

## Original Research Paper

## Fabrication Of Loofah Sponge as An Effective Natural Chromium Sequestrant

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## ABSTRACT

Industrialization is key to a nation's progress, but exposure to industrial effluents with heavy metal causes deadly diseases, so mitigation effects must be applied to safeguard human beings and the environment. In this study, loofah sponges, a natural heavy metal sequestrant, were modified with sodium hydroxide, acetic acid, sequestering, and wetting agents and showed increased mechanical strength with strong interfacial bonding with the composite materials than untreated loofah sponge. Characterization techniques including ASTM-D570, FTIR, SEM, SEM-EDX, TGA (thermogravimetry analysis), XRD (X-ray diffraction spectra), and Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES) were employed to investigate the SSGP-pretreated loofah sponge. The FTIR and SEM examination revealed removal of waxes and contaminants, and heavy metal adsorption on the loofah sponge noted in SEM-EDX image. The ICP-OES analysis revealed that pretreated loofah sponge adsorbed about 39 mg/g of chromium. This study proved that pretreated loofah sponges adsorbed about 12.4% more chromium than untreated loofah sponge. So, pretreated loofah sponge, an environmental-based green technology, can be used as an effective biocarrier for heavy metal contaminated effluents. This study supports sustainable development goals of clean water and sanitation and recommends using modified-loofah sponge for large scale application.

Key Words	Loofah sponge, natural fibers, surface modifications, fiber, chromium, heavy metals, industry, industrial effluents
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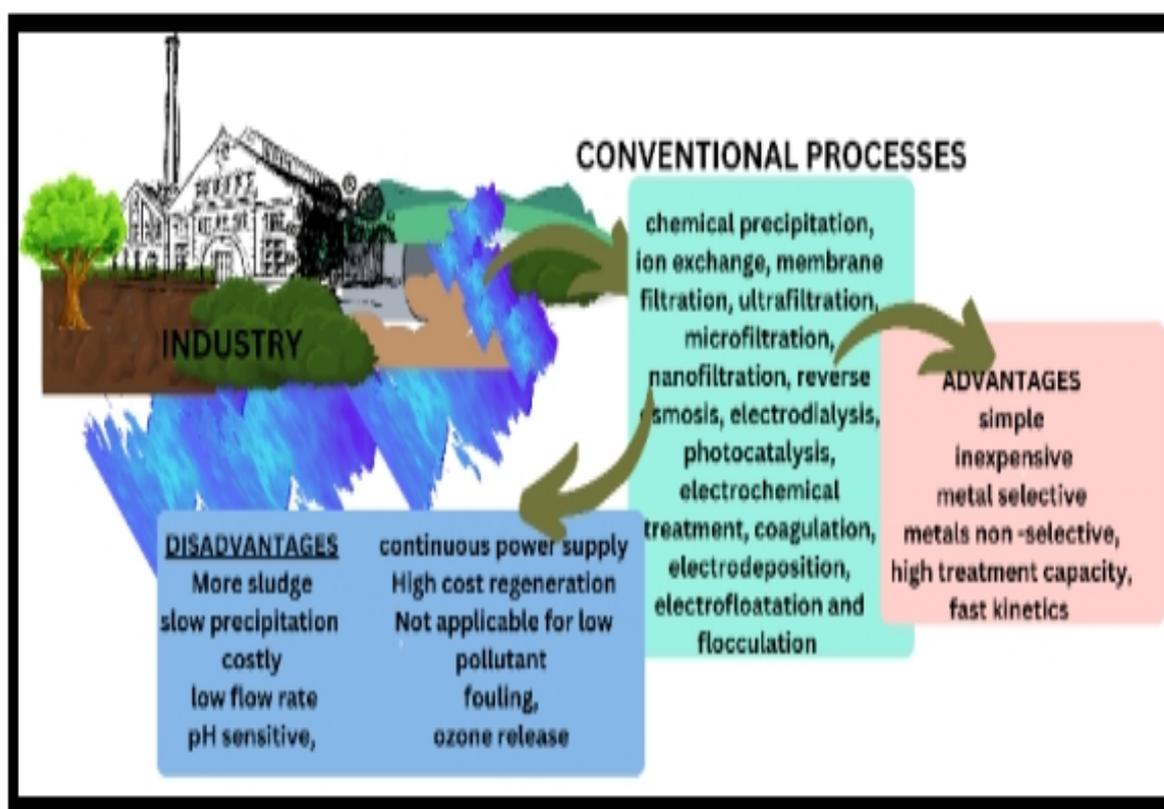
## INTRODUCTION

Public Health Services of different countries reported the exposure of hazardous excess metals to significant numbers of people in different ways (Cotruvo, 2017). The high atomic density and weight are the properties of the recalcitrant heavy metal that make them quite toxic even at low density when released into the environment. The heavy metal were released into the environment after industrialization as untreated or partially treated effluents, discharged in the water stream, and due to their constant and bioaccumulation normality, the heavy metal get constantly accumulated in the environment (Saxena et al., 2024; Vidhya et al., 2023). The heavy metal are naturally released into the environment either through rock and soil leakage to water streams, or by human activities, such as unprocessed wastewater that was directly discharged to rivers (Rezooqi et al., 2021). According to the US-EPA, lead, cadmium, and chromium are designated as the top priority contaminants that are of major public concern (Faheem et al., 2020). In general, heavy metal are carcinogenic and mutagenic, and they also aid cell apoptosis, so to mitigate such heavy

metals, natural methods must be taken into account. The advantages and disadvantages of conventional processes are depicted in Fig. 1.

