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IMPACT OF DELIGNIFICATION ON TENACITY AND LIGNIN CONTENT OF *GREWIA OPTIVA* FIBRES

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ABSTRACT

Grewia optiva a is deciduous tree from the *Tiliaceae* family, it is widely distributed in the hilly regions of north-western India. *Grewia optiva* fibers were traditionally used in rope-making. The fibre extraction process involves retting of stems in water bodies followed by removal of the bark to obtain bast fibers. Lignin is a rigid, hydrophobic polymer that makes the fibers stiff and less flexible. Removing lignin enhances the flexibility and makes them more suitable for applications in textiles and composites. The delignification process aimed to remove lignin which is a rigid polymer that affects flexibility and processing efficiency of the fibers for industrial applications. The study impact of chemical concentrations on reduction of lignin content and fiber tenacity. The results demonstrated that chemical treatments significantly reduced lignin content in *Grewia optiva* fibers and enhanced the suitability of *Grewia optiva* fibers for various textiles and industrial applications.
***Keywords*:** Delignification, *Grewia optiva*, Lignin, Mercerization, Textile

Introduction

Grewia optiva belonging to the *Tiliaceae* family is commonly known by various names such as *bhimal, behel, bewal, bhengal, bihul* and *biu* in the Uttarakhand state of India. This deciduous tree typically attains a height ranging from 10 to 12 meters and is widely distributed in the hilly regions of north-western India, including Jammu and Kashmir, Himachal Pradesh and Uttarakhand. *Grewia optiva* exhibits a notable tolerance to frost, which is prevalent during the autumn and winter seasons in these areas (Kashyap *et al.,* 2015). The tree is characterized by its pale silvery-brown branches, which have a thick and rough texture, and its crown displays a spreading habit. The leaves are arranged in an alternate phyllotaxy. *Grewia optiva* fibers are bast fibers derived from the bark of the *Grewia optiva* tree. The extraction process begins when the stems of the tree shed their leaves and are subsequently allowed to dry. The dried stems are then subjected to retting in a water pond, where the bark is loosened. After water retting, the bark is removed, washed, and dried in sunlight to reduce moisture content. These fibers are categorized as bast fibers due to their origin from the bark of *Grewia optiva* stems. In the Uttarakhand region, local communities are actively involved in the entire extraction process, which includes harvesting, soaking, beating, and shade-drying of *Grewia optiva* fibers. These fibres are obtained in ribbon form (Gupta *et al.,* 2023).

The fibers extracted from *Grewia optiva* are traditionally used for making ropes. The chemical composition of these fibers varies significantly among plants and even within specific fibers, depending on factors such as genetic characteristics, plant age, soil

properties and the conditions under which the plant was grown and harvested during fibers extraction (Mukherjee *et al*., 2018). Various studies have reported that the chemical composition of *Grewia optiva* fibers is influenced by the retting processes and extraction methods employed. The lignin content of *Grewia optiva* fibers varies depending on the extraction method and the retting process used. Singha and Rana (2012) reported that the lignin content in *Grewia optiva* fibers ranges from 14% to 16%. In contrast, Karakoti *et al.* (2022) found a slightly lower lignin content as 12.8 ± 1.5% (w/w). Additionally, Sindwani *et al.* (2017) observed that fibers with manually removed fibrous layers have higher lignin content, while fibers processed with urea retting at 5% weight of material, combined with 5 g/L hydrogen peroxide, exhibit a lower lignin content. Lignin is a rigid, hydrophobic polymer that makes the fibers stiff and less flexible. High lignin content can interfere with the mechanical and chemical processes involved in textile production. Lignin acts as a barrier to the cellulose and hemicellulose within the plant cell wall, limiting their accessibility. Lignin can also negatively impact dye absorption and color uniformity during dyeing processes. Removing lignin enhances the flexibility and makes them more suitable for different applications in textiles and composites. Removing lignin, fibers become easier to process, leading to improved efficiency in textile manufacturing. Removal of lignin (delignification) increases cellulose which is essential for applications where cellulose is the primary component such as in the production of papers and textiles. Removing lignin can increase dyeability of fibers resulting in more consistent and vibrant dyes, which is important for textile finishing (Bajpai, 2018). The delignification process can be achieved through chemical or microbial methods. It facilitates the separation of the fibers into smaller fibrils, known as cellulose microfibrils by eliminating the amorphous components such as lignin and hemicellulose. This process is important for improving the physical and chemical properties of *Grewia optiva* fibers for making them more suitable for production of bio-based materials, papers and textiles (Ilyas *et al*., 2017). In the present study attempt was made to assess the impact of delignification on fibre tenacity and lignin content by varying concentration of different chemicals used for delignification.

