



Development of Lignin – PVA Hydrogel and Its Application in Lead Removal for Waste Water Treatment.

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ABSTRACT:

Water is very essential part of life. Most form of life on earth depends on water directly or indirectly. Humans are almost >70 % water. Each individual consumes about 4 liters / day to continue its vital metabolic activities(Mitchell et al., 1945). Water is a vital natural resource that supports life on Earth, serving as a critical component in biological, chemical, and ecological processes. Covering approximately 71% of the Earth's surface, it exists in various forms, including liquid, solid, and vapor (Shiklomanov, 1993). Water plays a fundamental role in regulating Earth's climate by absorbing, storing, and redistributing heat (Intergovernmental Panel On Climate Change (Ippc), 2023). It is essential for human survival, with uses ranging from drinking and agriculture to industrial processes. The hydrological cycle maintains its continuous movement through evaporation, condensation, precipitation, and infiltration (Gleick, 1996). However, only 2.5% of Earth's water is freshwater, and less than 1% is accessible for human use, making conservation critical (Postel et al., 1996). Increasing demand, pollution, and climate change pose significant challenges to sustainable water management (Bates et al., 2008). Protecting water resources is vital for ecological balance and human development.

1). Introduction

Water is very essential part of life. Most form of life on earth depends on water directly or indirectly. Humans are almost >70 % water. Each individual consumes about 4 liters / day to continue its vital metabolic activities(Mitchell et al., 1945). Water is a vital natural resource that supports life on Earth, serving as a critical component in biological, chemical, and ecological processes. Covering approximately 71% of the Earth's surface, it exists in various forms, including liquid, solid, and vapor (Shiklomanov, 1993). Water plays a fundamental role in regulating Earth's climate by absorbing, storing, and redistributing heat (Intergovernmental Panel On Climate Change (Ippc), 2023). It is essential for human survival, with uses

ranging from drinking and agriculture to industrial processes. The hydrological cycle maintains its continuous movement through evaporation, condensation, precipitation, and infiltration (Gleick, 1996). However, only 2.5% of Earth's water is freshwater, and less than 1% is accessible for human use, making conservation critical (Postel et al., 1996). Increasing demand, pollution, and climate change pose significant challenges to sustainable water management (Bates et al., 2008). Protecting water resources is vital for ecological balance and human development.

Drinking water resources are essential for human survival, yet their availability and quality are increasingly under threat. Freshwater sources, such as rivers, lakes, and groundwater, are the primary suppliers



of potable water. Groundwater, which accounts for approximately 30% of the world's freshwater resources, is a critical source of drinking water globally (WWAP, 2018). However, overextraction, pollution, and saltwater intrusion have compromised its availability in many regions (Gleeson et al., 2012). Surface water, while accessible, is often contaminated with industrial, agricultural, and domestic pollutants, necessitating extensive treatment before it can be consumed (UNICEF, 2021).

In rural areas, unprotected water sources pose significant health risks due to microbial contamination, contributing to diseases such as diarrhea and cholera (Prüss-Ustün et al., 2019). Urbanization further strains drinking water systems, leading to disparities in water access, particularly in developing countries (Van Koppen et al., 2022). Climate change exacerbates these challenges by altering precipitation patterns, reducing water availability in some regions while increasing the risk of flooding and contamination in others (Mekonnen & Hoekstra, 2016). Advances in water treatment technologies, such as membrane filtration and UV disinfection, offer solutions for improving drinking water quality (Shannon et al., 2008). Additionally, integrated water resource management (IWRM) and sustainable practices are vital for ensuring equitable access to safe drinking water (Rasul & Sharma, 2016). Prioritizing investment in water infrastructure and policies that protect water resources is essential for achieving universal access to clean drinking water.