**Fig. 1: Advantages and disadvantages of conventional process**

Though advantages are present in conventional processes (Agarwal et al., 2006), the disadvantages overpower the goodness, so biosorption can be a glimpse of the future (Kanamarlapudi et al., 2018). Naturally, chromium is predominantly encountered in its oxidation states Cr (VI) and Cr (III) (Goddeti et al., 2020), where Cr (VI) showed several times more toxicity, as it is present in industrial effluents that are discharged in tones, so chromium mitigation must be propelled as it is carcinogenic and mutagenic. Loofah sponge (LS) obtained from dried fruit of *Luffa cylindrica* (L.), a member of the cucurbitaceous family, has a polyporous structure and forms a natural mat when dried that can serve as a bio-adsorbent for treating industrial effluents. Asia, Africa, and tropical America are the main cultivation spots for loofah plants (Oboh and Aluyor, 2009). The LS has more strength, stiffness, and energy absorption capacity and contains cellulose (62%), hemicelluloses (20%), lignin (11.2%), extractives (3.2%), and ash (0.4%), with functional groups to adsorb metals from any sources. Over the past decade, LS was explored less than other fibers as the source of wastewater treatment, so, this research explored LS as a heavy metal sequestrant for



wastewater treatment. This study evaluated the potential of chemically SSGP-pretreated loofah (Sodium hematophosphate-sodium hydroxide, glycerol, potassium permanganate treatment), an eco-friendly renewable agricultural product, as heavy metal sequestrant for the treatment of industrial wastewater. Unmodified loofah adsorbed about 34.7 mg/g of chromium, while SSGP-pretreated LS adsorbed about 39 mg/g of chromium.

## 2. MATERIALS AND METHODS

### 2.1. Fiber collection

Dried LS in size between 18 and 22 cm was chosen and collected from a local market in Perambur, Chennai, Tamil Nadu, and washed thoroughly with distilled water to remove dirt and soil, and they were dried and cut down to size 2 cm and used.

### 2.2 FIBER MODIFICATION PROCESS

**2.2.1 WASHING AND PRE-STERILIZATION:** The 0.85% of saline was used to remove dirt, biofilms, and then loofah were cut into 2 cm height and then pretreated with chemical treatments.

**2.2.2 ALKALI TREATMENT:** To increase the tensile strength of LS, 4% NaOH was used to treat LS at 120°C for 4 hours using a hot plate stirrer. Then washed and dried in a hot air oven at 120°C.

**2.2.3 ACID TREATMENT:** LS was treated with 2% acetic acid at room temperature for 30 minutes and washed with distilled water.

**2.2.4 NEUTRAL SALT TREATMENT:** LS was treated with 0.6% potassium permanganate for 5 minutes at room temperature and washed thoroughly till neutral pH was reached.

**2.2.5 SEQUESTERING AGENT:** LS treated with 2 % (2 g/l) of sodium hematophosphate at 50 °C for 3 hours and washed with distilled water.

**2.2.6 WETTING AGENT:** LS further treated with 30% glycerol for 30 minutes and washed again with distilled water and 70% ethanol several times to remove dirt and scum, then dried in a hot air oven at 160°C for 4 hours and used. The LS and its treated are depicted in Fig. 2.



**Fig. 2: (a) LS (b) cross section of LS (c) cut piece of LS (d) alkaline, acid treated LS (e) KMnO<sub>4</sub> treated LS (f) SSGP-pretreated LS.**

The fabricated LS were modified using sodium hydroxide, acetic acid, potassium permanganate (Hallad et al., 2019), and sodium hematophosphate. The inorganic cyclophosphate sodium hematophosphate increases permeability (Sampaio et al., 2022), and it removes the surface impurity and acts as a surfactant as well as sequestrant (Karim et al., 2024). It was used with alkali, potassium permanganate, which improves the strength of the fiber. The tenacity was increased, elongation of fiber occurs (Wetaka et al., 2016), and delignification of fiber occurs (Ghali et al., 2009), which increases the shelf-life of fibers. Acetic acid replaces water and aids plastination of fiber (Dhir et al., 2020),

and shifts pH to neutral because of alkali treatment. Glycerol decreases surface tension and decreases molecule propensity to stick together (Karim et al., 2024).

## 2.3 PHYSICOCHEMICAL CHARACTERIZATION OF LOOFAH SPONGE

### 2.3.1 ASTM-D570 TEST

The ASTM D570 procedure is used for adsorption rate interpretation of untreated and SSGP-pretreated LS. About 10 x 0.5 x 0.1 mm LS were immersed in a separate container containing distilled water for an hour at room temperature, and samples were removed at 15-minutes interval and kept between filter sheets between weights. LS water absorption percentages were calculated by weighing filter paper with a digital balance machine (Tanobe et al., 2014).

$$\text{Water Absorption \%} = \frac{\text{weight after immersion} - \text{weight before immersion}}{\text{weight before immersion}} \times 100$$

### 2.3.2 FTIR

The positional status of LS chemical functional groups was ascertained by a Fourier Transform InfraRed spectrophotometer (FTIR, Shimadzu IRTRACER 100, Japan). The untreated and treated LS of about 1 mg was crushed and mixed with a 200 mg dosage of potassium bromide at room temperature and 65% humidity. The interaction of infrared light with untreated and SSGP-pretreated LS produces its molecular fingerprint in the wave number range of 400–4000  $\text{cm}^{-1}$  at a scan rate of 32 with a resolution of 2  $\text{cm}^{-1}$ .

### 2.3.3 XRD

The crystalline and amorphous structure of untreated and treated LS was investigated by X-ray Diffraction (XRD, X'Pert PRO MRD, PANAnalytical, Netherlands) at 50 mA current and 40 kV voltage. The untreated and treated LS diffraction measurements were scanned from 5-100 ( $2\theta$ ) with 0.9°  $\text{min}^{-1}$  scanning rate at 25 °C.

### 2.3.4 TG ANALYSIS

Thermogravimetry (TG) was carried out on untreated and treated LS using a thermogravimetry analyzer (TGDTA, NETZSCH, STA 449F5, Germany) applying a heating rate of 30°C/10.0(K/min)/700°C.

### 2.3.5 SEM (SEM, AMETEK)

The loofah fibers surface morphological property was noted by scanning electron microscopy (SEM, AMETEK, United States), imaging mode at 2 micrometers at a working distance of 20 mm. The loofah fibers were prepared by aluminium carrier precipitation and covered with thin gold film under high vacuum at 15 kV voltage.

### 2.3.6 SEM EDX

The gold sputtered loofah fibers were analyzed for the different elements to determine their strength and distribution using SEM equipped with an energy-dispersive X-ray (EDX) detector (EDX AMETEK BV, Tilburg, Netherlands).

### 2.3.7 ICP-OES

The samples were acid digested and analyzed for elemental characterization using Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES, Agilent Technologies).