Material and Methods

Bhimal fiber used in the study was procured from the Uttarakhand Bamboo & Fibre Development Board (UBFDB) Dehradun. Three chemicals were used in the delignification namely sodium chlorite ($NaClO_2$), acetic acid (CH_3COOH), sodium hydroxide (NaOH) and sulfuric acid (H_2SO_4) was used in testing lignin content.

Delignification Process

The fibre ribbons were delignified using a method reported by Ilyas *et al.,* 2017 which involved delignification followed by mercerization process. Sodium chlorite and acetic acid were used for delignification and sodium hydroxide for mercerization. These treatments helped to remove constituents in the fibres like lignin, hemicellulose, pectin, etc. Removal of these components resulted in splitting of the fibres into smaller microfibrils. The process of delignification helped to remove the lignin from the fibre through chemical dissolution while the mercerization process utilized base treatment to yield changes as swelling ability, structure and dimension. The fibres ribbons were cut in short lengths of 15 to 20 cm for ease in handling and to prevent entanglement during chemical treatment. The delignification was accomplished in two stages. In stage I fibre ribbons were soaked in hot distilled water at 70℃ maintaining MLR 1:30 for 3 hours on water bath. Sodium chlorite ($NaClO_2$) and acetic acid (CH_3COOH) were used in stage I. In stage II samples from stage, I was washed under running water and then soaked in solution containing NaOH for 70 minutes at room temperature. After 70 minutes few drops of acetic acid were added to neutralize the fibres then the fibres were washed under running water, after rinsing the fibres shade dried. Experiments were conducted in controlled conditions in the study, different concentration of chemicals were used on the basis of review collected from various researchers on bast fibre processing. experiments were conducted using three chemicals. Three experiments were by varying concentration of acetic acid, sodium chlorite and sodium hydroxide separately. Detailed procedure is explained under following subheadings.

Effect of different concentrations of acetic acid keeping sodium chlorite and sodium hydroxide constant on *Grewia optiva* fibers

The effects of varying concentrations of acetic acid keeping sodium chlorite and sodium hydroxide constant on lignin removal and fibre tenacity. *Grewia optiva* fibers was assessed by performing delignification in a controlled conditions which included a fixed treatment time of 3 hours, temperature of 70°C, sodium chlorite concentration of 17.15% and a sodium hydroxide concentration of 0.68% with a treatment duration of 70 minutes. The concentration of acetic acid was varied within the range of 14.5% to

16.5% in different experimental runs. For each experiment the fibers were assessed to evaluate the impact of acetic acid concentration on both lignin content and fiber tenacity. The different acetic acid concentrations along with experimental conditions are presented in Table 1.

Table 1: Acetic acid concentrations along with experimental conditions

Stage	Chemicals	Chemical concentration	Time**	Temp**
Stage I	Acetic Acid*	**14.5%, 15%, 15.5%, 16%, 16.5%**	3 hr.	70°C
	Sodium Chlorite**	17.15%		
Stage II	Sodium Hydroxide**	0.68%	70 min	Room temp.

*Variable **Constant parameters

Effect of different concentrations of sodium chlorite keeping acetic acid and sodium hydroxide constant on Grewia optiva fibers

The impact of varying concentrations of sodium chlorite, keeping acetic acid and sodium hydroxide constant on lignin removal and fibre tenacity of *Grewia optiva* fibers. The controlled parameters were treatment time of 3 hours at temperature of 70°C for sodium chlorite and acetic acid treatment (stage I). Acetic acid concentration of 15.5% and sodium hydroxide (NaOH) concentration of 0.68% with a treatment duration of 70 minutes (stage II). Sodium chlorite concentrations were varied within the range of 16.75% to 17.5%. For each experiment tenacity of the fibers and lignin content were measured to determine how the different sodium chlorite concentrations influenced both lignin removal and the strength *Grewia optiva* fibers. The different sodium chlorite concentrations along with experimental conditions are presented in Table 2.

Table 2: Sodium chlorite concentrations along with experimental conditions

Stage	Chemicals	Chemical concentration	Time**	Temp.**
Stage I	Acetic Acid**	15.5%	3 hr.	70°C
	Sodium Chlorite*	**16.75%, 17%, 17.25%, 17.5%, 17.75%**		
Stage II	Sodium Hydroxide**	0.68%	70 min	Room temp.