Heavy metals are naturally occurring elements present on earth's crust. These are present in the form of salt or ores and pose no such issues (Rahman & Singh, 2019). However, with industrial revolution, mining of heavy metal has introduced it into various resources that lead to their intrusion in living forms. They are of significant risk (Jarup, Lars, 2003; Martin & Johnson, 2012). Heavy metal polluted water bodies have deleterious effects on human life. Heavy metals are known to bind to biomolecules like DNA and protein, damaging the cell (Jan et al., 2015). Several steps have been taken up to remove heavy metal from water bodies but they either produce secondary pollutants or not cost effective (Briffa et al., 2020; Khulbe & Matsuura, 2018; Qasem et al., 2021).

Among several types of metals, lead (Pb) is a potential toxic for both animals and plants. Pb is present in earth's crust as 0.002%. Even at this low concentration it is the second most toxic material for all living beings. It is mostly produced by industries dealing with automobiles, batteries, paints and others. Lead comes in contact with humans through diet, paint, battery vents mining and others (Raj & Das, 2023; Sahoo et al., 2023; Tripathy et al., 2024). Lead is no new for humans as it is found in bones, teeth, brain, kidney, and liver at a total concentration of 10µg/dL in adults and slightly higher in children. At a concentration above it affects the functioning of kidney and brain. As it acts as an analog to calcium, it is absorbed by the bones and affects the reproductive organ in both male and female. At microscopic level it affects DNA transcription within a cell, thus altering its metabolic activities (Collin et al., 2022; Flora et al., 2006; Jiwani & S, 2011; Jusko et al., 2008; Yedjou et al., 2010). Therefore removal of Pb from water effluents from industries is a vital step towards remediation of Pb.

In view of it several works are continuously carried out to find effective method for the removal of Pb from environment. The removal of lead ions from water and wastewater is essential for ensuring safe and clean drinking water. Several effective methods are commonly employed for this purpose, including precipitation, coagulation-flocculation, ion exchange, adsorption, and membrane separation. While these methods are effective, they tend to produce significant quantities of sludge, which must be properly managed and disposed of to avoid additional environmental concerns. Other techniques such as ion exchange, adsorption, and membrane separation offer alternative approaches that can effectively reduce lead ion concentrations without the excessive sludge production associated with the earlier methods (Chowdhury et al., 2022; Crini & Lichtfouse, 2019). Among all these adsorption is the most efficient technique. As it requires less to no chemicals and is efficient economically. It is most preferred by developing nations. Though adsorption is mainly affected by temperature, pH, As concentration, adsorbent dose, and exposure time (Sarkar & Paul, 2016).

Hydrogels are one such adsorbing material. They are a network of polymers that have the capacity to absorb water through its hydrophilic groups. Hydrogels can be



synthesized from natural and synthetic polymers (Ahmed, 2015). Recently, industries have been widely using biopolymer based hydrogels for the removal of heavy metals from their effluents. Hydrogels adsorption have been used for the removal of iron, copper, cobalt, cadmium, arsenic, lead and other heavy metals. Biopolymers like cellulose, hemicellulose, alginate, chitin, starch and their derivatives have been vastly used for preparation of hydrogels for heavy metal removal (Pathan & Bose, 2018). Unlike other adsorbing materials hydrogels provide more binding sites per unit, which makes it efficient for waste water treatment. Hydrogels also adsorb heavy metal in high quantity due to their porous structure. The activity of hydrogels can be enhance by modifying them by addition of functional group (Darban et al., 2022). Biopolymers based hydrogels have recently attracted researchers to develop hydrogels for Pb removal. Biopolymers contains several functional groups that acts as natural magnets for attaching to heavy metals (Masoom et al., 2024).

Among all the natural biopolymers lignin is the only aromatic biopolymer that attracts researchers for developing functional hydrogel with wide applications in pharmaceuticals, environmental, bioremediation, and biotech industries (Meng et al., 2019). As compared to others, lignin increases mechanical property of hydrogel like rheological properties and tensile strength (Mushi et al., 2016; Teng et al., 2017). The lignin based hydrogels does not have a smooth surface but have high porosity at particular lignin concentration (Sun et al., 2016). Lignin based hydrogels have been widely reported for the adsorption of metal ions from water and waste water.

