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Enhancing Crease Recovery Finish on Linen Fabric using Citric Acid Treatment

Dr. Bhaarathi Dhurai¹, Dr. Saravanan M², Rajkumar G³, Shree Dharshini B⁴, Tamizhthendral A⁵,

Laksha Briendel X⁶

Professor, Department of Fashion Technology, Kumaraguru College of Technology, Tamil Nadu, India¹

Assistant Professor - II, Department of Textile Technology, Kumaraguru College of Technology, Tamil Nadu, India²

Associate Professor, Department of Fashion Technology, Kumaraguru College of Technology, Tamil Nadu, India³

Final Year B. Tech Textile Technology, Kumaraguru College of Technology, Tamil Nadu, India⁴⁻⁶

ABSTRACT: Linen fabric is highly valued for its strength, breathability, and natural texture, making it a preferred choice in textiles. However, its tendency to wrinkle and shrink needs the application of various finishing techniques to enhance its performance and appearance. Common finishing methods include mechanical, chemical, and eco-friendly treatments aimed at improving properties such as wrinkle resistance, shrinkage control, softness, and durability. While traditional treatments, such as resin-based finishes, provide effective results, concerns about environmental impact have driven the development of sustainable alternatives. This review explores the advancements in linen fabric finishing, analysing their effectiveness, advantages, and limitations

Keywords: Linen fabric, textile finishing, wrinkle resistance, shrinkage control, mechanical finishing, chemical finishing, fabric durability, eco-friendly treatments.

I. INTRODUCTION

Linen, a bast fibre derived from the flax plant (Linum usitatissimum), is valued for its strength, durability, and sustainability. It is primarily cultivated in Russia, Poland, the Czech Republic, Ukraine, Belgium, France, the Netherlands, and China. Known for its high tensile strength, breathability, and moisture-wicking properties, linen is widely used in home textiles, fashion, and industrial applications. Its temperature-regulating ability makes it suitable for various climates.

Despite its advantages, linen has a major drawback—its tendency to wrinkle easily. This is due to its high crystallinity and rigid cellulose structure, which limits elasticity. The formation of wrinkles is caused by the breaking and reforming of hydrogen bonds within the cellulose molecules under mechanical stress. Linen's low resilience further worsens its creasing tendency, as its rigid polymer structure restricts its ability to recover from deformation. Additionally, linen's high moisture absorption (12–20%) facilitates wrinkle formation, especially during washing, as absorbed water disrupts hydrogen bonds, allowing cellulose chains to realign increased positions.

To improve linen's crease recovery, various finishing treatments have been explored. Enzymatic treatments with cellulase modify the fiber surface, enhancing softness with minimal environmental impact. Silicone-based softeners, like polydimethylsiloxane (PDMS), form a protective coating that enhances smoothness and flexibility, reducing creasing. Cross-linking agents, such as citric acid and sodium hypophosphite (SHP), create stable bonds within the cellulose matrix, improving dimensional stability. While traditional cross-linking agents like dimethylol dihydroxy ethylene urea (DMDHEU) are effective, they raise environmental concerns due to formaldehyde release, prompting research into eco-friendly alternatives. Mechanical treatments like calendaring, mercerization, and softening finishes also improve wrinkle resistance. Blending linen with synthetic fibers such as polyester or elastane enhances elasticity while maintaining natural aesthetics. Advances in textile finishing continue to optimize crease recovery, ensuring linen retains its durability and usability in modern applications. Research on simultaneous dyeing and finishing with reactive dyes and citric acid has shown improved colour index and fabric strength. Studies on formaldehyde-free durable press





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finishing using citric acid, 1,2,3,4-butanetetracarboxylic acid (BTCA). The Box-Behnken method has been used to optimize citric acid finishing parameters for linen, furthering eco-friendly fabric enhancements.

II. MATERIALS AND METHODS

2.1: Materials

A Linen fabric, having the specifications plain weave, count yarn (warp x weft), 56 ends/cm, 55 picks/cm and 140 gsm weight, was used for the study. Crosslinking agent citric acid, catalyst sodium hypophosphite was used to impact crease resistant finish to the fabric.