## 2.4 CHROMIUM REMOVAL BY LOOFAH SPONGES

About 1 gram of LS was added to 100 ml of 200 ppm chromium containing conical flasks and incubated for 6 days at 120 RPM in 37 °C, and then filtered to remove the filtrate and analyzed for the presence of chromium in the sample using ICP-OES analysis (Agilent Technologies, USA).

## 3.RESULTS

### 3.1 Water adsorption test

The LS was cut into 10 x 0.5 x 0.1 mm and used for the adsorption test. The LS showed increased adsorption with an increase in contact time. The absorbency of untreated and SSGP-pretreated LS showed 123 and 114% of adsorption within 15 minutes, respectively, and further increase in adsorption was noted, the highest adsorption rate was 132 and 137% for untreated and SSGP-pretreated fiber noted within 24 hours. SSGP-pretreated LS has more water adsorption capacity than untreated LS, so it has great efficiency in absorb the contaminated effluents that benefits the contamination removal as well as heavy metals removal.

### 3.2 FTIR

FTIR results indicate that sodium hematophosphate-sodium hydroxide, glycerol, and potassium permanganate treatments were perfectly incorporated into fiber and chemical modifications shifted peaks of LS from higher wavelength to lower wavelength. The FTIR peaks show the chemical complexity nature of untreated LS and SSGP pretreated LS, as depicted in Fig. 3. Functional groups and chemical stretching involved in pretreated and SSGP-modified LS at a wavelength between 400 and 4000  $\text{cm}^{-1}$  were shown in Table 1. The untreated sample with bond 430  $\text{cm}^{-1}$ , 513  $\text{cm}^{-1}$  denote highly polar covalently bonded C-Cl stretching and C-H bending vibrations, respectively, which are not present in the treated sample. The chemical modifications show an increase in bond dissociation energy with increased heat and break bonds to simpler forms or free bond which helps sequestration of heavy metal to LS.