*Variable **Constant parameters

Effect of different concentrations of sodium hydroxide keeping acetic acid and sodium chlorite constant on *Grewia optiva* fibers

Determination of effects of sodium hydroxide (NaOH) concentration on *Grewia optiva* fibers was also done by performing delignification in a controlled condition. The treatment time was fixed at 3 hours with a temperature of 70°C for acetic acid and sodium chlorite treatment (stage I). The concentration of acetic acid was maintained at 15.5%, and sodium chlorite was at 17.5%. The concentration of NaOH was varied between 0.55% and 0.75% while the treatment time for NaOH was kept constant at 70 minutes (stage II). A total of five experiments were conducted to assess the lignin content and tenacity of the treated fibers. The different sodium hydroxide concentrations along with experimental conditions are presented in Table 3.

Table 3: Sodium hydroxide concentrations along with experimental conditions

Stage	Chemicals	Chemical concentration	Time**	Temp**
Stage I	Acetic Acid**	15.5%	3 hr.	70°C
	Sodium Chlorite**	17.5%		
Stage II	Sodium Hydroxide*	**0.55%,0.60%, 0.65%, 0.70%, 0.75%**	70 min	Room temp.

*Variable **Constant parameters

Evaluation Methodology for Lignin content and Physical properties of fibre

- **Lignin content**

Determination of Lignin was done using TAPPI standard method, T 222 cm-99. In a 250 ml beaker, 1 gm of samples were taken. 15 mL of 72% sulfuric acid pre-cooled to 15 ºC was added gradually into the beaker. The sample was stirred using a glass rod to help the dispersion of samples After that, the beaker was covered and kept at 20 ± 1°C for 2 hrs, with occasional stirring during this period. The beaker's contents were then transferred slowly into a 1 L flask containing 400 mL distilled water. The beaker was rinsed with 80 mL of (3% H_2SO_4) twice, and the contents were added to the flask to ensure complete

transfer of sulfuric acid. The flask was kept under reflux with a constant volume with the help of a reflux condenser for a total period of 4 hours. The solution was kept overnight to settle and precipitate the lignin. Lignin was collected by filtration, dried at 105°C to a constant weight. The percentage yield was calculated based on the original weight of the sample

- **Tenacity of fibres**

Assessment of fibre tenacity was carried on digital instrument comprising of semiautomatic microprocessor with controlled tensile strength tester based on the principle of constant rate of extension. It estimated the tenacity (g/denier) of single fibre strand. The fibre specimen was clamped between the two jaws which were separated vertically at the gauge length of 20mm. the upper jaw of instrument was fixed whereas the lower jaw was movable in upward direction. Each time, at one end of the fibre strand a pretension clip was hanged and a tensile force was exerted by lower jaw through movement in downward direction. The instrument stopped automatically at the moment of fibre break. The statistical value of tenacity and elongation was recorded after each test.

Results and discussions

Impact of acetic acid concentration on lignin and tenacity of *Grewia optiva* fibres

Table 4 shows impact of change in concentration of acetic acid keeping sodium chlorite and sodium hydroxide constant on lignin content and tenacity of fibres.

Table 4 : Impact of different concentration of acetic acid concentrations on lignin and tenacity of *Grewia optiva* fibres

Exp.	Acetic acid (%)	Lignin (%)	Tenacity (g/den)
1	14.5	14.3	1.94
2	15.0	8.89	2.25
3	15.5	4.65	2.67
4	16.0	3.54	2.08
5	16.5	2.78	1.48

As the acetic acid concentration increased a change was observed in both lignin content and fiber tenacity as shown in table 4. Initially, when acetic acid concentration was 14.5%, the lignin content was relatively high at 14.3%, and the tenacity of the fiber was 1.94 g/den. As the concentration of acetic acid increased to 15.0%, the lignin content decreased to 8.89%, and the tenacity improved to 2.25 g/den. When the acetic acid concentration reached 15.5%, lignin content dropped further to 4.65%, and the tenacity reached its highest value at 2.67 g/den. Beyond this point, increase in the acetic acid concentration to 16.0% and 16.5% results in a further reduction in lignin content to 3.54% and 2.78%, respectively. However, the fiber tenacity decreased to 2.08 g/den and 1.48 g/den respectively. This indicates that optimal acetic acid concentration was 15.5% for maximizing fiber tenacity, beyond which further delignification reduces the structural integrity (tenacity) of the fibers. Figure 1 shows the comparative effect of different acetic acid concentrations on lignin content and tenacity of fibres.