2.2. Methods:

To optimize the process parameters for enhancing the crease recovery of linen fabric, an experimental plan was designed using the Box-Behnken method. This statistical approach was employed to systematically analyse the effects of multiple factors on the treatment outcome. In this study, three independent variables were selected at three levels (Table 1). The linen fabric was treated in a solution containing citric acid, used based on the liquor weight, and sodium hypophosphite, applied on an owf (on weight of fabric) basis, with a material-to liquor ratio of 1:10. During the padding0 process, the wet pick-up was maintained at 100% owf to ensure uniform chemical application. After padding, the fabric was dried at 80°C, followed by a curing process conducted at three different temperatures—130°C, 140°C, and 150°C—for 2 minutes. Post-curing, the fabric underwent a washing treatment using a soap solution for 2 minutes, followed by rinsing for 10 minutes at room temperature and subsequent drying. Based on the Box-Behnken design, a total of fifteen experimental trials were conducted to evaluate the influence of process parameters on the fabric's crease recovery properties

Table 1-Details of process parameters and different levels for Crease Recovery finish.

Process parameters	Different levels				
	-1 0 1				
Citric acid conc.(X1), %	15 30 45				
(Liquor weight)					
Sodium Hypophosphite conc.(X2), 4 5 6					
%(owf)					
Curing temperature. (X3), °C	130 140 150				

2.3Test methods

2.3.1. Crease recovery angle:

Evaluation was performed using MAG Crease Recovery Tester, AATCC 66. For crease recover test, 40 mm \times 15 mm rectangular specimens were cut and folded for 5 min under 500gms load according to AATCC 66 test standard method. The creasing load was then released and the specimen allowed to recover for a further five minutes in the crease recovery tester and the crease recovery angle recorded.

2.3.2. Tensile strength tester:

Tensile strength was determined using a Universal Testing Machine (UTM) in accordance with ASTM D-5035. Test specimens measuring $30 \text{cm} \times 5 \text{cm}$ were cut and clamped in a tensile testing machine and stretched until it breaks. The test results are recorded.

2.3.3. Tear strength tester:

The tearing strength of the fabric samples was determined by the Elmendorf tearing tester in accordance with ASTM Test Method D 1424-96. A template was used to cut fabric strips of 100 ± 2 mm length and 63 ± 0.15 mm width. The tearing strength was then calculated by the following formula: Tearing strength (g) = Scale reading × 64.

2.3.4. Air permeability:

Air permeability is measured using the Shirley Air Permeability Tester following ASTM D-737-99 The fabric sample is placed over the test head and secured with the clamping mechanism. The test pressure is applied, and the instrument is





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allowed to stabilize. The air permeability is measured, which is the rate of airflow through the fabric, typically in $cm^3/cm^2/s$ or $ft^3/ft^2/min$ (CFM). Air permeability is expressed as:

Where:

Q = Air permeability (cm³/cm²/s or ft³/ft²/min)

V = Measured airflow rate (cm³/s or ft³/min)

A = Test area (cm² or ft²)

Table 2- physical properties of untreated and treated samples using different parameter

Sample code		Process parameter			properties	
	Conc of citric acid %	Conc of sodium hypophosphite %	Curing temperature ⁰ C	Tensile strength kg	CRA	Add-on %
Untreated(U1)	-	-	-	169.1920	60	-
Treated						
T1	15	4	140	135.4161	67	2.9
T2	15	6	140	161.0524	77	1.22
Т3	45	4	140	167.4887	60	1.5
T4	45	6	140	156.6633	66	4.5
Т5	15	5	130	104.5405	77	8.8
T6	15	5	150	124.9873	77	9.04
Τ7	45	5	130	158.1333	66	2.83
Т8	45	5	150	115.3909	72	6.54
Т9	30	4	130	85.7511	78	4.59
T10	30	4	150	60.2082	75	7.78
T11	30	6	130	89.8889	78	5.45
T12	30	6	150	175.9577	70	3.09
T13	30	5	140	125.8000	77	2.22
T14	30	5	140	111.6724	77	2.14
T15	30	5	140	73.1947	81	10.8