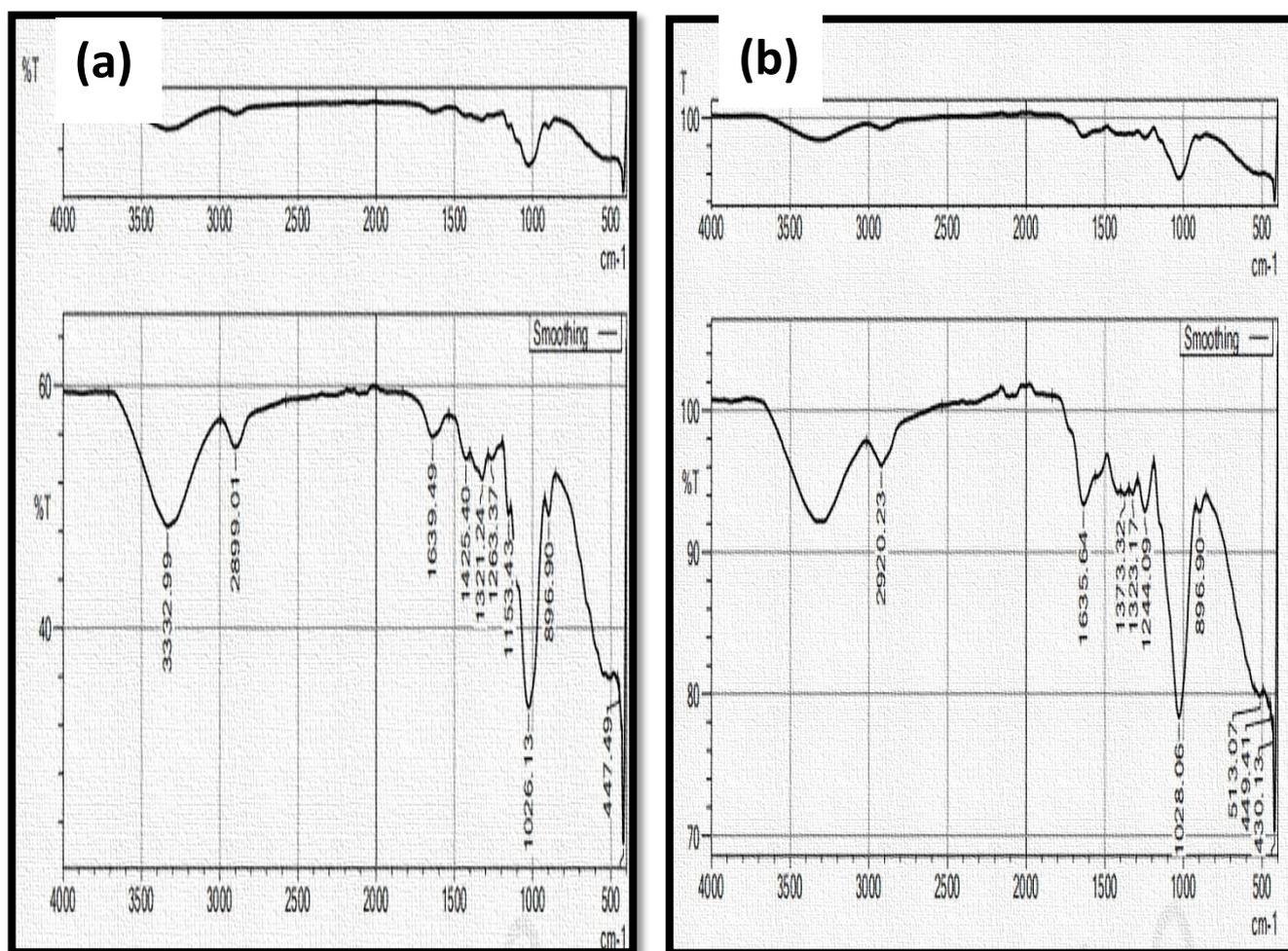


Fig. 3: FTIR spectra peaks of (a) untreated (b)SSGP-pretreated LS

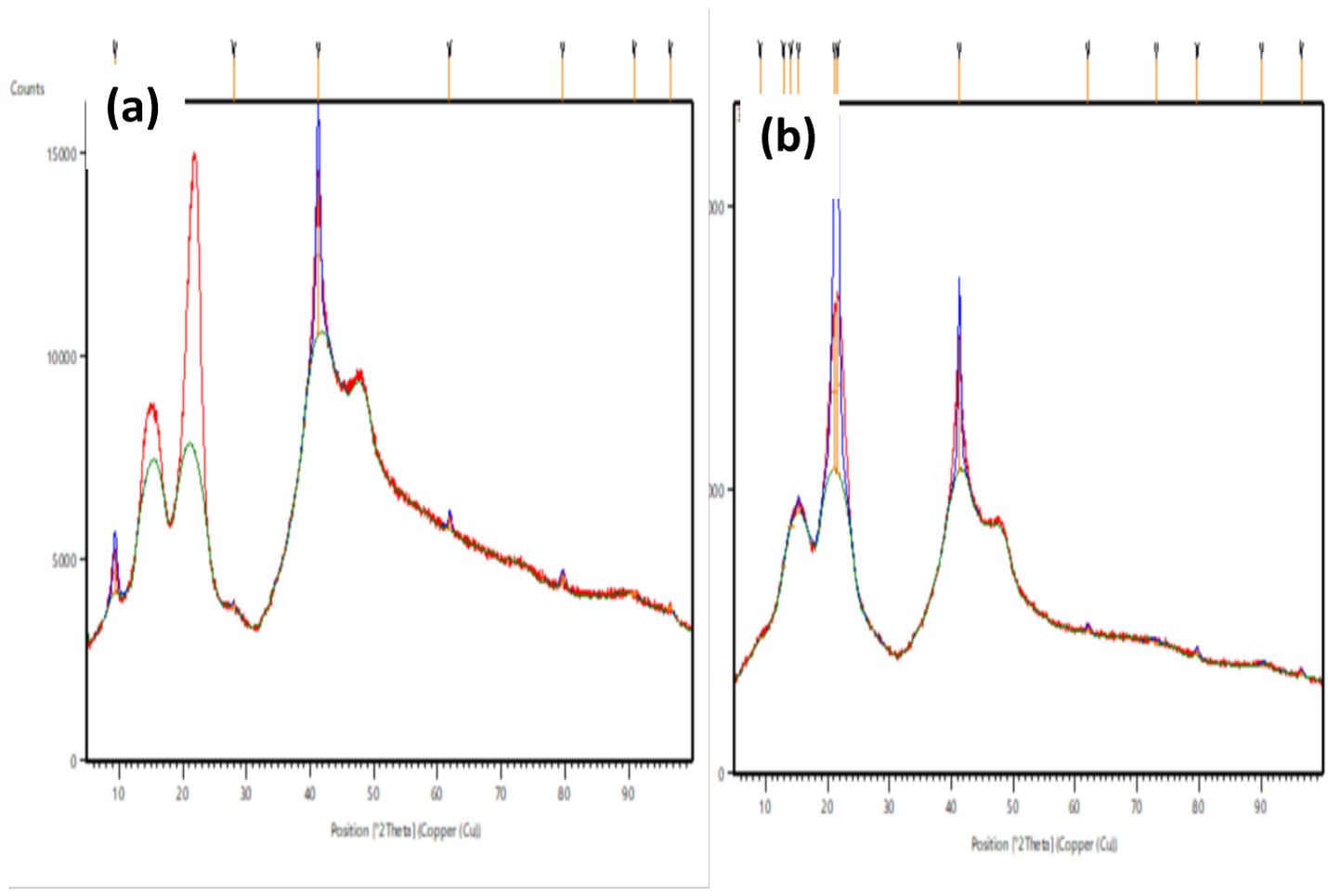
Table 1: Chemical stretches and bending distributions and peak locations of Luffa fiber

Untreated	Treated	Chemical stretching	Reference
420	420	Aryl disulphides S-S stretch	(Coates, 1996)
430	—	C-Cl stretching vibration	(Balachandran et al., 2011)

449	447	449cm <sup>-1</sup> (PO <sub>4</sub> bond) to aryl disulphides S-S stretch.	(Coates, 1996; Ramdan et al., 2017)
513	-	C-H bending vibrations.	(Jeya et al., 2018)
897	897	C-O-C stretching, β-glycosidic linkage.	(Rahman and Khan, 2007)
1028	1026	Pectin-OH bond stretches more in SSGP-pretreated LS than untreated alkyl and aryl halides. In untreated loofah fibers C-H stretches happened at higher points.	(Mollah et al., 2023)
1244	1153	C-O stretching (1244 cm <sup>-1</sup> ) to C-O-C bond (1153 cm <sup>-1</sup> ).	(Rusu et al., 2008; Saito and Iwata, 2012)
1323	1263	NO <sub>2</sub> stretches (1323 cm <sup>-1</sup> ) shifts to C-H stretching at 1263 cm <sup>-1</sup> in the fingerprint region-pyranose ring.	(Thombare et al., 2023; Zhang et al., 2018a)
1373	1321	Phenol O-H bending (med) of untreated gets shifted to alkane C-H stretch (methyl and methylene groups of cellulose, hemicellulose, and lignin).	(Lakshmanan et al., 2018)
1636	1425	Alkene C=C symmetric stretch (med) in untreated fibers gets stretched to CH <sub>2</sub> bending hidden in fingerprint regions.	(Senthamaraikannan et al., 2019)
2920	1639	Alkane CH <sub>2</sub> asymmetrical stretching (2920 cm <sup>-1</sup> , medium bond) of waxes in loofah fibers, gets shifts to alkene C=C stretch (med) in SSGP-pretreated fibers.	(Zhang et al., 2018b)
3333	2899	Sharp alcoholic O-H stretch (med) of untreated gets shifted to alkane C-H stretch (med), present in lignin, cellulose, hemicellulose methyl and methylene groups.	(Zeng et al., 2019))
3758	3333	Pectin, sharp alcoholic O-H stretching of untreated fibers (α – cellulose) shifts to sharp alcoholic O-H stretch (med stretching (lignin).	(Karim et al., 2023)

### 3.3 XRD

Sharp and strong peaks indicate the crystallinity property of both LS samples, SSGP-pretreated LS shows increased tensile strength. The pattern of untreated loofah exhibits a broad peak at  $2\theta = 21.6^\circ$  and a small peak at  $21.1^\circ$ ,  $17.9^\circ$ . The pattern of SSGP-pretreated loofah exhibits a broad peak at  $2\theta=41.24^\circ$ ,  $27.9^\circ$ , and a small peak at  $9.37^\circ$  (Fig. 4).