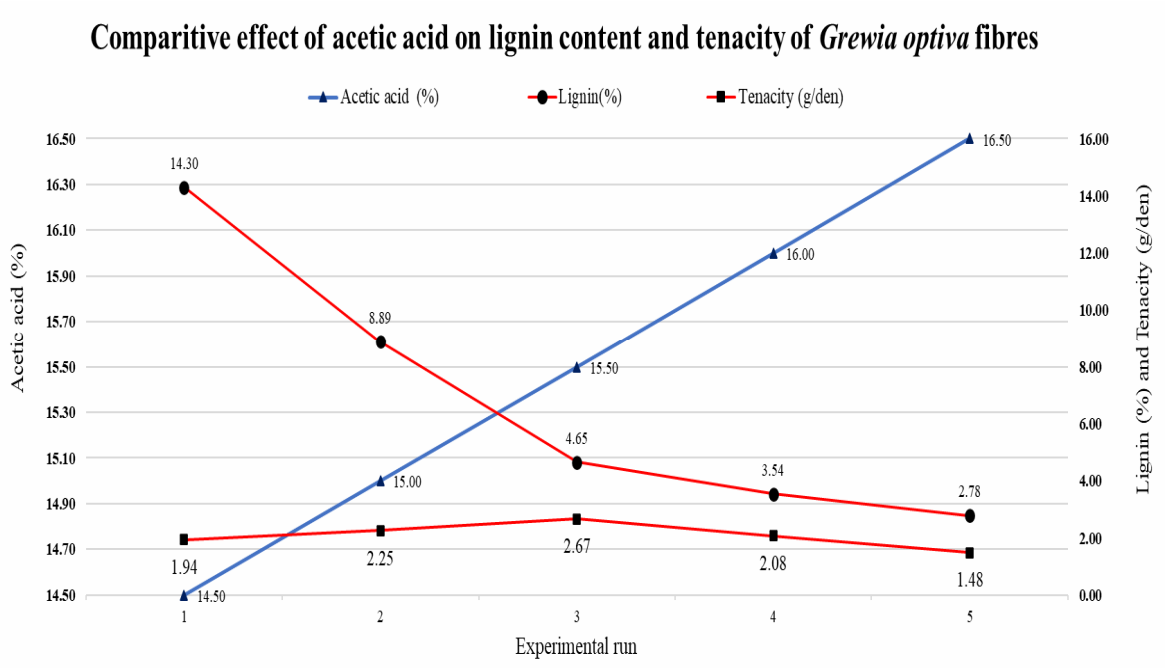

Fig. 1 : Comparative effect of acetic acid on lignin content and tenacity of *Grewia optiva* fibres

Impact of sodium chlorite concentration on lignin and tenacity of *Grewia optiva* fibres

Table 5 shows impact of various concentration levels of sodium chlorite on lignin content and fibre tenacity. Sodium chlorite at different concentrations showed significant changes in both lignin content and fibre tenacity.

Table 5 : Impact of different concentration of sodium chlorite on lignin and tenacity of *Grewia optiva* fibres

Exp.	Sodium chlorite (%)	Lignin (%)	Tenacity (g/den)
1	16.75	18.7	1.40
2	17.00	14.3	1.79
3	17.25	11.3	2.43
4	17.50	5.3	2.78
5	17.75	3.9	2.30

The effects of sodium chlorite concentration on lignin content and fiber tenacity revealed that as the sodium chlorite concentration increased from 16.75% to 17.75% there was change in both lignin content and fiber tenacity as shown in table 5. Initially, at a concentration of 16.75% of sodium chlorite, the lignin content was high at 18.7%, and the fiber tenacity was relatively low at 1.40 g/den. When the concentration of sodium chlorite was increased to 17.00%, the lignin content decreased significantly to 14.3%, and the tenacity improves to 1.79 g/den. As the concentration increased further to 17.25%, lignin content continued to decrease to 11.3%, and fiber tenacity improved to 2.43 g/den. At 17.50% sodium chlorite concentration, the lignin content dropped further to 5.3%, and the tenacity reached to peak at 2.78 g/den. However, when the sodium chlorite concentration increased to 17.75%, the lignin content reduced further to 3.9%, but the tenacity decreased slightly to 2.30 g/den. Optimal sodium chlorite concentration (17.50%) for achieving maximum fiber tenacity, beyond which further increased in concentration reduced the lignin content but also decreased the tenacity of fibres. Comparative effect of sodium chlorite Concentration on lignin content and tenacity is depicted in Figure 2.