2.3.5. Whiteness index

The whiteness index (WI) of fabric was assessed using a DataColor spectrophotometer, following ASTM E313-67, which quantifies the degree of whiteness based on spectral reflectance measurements. Fabric samples were conditioned at 21°C and 65% relative humidity before testing to ensure accuracy. The spectrophotometer was calibrated using a white reference standard and set to illuminant D65 with a 10° standard observer. The WI was calculated using the formula WI = Y + 800(X - Z), where Y represents luminance, and X, Z are tristimulus values. Multiple readings were taken to ensure uniformity, with higher WI values indicating brighter, whiter fabrics, while lower values suggested yellowing or dullness. Factors such as fabric composition, dyeing, bleaching, and the presence of optical brighteners influenced the results. The whiteness index of the fabric was evaluated using a spectrophotometer (Data Colour) according to ASTM DE 313-67.

2.4. Statistical Analysis:

A Box-Behnken Design is a type of response surface methodology (RSM) used for optimizing processes, fitting a quadratic model with three levels per factor, and avoiding extreme combinations of factors, focusing on the midpoints of edges and centre points of the design space.





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A quadratic polynomial was used to analyse the relationship of each response with the three independent variables as shown below:

 $Y=b_0+\sum_{i=1}^3 bixi+\sum_{i=1}^3 biixi^2+\sum_{i=1}^3 bijxixj$

Where b_0 , b_i , b_{ii} , and b_{ij} are the coefficients of the regression equations; I and j are the integers; and Y the response of the dependent variable.

III. RESULTS AND DISCUSSION

3.1 Physical Properties

Table 2 shows the various physical properties of linen fabric treated with citric acid as crosslinking agent in the presence of sodium hypophosphite as catalyst at different curing temperatures. The regression equations for the various properties are given in Table 3. It is found that the Conc of Citric acid(X1), Conc of SHP(X2), Curing Temperature (X3), tensile strength (Y1), crease recovery angle (CRA) (Y2) dependent on second order polynomial equation. The properties show only linear and interaction or combined effects on the process variables.Based on the best fit regression equations, the values of the physical properties possible combinations are calculated (Table 4). For optimization, the properties such as crease recovery angle, tensile strength are given in the table.

Table 3 – Regression equation for various properties

property	Regression equation	\mathbb{R}^2	Adj R ²
Crease Recovery	$\begin{array}{l}Y = & 136.58 \ + \ 0.394 \ X_1 \ + 63.79 \ X_2 \ - 3.12 \ X_3 \ - 0.029 \ X_1^2 \ - 4.29 \ X_2^2 \\ & + 0.01 X_3^2 \ - 0.0667 X_1 X_2 \ - 0.125 X_2 X_3 \ + 0.01 X_1 X_3 \end{array}$	0.7993	0.4382
angle (deg) Tensile strength (kg/sq.cm)	$\begin{array}{l} Y = -1077 + 8.46 X_1 \ \text{-}499.51 \ X_2 + 31.68. X_3 + 0.165 \ X_1{}^2 + 14.39. X_2{}^2 - \\ 0.149 X_3{}^2 - 0.607 X_1 X_2 + 2.79 X_2 X_3 - 0.105 X_1 X_3 \end{array}$	0.7938	0.4225

Table 4 - Calculated values of response for various process parameters

Number	Conc of CA	Conc of SHP	Curing Temperature	CRA	Tensile strength	Desirability
1	37.311	6.000	150.000	71.368	153.533	0.798
2	37.199	6.000	150.000	71.443	153.384	0.798
3	37.446	6.000	150.000	71.276	153.720	0.798
4	37.605	6.000	150.000	71.168	153.944	0.798
5	37.728	6.000	150.000	71.082	154.127	0.798
6	37.094	6.000	149.974	71.510	153.259	0.798
7	37.961	6.000	150.000	70.917	154.483	0.798
8	38.130	6.000	150.000	70.796	154.751	0.798
9	37.599	5.993	150.000	71.238	153.427	0.798
10	36.894	5.993	150.000	71.704	152.534	0.798
11	37.404	5.991	150.000	71.381	153.074	0.798
12	38.326	6.000	150.000	70.653	155.078	0.798
13	37.268	5.991	150.000	71.481	152.823	0.798
14	37.518	6.000	149.898	71.214	153.872	0.798
15	36.715	6.000	149.910	71.749	152.824	0.797