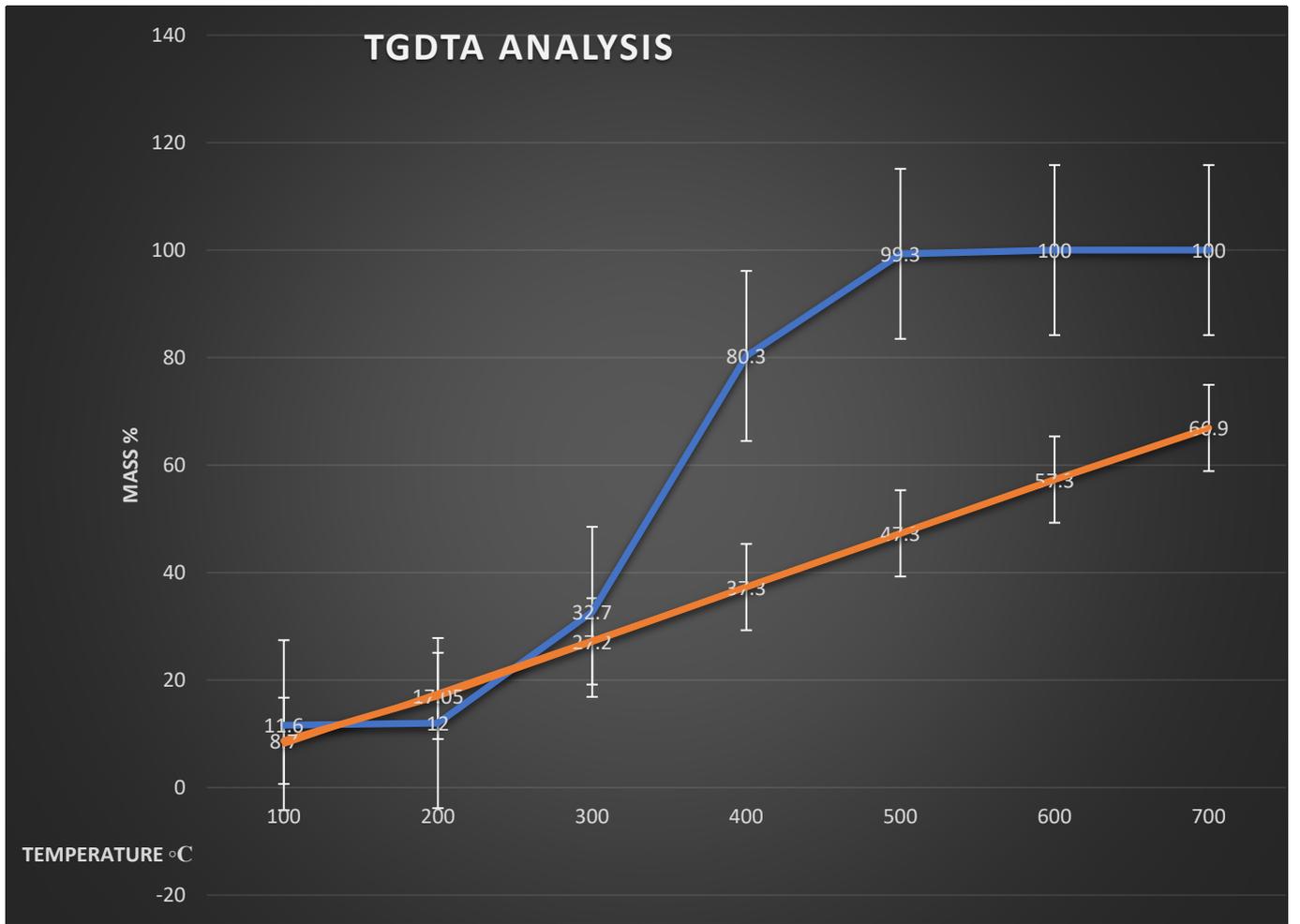


**Fig. 4: (a) XRD graph interpretation; (b) XRD-SSGP-pretreated; (c) XRD-untreated LS.**

The characteristic peaks at  $9.3^\circ$  were not present in untreated LS. The  $41.27^\circ$  peak shifts to  $41.24^\circ$ , and  $27.6^\circ$  peak shifts to  $27.9^\circ$  with higher relative intensity. The increase in crystallinity index shows high tensile strength and microstructural regularity of SSGP-pretreated LS. So, chemical modifications in LS increased crystalline index so, the modified LS has high strength or performance, wear resistance, strength and toughness and ductility than untreated LS.

### 3.4 TG/DTA

Thermogravimetric analysis done by a thermal analyser (NETZSCH STA 449F5, Germany) for both untreated and treated LS was investigated at a heating rate of 10 K/min at 250 ml min<sup>-1</sup> nitrogen flow rate in the range of 30-700 °C (Matter, Hassan, Elfaramawy & Esmail 2024). The result shows that at temperature 700°C, only 66.9 % mass loss occurred in SSGP-pretreated LS, but untreated LS mass loss happened within 600°C, shows SSGP-pretreated LS was greatly strengthened by the modification treatment by SSGP, and as the sturdiness is increased in SSGP-modified LS, it can be reused more than untreated LS fig. 5, Table 2.