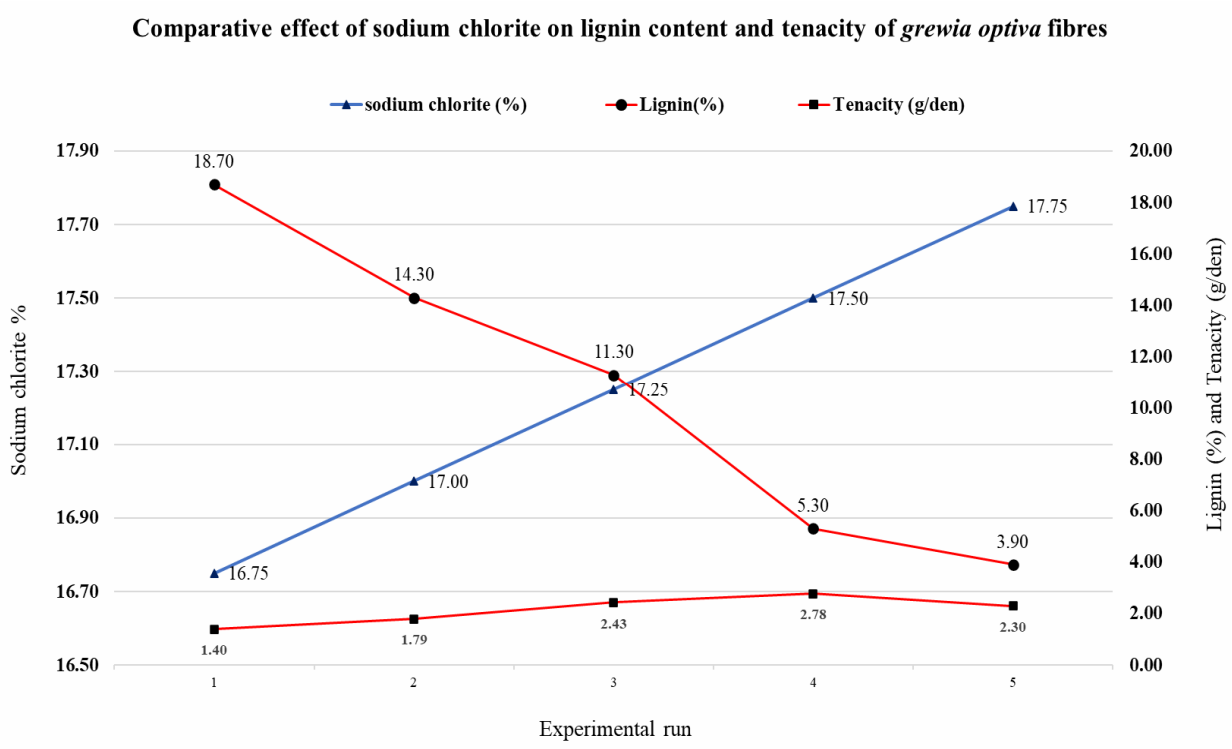

Fig. 2: Comparative effect of sodium chlorite on lignin content and tenacity of *Grewia optiva* fibres

Impact of sodium hydroxide concentration on lignin and tenacity of *Grewia optiva* fibres

Sodium hydroxide concentration at different levels shows changes in both lignin content and fibre tenacity. As the concentration of NaOH increases, there is a clear pattern in both lignin content and fiber tenacity. The effect of sodium hydroxide on lignin content and fibre tenacity of *Grewia optiva* fibres is presented in Table 6.

Table 6 : Impact of different concentration of Sodium hydroxide on lignin and tenacity of *Grewia optiva* fibres

Exp.	Sodium hydroxide %	Lignin (%)	Tenacity (g/den)
1	0.55	15.1	2.29
2	0.60	13.7	2.31
3	0.65	11.5	2.62
4	0.70	7.60	3.22
5	0.75	4.16	1.94

Table 6 reveals that initially, at a concentration of 0.55%, the lignin content was 15.1%, and the fiber tenacity was 2.29 g/den. As the NaOH concentration increases to 0.60%, the lignin content decreases to 13.7%, and the fiber tenacity slightly increases to 2.31 g/den. With a further increase in NaOH concentration to 0.65%, the lignin content decreases to 11.5%, and the tenacity improves to 2.62 g/den. At 0.70% NaOH concentration, the lignin content drops more significantly to 7.60%, and the fiber tenacity reaches its peak value of 3.22 g/den. However, when the NaOH concentration increases further to 0.75%, the lignin content reduces to 4.16%, but the fiber tenacity decreases to 1.94 g/den. These observations show that increasing NaOH concentration reduces the lignin content in the fiber, which initially enhances fiber tenacity up to an optimal concentration (0.70%), beyond this concentration lignin content continues to decrease but excessive delignification damages to the fiber structure that reduces fibre tenacity. Comparative effect on lignin content and tenacity of sodium hydroxide concentration at different levels is depicted in Figure 3.