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16	37.104	6.000	149.830	71.486	153.337	0.797		
17	37.561	5.972	150.000	71.449	151.934	0.796		
18	39.002	5.982	150.000	70.303	155.088	0.796		
19	34.633	6.000	150.000	72.968	151.093	0.795		
20	34.127	6.000	150.000	73.223	150.897	0.793		
21	38.239	5.930	150.000	71.325	150.215	0.793		
22	33.785	6.000	150.000	73.387	150.813	0.792		
23	38.495	5.906	150.000	71.338	149.071	0.791		
24	39.299	5.915	150.000	70.653	151.205	0.791		
25	37.355	5.897	149.999	72.218	146.603	0.790		
26	37.572	5.798	150.000	72.827	140.546	0.779		
27	39.371	5.768	150.000	71.729	142.075	0.777		
28	42.268	5.865	150.000	68.483	155.641	0.776		
29	42.529	5.928	150.000	67.699	160.446	0.776		
30	39.989	5.575	150.000	72.453	132.286	0.75		

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3.2. Optimization of Process Parameters

No	Conc of CA	Conc of SHP	Curing Temperature	CRA	Tensile strength	Desirability	
1	37.295	6.000	150.000	71.379	153.512	0.798	Selected

The optimization of crease recovery finishing for linen fabric was carried out, considering the effects of citric acid (CA) concentration, sodium hypophosphite (SHP) concentration, and curing temperature on fabric properties. The software-generated optimized conditions were determined to be a CA concentration of 37.295 g/L, SHP conc of 6.000 g/L, and a curing temperature of 150°C. Under these conditions, the predicted crease recovery angle (CRA) was 71.379°, while the tensile strength was maintained at 153.512 N. The desirability value of 0.798 indicates the reliability and effectiveness of the optimization process in achieving a balance between enhanced crease recovery and fabric strength. These findings provide a practical approach to improving the functional performance of linen fabric through an eco-friendly finishing process.

3.3.1 Influence of Citric Acid Concentration and Curing Temperature

Figure 1 shows the influence of citric acid concentration and curing temperature on the crease recovery angle (CRA) of the fabric. At lower concentrations of citric acid and lower curing temperatures, the CRA values are relatively low. As the curing temperature increases, CRA also shows a rising trend, particularly at moderate citric acid concentrations. However, at very high citric acid concentrations, the CRA tends to decrease, suggesting an optimal range for maximum recovery. The maximum CRA value observed is approximately 81, indicating that both factors significantly influence fabric performance, with an evident interaction between citric acid concentration and curing temperature.





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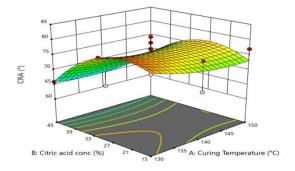


Fig 1- Influence of citric acid and curing temperature

3.3.2. Influence of Citric Acid and Sodium Hypophosphite Concentration

Figure 2 illustrates the influence of citric acid and sodium hypophosphite concentrations on the crease recovery angle (CRA) of the treated fabric. At lower concentrations of both citric acid and sodium hypophosphite, CRA values are relatively low. As the concentrations of both agents increase, the CRA improves significantly, reaching a peak around the mid-range of citric acid and higher levels of sodium hypophosphite. Beyond this optimal zone, CRA shows a slight decline, indicating saturation or overloading effects. The maximum CRA value reaches approximately 81, suggesting a synergistic interaction between citric acid and sodium hypophosphite in enhancing the creas e recovery performance.

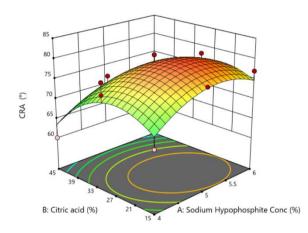


Fig 2- Influence of citric acid and sodium hypophosphite

3.3.3. Influence of Curing Temperature and Sodium Hypophosphite Concentration

Figure 3 represents the influence of curing temperature and sodium hypophosphite (SHP) concentration on the crease recovery angle (CRA). The surface plot shows a flat trend, indicating that variations in curing temperature and SHP concentration have minimal interaction effect on the CRA within the studied range. Despite changes in both parameters, the CRA remains relatively constant, centered around 72–74°C. The data points, however, show some deviations above and below the surface, suggesting that while individual experimental runs varied slightly, the overall combined effect of these two factors did not significantly influence the crease recovery angle.