**Fig. 5: TGDTA ANALYSIS OF UNTREATED AND TREATED LS**

**Table 2: TGDTA ANALYSIS OF LS**

Temperature °C	TGDTA Untreated %	TGDTA SSGP-pretreated %
100	11.6	8.7
200	12	17.05
300	32	27.2
400	80	37.3
500	99.3	47.3
600	100	57.3
700	100	66.9

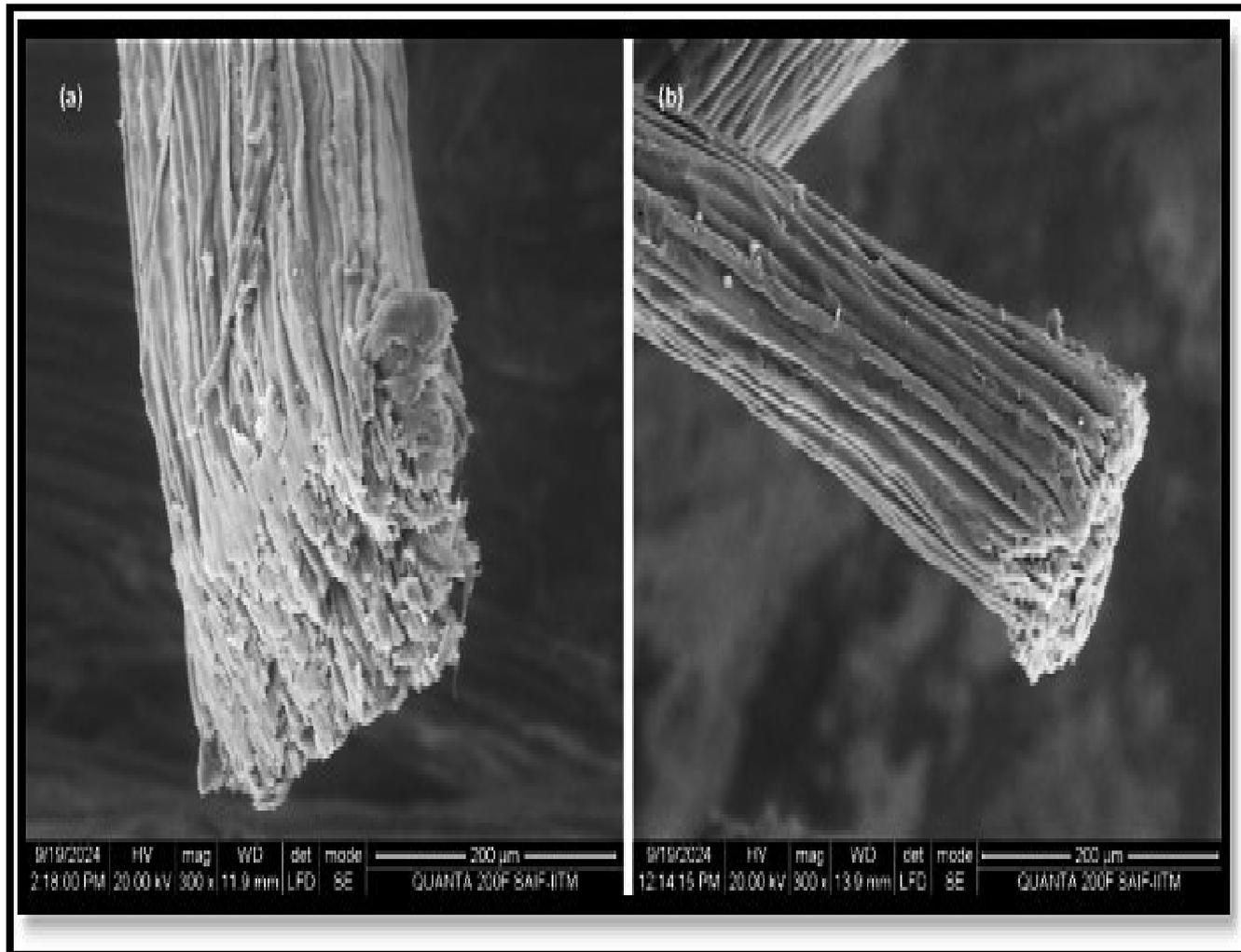
**3.5 SEM**

The morphological properties of untreated and treated fiber at 300 X magnification were captured using Scanning Electron Microscopy images (FEI Quanta 600 FEG ESEM, USA). The change in surface morphology, removal of waxes and surface impurities on surface visualized in SEM images. The waxes and impurities in the loofah surface prevent water adsorption in untreated LS, and after chemical treatment, waxes, dirt, impurities-free surface were visible in SEM images using SEM (Fig.6).

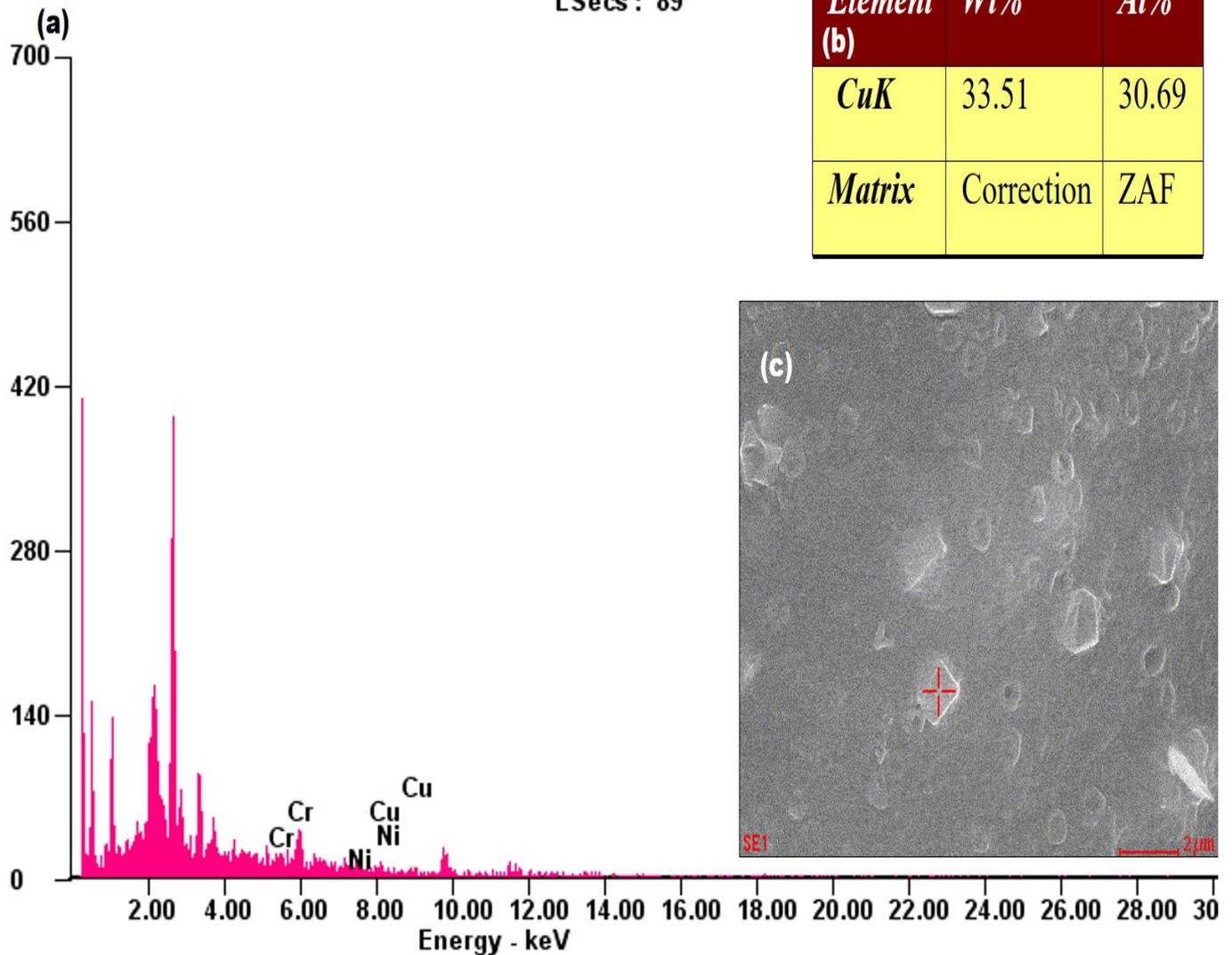
**Fig. 6: SEM images of (a) untreated; (b) SSGP-pretreated**