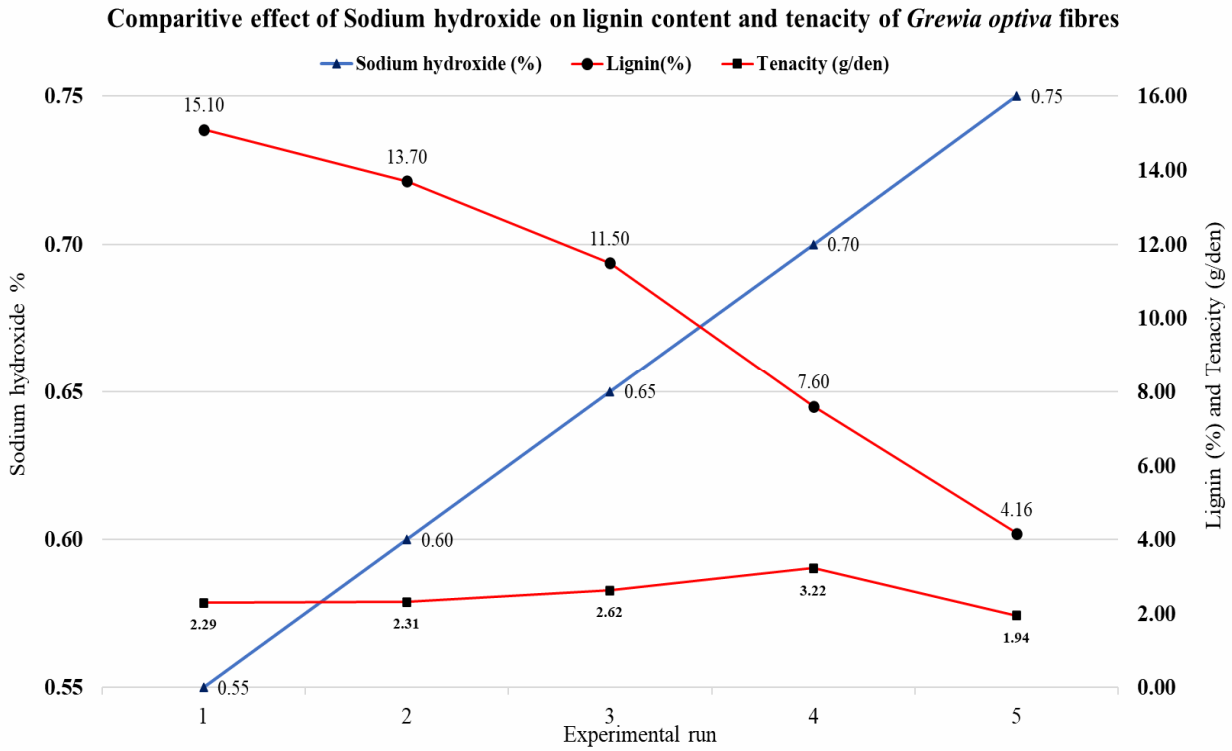

Fig. 3 : Comparative effect of Sodium hydroxide on lignin content and tenacity of *Grewia optiva* fibres

Conclusion

The present study examined the impact of varying concentrations of sodium hydroxide (NaOH), acetic acid and sodium chlorite on the delignification of *Grewia optiva* fibers focusing on their impact on lignin content and fiber tenacity. The results demonstrated that each chemical treatment has an optimal concentration that effectively reduces lignin content while maintaining fiber tenacity. The most effective concentrations were 0.70% for NaOH, 15.5% for acetic acid and 17.5% for sodium chlorite. These concentrations resulted in the greatest reductions in lignin content and the highest fiber tenacity values indicating that controlled delignification can improve the mechanical properties of *Grewia optiva* fibers. Exceeding these optimal concentrations led to decrease in fiber tenacity it highlights the importance of controlled delignification in order to the preserve fiber tenacity. The findings of the study suggest that careful optimization of chemical treatments can significantly

enhance the suitability of *Grewia optiva* fibers for various textiles and industrial applications.

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