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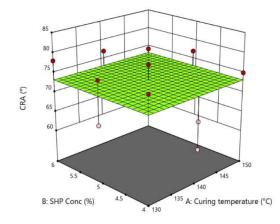
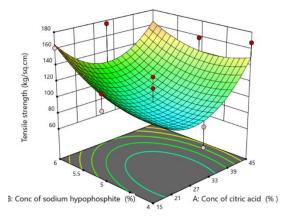
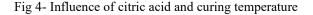


Fig 3- Influence of sodium hypophosphite and curing temperature

3.3.4. Influence of Citric Acid Concentration and Curing Temperature on Tensile Strength

Figure 4 illustrates the influence of citric acid concentration and curing temperature on the tensile strength of the fabric. At lower citric acid concentrations and lower curing temperatures, tensile strength remains relatively high. However, as the curing temperature increases and the citric acid concentration reaches moderate levels, tensile strength significantly drops. Interestingly, at higher citric acid concentrations and higher curing temperatures, the tensile strength recovers and increases again, forming a curved surface with a distinct dip in the center. This suggests that optimal tensile strength can be achieved either at lower chemical intensities or at their higher combinations, while intermediate levels may lead to fiber degradation or incomplete reaction, resulting in reduced tensile performance. The maximum tensile strength observed reaches approximately 176 kg/sq.cm.





3.3.5. Effect of Citric Acid and Sodium Hypophosphite Concentrations on Tensile Strength

Figure 5 presents the interactive effect of citric acid concentration and sodium hypophosphite (SHP) concentration on the tensile strength of the fabric. The surface plot shows a clear U-shaped curvature, indicating that at intermediate concentrations of both citric acid and SHP, the tensile strength is at its lowest. As either the citric acid or SHP concentration increases or decreases from these midpoints, tensile strength improves significantly. The lowest tensile strength dips to around 60 kg/sq.cm, whereas the maximum tensile strength reaches approximately 176 kg/sq.cm. This trend implies that both too little or too much of either chemical can lead to better fiber retention, while moderate levels may cause fiber damage or insufficient cross-linking, weakening the fabric.





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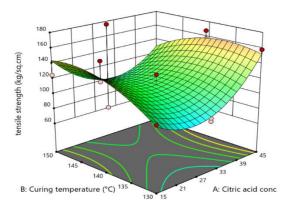


Fig 5- Influence of citric acid and sodium

3.3.6. Effect of Curing Temperature and SHP Concentration on Tensile Strength

Figure 6 illustrates the combined effect of curing temperature and sodium hypophosphite (SHP) concentration on the tensile strength of the treated fabric. The surface shows a consistent rise in tensile strength with an increase in both curing temperature and SHP concentration. The lowest tensile strength (~60 kg/sq.cm) occurs at lower levels of both factors, while the maximum tensile strength (~176 kg/sq.cm) is achieved at the highest curing temperature (150 °C) and SHP concentration (6%). This trend suggests that higher curing temperatures enhance cross-linking efficiency, and higher SHP levels act effectively as a catalyst for citric acid cross-linking, thus contributing to fabric integrity and improved mechanical strength.

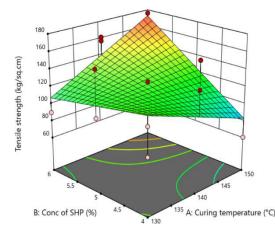


Fig 6- Influence of sodium hypophosphite and curing temperature

3.4.1 Effect of process parameters on Crease Recovery angle

The 3D surface plot illustrates the relationship between the Citric acid (CA) concentration, Sodium hypophosphate (SHP), Crease Recovery Angle (CRA) at a constant Curing temperature of 140°C. The results show that increasing CA concentration enhances CRA due to improved crosslinking of cellulose molecules, while SHP acts as a catalyst, further boosting CRA up to an optimum level. The highest CRA (~81°) is observed at the highest CA (45%) and SHP (6%) concentrations, whereas the lowest CRA (~60°) occurs at lower concentrations (CA=15%, SHP= 4%). The nonlinear nature of the response suggests that while CRA improve with increasing CA and SHP, excessive concentrations may not yield further benefits. The contour plot at the base highlights the rate of CRA improvement, with denser lines indicating





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significant changes. Overall, optimizing CA and SHP concentrations is crucial for maximizing CRA while maintaining fabric integrity.