### 3.6 SEM-EDX

SEM with energy dispersive X-ray spectrometry (SEM-EDX; FEI Quanta 600 FEG ESEM, USA) elemental analysis



showed the presence of the Crystal lattice structured chromium heavy metal presence in the surface of the both untreated and SSGP modified LS (Fig.7). The weight and atomic percentage of up to 26.82 % and 30 % were noted in the surface of LS, which shows free bonds sequestered the heavy metal on the LS surface as well as layers of LS. So, the modified LS show the chromium adsorption capability on all layers of LS.



**Fig. 7: SEM-EDX OF ELEMENTAL CHROMIUM**

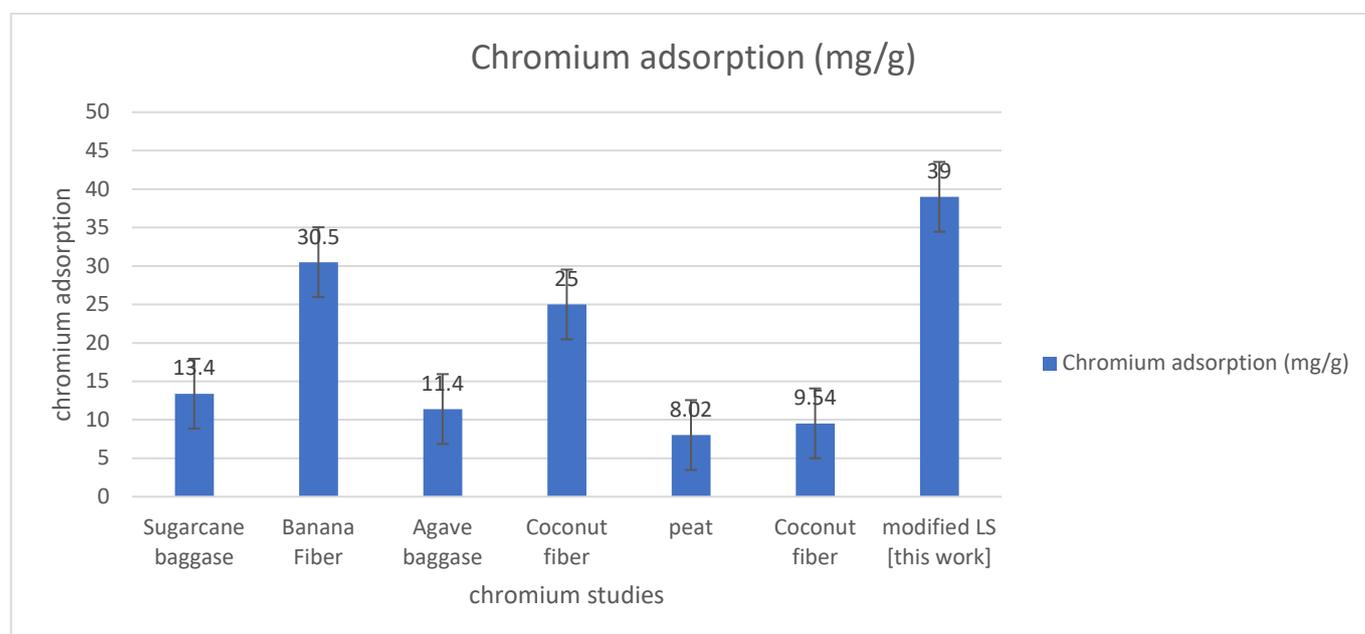
### 3.7 ICP-OES ANALYSIS

The ICP-OES analysis uses argon plasma to excite the HM containing solution and emits light, which has a wavelength proportional to elements. The analysis showed that untreated loofah adsorbed only 34 mg/g of chromium, but SSGP-pretreated LS adsorbed about 39 mg/g of chromium, which is 12.4 % more than untreated LS. Thus LS modification have increased the chromium adsorption in surface and layers which is proved by the ICP-OES analysis.

