance properties of the absorbent cotton prepared by different methods have been presented in Table 3. Results indicate that cotton after subjecting to conventional NaOH scouring itself becomes highly absorbent and is able to meet the sinking time, water holding capacity and sulphated ash criteria for absorbent cotton. Its appearance is however, yellowish which is reflected by its low whiteness index of about 38 due to insufficient removal of colored impurities during the scouring process. These are removed during bleaching and whiteness is improved as seen from the CIE whiteness index of about 62. This bleached cotton is then found suitable for use as absorbent cotton. Enzymatically scoured cotton, on the other hand, is not absorbent and its sulphated ash content is also higher than the prescribed value. It is also less white than the NaOH scoured cotton as its whiteness index is only 32. However, after bleaching, it also becomes highly absorbent and its sinking time almost matches the sinking time of conventionally scoured and bleached cotton with higher water holding capacity. Its whiteness index is also higher than that of the conventionally prepared sample and these values could be achieved with 15% lower weight loss during the process. Weight loss during the single bath enzymatic scouring and bleaching process is still a little lower (18%). Lower weight loss during enzymatic processing may be attributed to the specific nature of the enzyme action. Single bath treated sample is also able to achieve the comparable sinking time and sulphated ash values. Water holding capacity of this sample is little lower but is found well within the prescribed limit and its whiteness index is at par with the conventionally prepared sample. This single bath enzyme scouring and bleaching process is thus suitable for absorbent cotton preparation and is advantageous, as a reduction in the number of baths results in savings of energy, water and time.

#### 3.5 Physico-chemical Changes in Treated Cotton Fibre

Purification treatments can cause physical and chemical damages to the cotton fibre. Extent of this damage is assessed by studying carboxyl content and FTIR spectra of conventionally and enzymatically (single bath) scoured and bleached cotton fibres.

##### *3.5.1 Carboxyl Content*

The carboxyl content in the conventional and enzymatic scoured and bleached cotton fibres is given in Fig. 1. The result indicates that the cotton fibre treated with conventional process has higher carboxyl content than enzymatic process. The carboxyl content

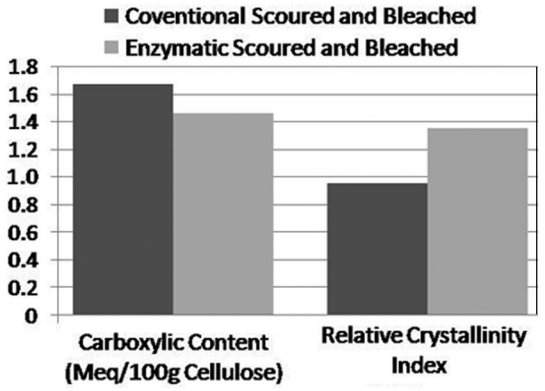


Fig. 1—Carboxylic content and relative crystallinity index of cotton fibres treated with conventional and enzymatic processes

Table 3—Performance properties of treated cotton

| Treatment | Weight loss % | Whiteness index | Sinking time, s | Water holding capacity g of water/g | Sulphated ash % |
|---|---|---|---|---|---|
| Raw Cotton | - | 20.5 | - | - | 1.2 |
| Conventionally scoured cotton | 5.6 | 37.8 | 2.0 | 26.6 | 0.39 |
| Conventionally scoured and bleached cotton | 6 | 61.8 | 1.5 | 26.5 | 0.30 |
| Enzyme scoured cotton | 4.7 | 32.6 | Not sink | - | 0.8 |
| Two bath enzyme scoured and bleached cotton | 5.1 | 65.4 | 1.9 | 27.6 | 0.35 |
| Single bath enzyme scoured and bleached cotton | 4.9 | 61.3 | 1.8 | 24.2 | 0.37 |