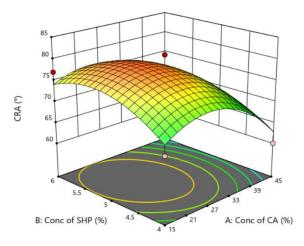


Fig 7- Effect of three process parameters on Crease Recovery angle

3.4.2. Effect of process parameters on Tensile strength

The 3D surface plot represents the relationship between Citric Acid (CA) concentration, Sodium Hypophosphite (SHP) concentration, and the tensile strength of the fabric at a constant curing temperature of 140°C. The graph indicates that at lower concentrations of CA and SHP, the tensile strength is significantly reduced, reaching a minimum of around 60.2 kg/sq.cm. However, as the CA and SHP concentrations increase, the tensile strength improves, with the highest values (~175.96 kg/sq.cm) observed at the highest CA (45%) and SHP (6%) concentrations. The curvature of the plot suggests a nonlinear interaction between the variables, where an optimal balance of CA and SHP is required to achieve the highest tensile strength without excessive degradation. The contour plot at the base visually represents the tensile strength distribution, with the denser regions indicating steeper variations in strength. This analysis highlights that while crosslinking agents enhance tensile properties, excessive concentrations could lead to Fiber embrittlement, affecting overall fabric performance.

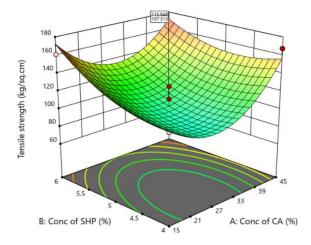


Fig 8- Effect of three process parameters on tensile strength





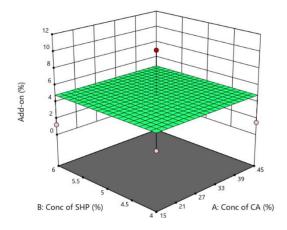
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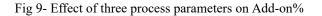
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3.4.3. Effect of process parameters on Add-on%

The 3D surface plot illustrates the relationship between Citric Acid (CA) concentration, Sodium Hypophosphite (SHP) concentration, and the fabric add-on percentage at a curing temperature of 140°C. The flat surface indicates that the add-on percentage remains relatively constant across varying CA and SHP concentrations, suggesting minimal influence of these factors on the overall add-on percentage. The observed add-on values range from 1.22% to 10.18%, with most data points clustering around a lower add-on percentage. The presence of outliers above and below the surface suggests some variability in the deposition process, likely influenced by interactions between the chemicals and fabric absorption characteristics. The contour plot at the base further confirms the uniformity in add-on values, with little significant variation across different CA and SHP concentrations.





IV. CONCLUSION

Citric acid has been proven to be an effective and eco-friendly crosslinking agent for improving the crease recovery of linen fabrics. The study reveals that higher concentrations of citric acid, when combined with sodium hypophosphite as a catalyst, significantly enhance the crease recovery angle. However, excessive concentrations may reduce the whiteness index. The optimal process parameters were identified as 37.3% citric acid, 6% sodium hypophosphite, and a curing temperature of 150°C. Under these conditions, the fabric exhibited improved wrinkle resistance while retaining its tensile strength, whiteness, and overall handle. The catalyst facilitates durable crosslinking without adversely affecting the strength or color of the fabric.

It was observed that a lower curing temperature (140°C), combined with 30% citric acid and 5% catalyst concentration, improved tensile strength while maintaining acceptable appearance and durability. However, higher curing temperatures and excessive chemical concentrations may drastically reduce tensile strength. Wicking and air permeability were not significantly affected under the optimized conditions, confirming the suitability of this finishing treatment for practical and industrial applications.

Industrial Importance: This process presents a sustainable alternative to conventional formaldehyde-based finishes, promoting safer textile processing while achieving superior wrinkle resistance in natural fabrics like linen.

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