### 5. DISCUSSION

The conventional treatment process is harmful to our environment as it uses chemicals in large amount, which makes the process expensive, and also the conventional approaches have many disadvantages, as discussed in Fig.1, so the secondary pollutants discharged in conventional treatments are also a great threat to our environment, and the treated water must be further treated in order to get rid of those chemicals, which makes the process not acceptable. The natural bioremediation of heavy metal is done by many approaches, among which activated carbon, biological organism-based heavy metal biodegradation and natural fibers-based bioremediation are vividly used. The activated carbon-based bioremediation results showed that activated carbons from different sources showed different results and had specificity in the adsorption. The activated carbon process needs tons of materials as the source, but only very less amount is produced at the end of pyrolysis, which even produces air pollution. The activated carbon cannot be implemented on a large scale as the source varies, the result also varies and then pyrolyzing tons of sources on a daily basis is also a great query, so implementing on a large scale is not approachable. The biological based treatment is a great source of bioremediation, but growing microbes in daily basis needs good source of amendments, so this

makes the process expensive, and contamination from the environment is possible, so failure of approach is also possible. Implementing one organism is not possible, so different micro-organism based bioremediation must be studied as the optimized heavy metal degradation is possible only at optimized conditions, which is specific for different organisms, so implementing on a large scale requires a bioreactor for propelling certain organisms at certain conditions, and using organisms that is not present in that habitat may cause imbalance in the environment, In certain studies, genetically modified organisms are also used, so ethical reasoning is also present in this approach, so this technique has a lot of lagging in large scale implantation. The LS, natural adsorbent is cost effective and locally available materials can be implemented as it is inexpensive. and makes this technique approachable. This technique can be combined with existing technology to make the process cheap or used as such as a novel approach. This technique doesn't need a new plant construction as it can be implanted in any type of treatment plant and it is also easy to train professionals for this approach. The collected samples were cut in 2 cm size and modified with SSGP. LS is composed of 55–90 % of cellulose, 8-22 % of hemi-cellulose, 10-23 % lignin, extractives >3.2 %, ash, and others 0.4% (Chen et al., 2019), as the impurity blocks the water adsorption, alkali treatment is done (Begum et al., 2021). The SSGP-pretreated LS showed about 137% of increased water adsorption with an increase in contact time (Tanobe et al., 2014). FT-IR spectra peaks at a wavelength between 400 and 4000  $\text{cm}^{-1}$  in untreated and SSGP-pretreated LS show change in functional groups, and certain wavenumbers were not present in modified LS, which shows incorporation of SSGP, lower bond energy, and also change in chemical stretching is also noted in modified LS as mentioned in Fig. 3, Table 1. An increase in tensile strength and crystallinity index was noted with sharp and strong peaks in modified LS (Mohanta and Acharya, 2016) and more peaks such as  $2\theta=9.37^\circ$  showed the sturdiness of the modified LS (Goddeti et al., 2020). Thermogravimetric analysis investigated for both LS and the SSGP-modified LS withstands approximately 40% of sample even at  $700^\circ\text{C}$  (Matter, Hassan, Elfaramawy & Esmail 2024). The SSGP-pretreated LS exhibited clear surface morphology without water adsorption impeding waxes and impurities (Goddeti et al., 2020; Matter et al., 2024) and chromium metal presence noted in the LS surface (Mohanta and Acharya, 2016). The ICP-OES analysis (Kotelnikova et al., 2024; Zeng et al., 2019) proved the adsorption status of LS, SSGP-modified LS about adsorbed 39 mg/g of chromium, which is more than previously reported studies with sugarcane bagasse (Sharma and Forster, 1994), banana fiber (Begum et al., 2020), agave bagasse (Bernardo et al., 2009), coconut fiber (Franguelli et al., 2019), peat and coconut fiber (Kaszycki et al., 2004) and more depicted in Fig. 8.



**Fig .8 Chromium adsorption by natural fiber other studies**

The modified LS was reported to be copper sequestrant in our previous study (Santhiya Jayakumar & Sharmila 2024). This study recommends the environmental based green technology of using SSGP-pretreated LS as a superior option for chromium sequestration. can be used in large scale applications. Thus, the SSGP-pretreated LS can be used as a

source of heavy metal sequestrant in industrial effluent contaminated by chromium, and can also be used in the metal contaminated sites for better chromium sequestration.

## **5.1 POTENTIAL CHALLENGES OF LARGE-SCALE APPLICATION**

Though using LS for secondary treatment can be used as a novel approach or it can be combined with conventional methods, approving new technology by public as well as government and private bodies takes lots of time.

LS is seasonal and takes lots of time to grow and reap, so fibers must be switched or stocked for the future usage.

## **5.2 FUTURE RESEARCH**

In future, the SSGP treatment must be tested for other fibers, as per the locally availability of India as well as other countries, for converting the conventional approach, which produces the secondary pollutants as the result of treatment, to a natural fiber based green approach for the betterment of one's country's agriculture sector as well as reducing contamination in the environment using agriculture based natural fibers such as LS. The SSGP-LS was tested only for copper and chromium sequestration, so more research must be done for other heavy metal and other pollutants, such as dyes, and other contaminants such as sulphate, nitrate, total dissolved solids, total suspended solids and more, removal.

## **6. CONCLUSION**

This study shows novel SSGP-pretreated LS, as an effective heavy metal adsorbent. The sodium hemetaphosphate, glycerol, potassium permanganate, acetic acid, sodium hydroxide modified, SSGP-pretreated LS, showed effective chromium adsorption, about 12.4 % increase in efficiency of chromium adsorption was noted. The FTIR results proved successful incorporation of SSGP in pretreated LS, and XRD results showed SSGP-pretreated LS is more crystalline and sturdier than untreated LS. SEM images showed that the chemical treatments greatly removed the impurities and waxes present in LS, that prevents the water adsorption capacity. The SEM-EDX results showed the efficiency of untreated and SSGP-pretreated LS as chromium adsorbent, the crystalline lattice structure of chromium was observed in LS. The ICP-OES analysis proved the chromium adsorption capacity of SSGP-pretreated LS, with 12.4 % further increase in adsorption noted. The SSGP-pretreated adsorbent shows good chromium adsorption about 39 mg/g, so it can be used as heavy metal sequestrant in heavy metal contaminated industrial discharges as well as sites that dump sludge with heavy metals. So, the SSGP-modified LS can be used as strong metals sequestrant. This study used simulated heavy metal for treatment, so in future research, the real time tannery effluent must be tested for observing the SSGP-LS efficiency in metal as well as other contaminants sequestration.

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#### **Author contribution**

The authors contribution to the paper as follows: study conception, design, data collection, analysis and interpretation of data, draft manuscript preparation: Santhiya Jayakumar; supervision: Dr K J Sharmila. All authors reviewed the results and approved the final version of the manuscript.

#### **Competing interest**

The authors, Mrs. Santhiya jayakumar, Dr K J Sharmila, declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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