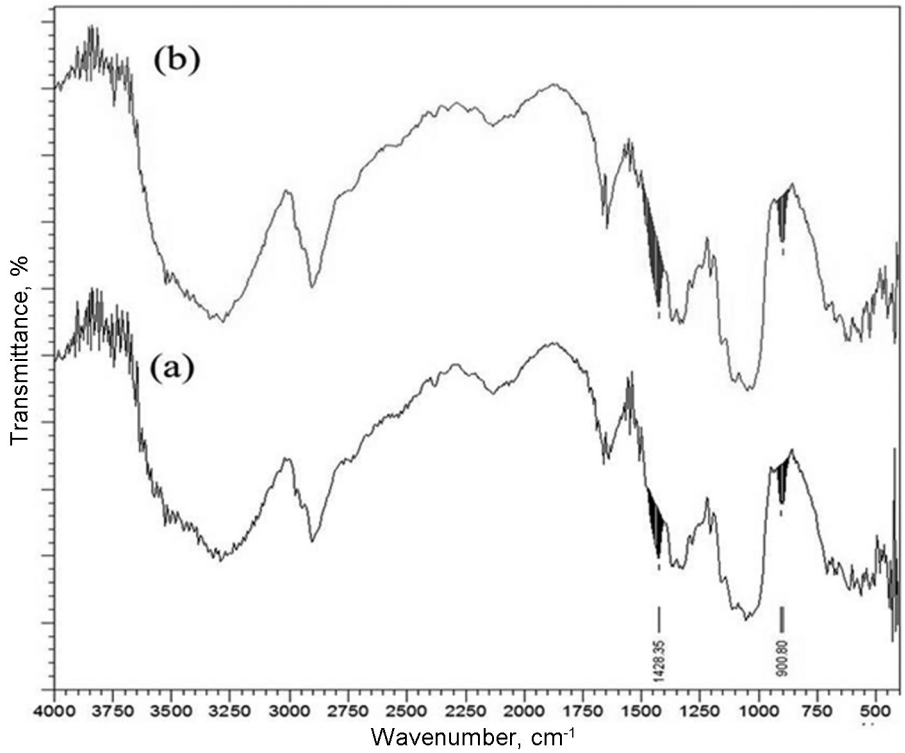


Fig. 2—FTIR spectra of cotton fibres treated with (a) conventional and (b) enzymatic processes

in the cotton increases with the formation of oxy cellulose. During alkaline scouring at high temperature, higher amount of hydroxyl groups (-CHOH) in the cellulose is oxidized into aldehyde (-CHO) and further into carboxyl groups (–COOH) which implies more chemical damage to cotton fibre as compared to enzymatic process. The damage to the cotton is lesser when subjected to enzymatic process due to the specific nature of action of the enzymes.

***3.5.2 FTIR Spectra and Relative Crystallinity Index***

FTIR spectra of conventional and enzyme scoured and bleached cotton fibres are given in Fig. 2. Both the spectra show peaks at 3420 $cm^{-1}$ (- OH), 2892$cm^{-1}$ (–CH stretching), 1420$cm^{-1}$ (-$CH_2$ bending), 900$cm^{-1}$ (Out -phase ring -OH) and 670 $cm^{-1}$ (-OH out of phase bending), corresponding to typical cotton fibre spectra[15]. There is no additional peak or peak shifting between conventional and enzymatic processes. The spectra are used to calculate the relative crystallinity index by measuring ratio of absorbance at 1428 $cm^{-1}$ and at 900 $cm^{-1}$ ($A_{1428}$/ $A_{900}$). The area of the above peaks is shown as filled one in the spectra. The index measures the relative changes in crystalline and amorphous region of the cotton fibre. From the result, it is found that conventional scoured cotton shows less crystallinity index as compared to enzymatic processed cotton. The reduction in the index may be due to the severity of alkali on cellulose chain during scouring and bleaching. The result is in good correlation with the carboxylic content test reported in section **3.5.1** of this paper.

Based on the above results, it is clear that the enzymatic process is gentle on cotton fibre with less chemical damage as compared to conventional scouring and bleaching process. At the same time, it produces required performance on cotton fibre as prescribed by pharmacopeia.

## 4 Conclusion

A single bath scouring and bleaching process has been developed using a mixture of pectinase and cellulase enzyme and hydrogen peroxide. The concentration of enzyme to be taken for absorbent cotton production has been optimized. Addition of cellulase enzyme to pectinase improves the absorbency of cotton to the required level as prescribed by pharmacopeia. The developed single bath process is ecofriendly as compared to conventional process with savings in energy, water and time. Apart from being ecofriendly, the enzymatic process causes less chemical damage to cotton fibre as compared to conventional process.

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