2). Material and method

2.1 Material

Lignosulfonate (CL), epichlorohydrin (ECH), NaOH, and Poly-vinyl alcohol (PVA) (MW= 6000 - 125000) were obtained from HiMedia. Nano organosolv lignin (NOSL) was prepared from rice stubble as described in our earlier work (Phillips et al., 2024). Lead acetate was obtained from Sun Chem for preparation of lead solution.

2.2 Preparation of PVA-LIG hydrogel

A 5% solution of PVA was prepared by dissolving it in distilled water at 80°C. The solution was continuously stirred until PVA completely dissolves. Solution was

cooled to room temperature and lignin was added and stirred continuously until homogeneity was attained. 0.15 ml of epichlorohydrin (crosslinker) was added to the solution and mixed well. The mixture was poured over a flat dish and kept at 70°C for 30 minutes for reaction to occur. Hydrogels were obtained and was washed with distilled water and kept in hot air oven at 50°C until constant weight. Four different concentration of lignin was used for hydrogel preparation viz, 1%, 3%, 5%, and 7%. Hydrogels were prepared from both NOSL lignin and commercial lignin (lignosulfonate).

2.3 Hydrogel characterization

The prepared hydrogel was characterized using Shimadzu IRAffinity 1 compact FTIR scanned in a range between 400 – 4000 cm⁻¹ with 50 scans in each scan with a resolution of 4 cm. FE-SEM images were developed using Carl Zeiss UHR FESEM model Gemini SEM 500 KMAT to understand the morphology of the hydrogel.

2.4 Swelling behavior

Swelling capacity of hydrogel was measured by immersing a known weight of lig-PVA hydrogel in 50 ml of distilled water until a constant weight of hydrogel is obtained. The initial weight (before swelling) and final weight of hydrogel after swelling was measured. The swelling capacity was calculated as follows:

$$S \% = \frac{W_{(f)} - W_{(i)}}{W_{(i)}} \times 100$$

Where S% is the swelling percent, W_(f) is final weight of hydrogel after complete swelling, W_(i) is the initial or dry weight of hydrogel. Effect of time on swelling capacity was also studied.

2.5 Removal of Pb⁺ from synthetic waste water

A synthetic Pb contaminated water solution was prepared using lead nitrate. The concentration of lead in water was 1000 ppb (1 ppm). A known weight of Lig-PVA hydrogel was immersed in lead solution for 24 hours in shaking incubator at room temperature. Agilent – 8900 ICP MS Tri Quad was used to estimate the amount of lead absorbed by the hydrogel. Effect of pH on lead absorption was also studied by UV – Visible spectroscopy. 1000 µl of PbNO₃ was taken and mixed with 1.5 ml of dithizone. To this mixture 1 ml of HCl was added and the volume was made up to 10 ml with 0.03



M SDS. Absorbance was measured at 500 nm in Shimadzu UV 1800 spectrophotometer.

3). Result

3.1 Hydrogel synthesis

The Lig-PVA hydrogels obtained were dark brown in color due to the presence of lignin. The color of hydrogel becomes darker with the increase of lignin concentration. The PVA concentration was kept constant at 5% because at lower concentration PVA does not form stable hydrogels. Beyond 5% concentration, hydrogels showed reduced water absorbability. At 5% concentration PVA shows maximum water uptake and forms a stable hydrogel. However, PVA only hydrogel was fragile in nature after water absorption, whereas the Lig-PVA hydrogel showed good mechanical strength. The hydrogel even at 1% lignin concentration, after water adsorption, hydrogel remained intact whereas PVA hydrogel at 5% PVA concentration was fragile after water absorption. All the hydrogels showed good mechanical strength in dried condition. The yield percentage of lignin was calculated using the following formula:

$Y\% = W_d/W * 100$ (where Y is yield, W_d is dry weight and W is total weight of lignin and PVA used). The yield percent calculated is displayed in the table 1.

S.No.	Lignin %	PVA %	Yield %	
			CL	NOSL

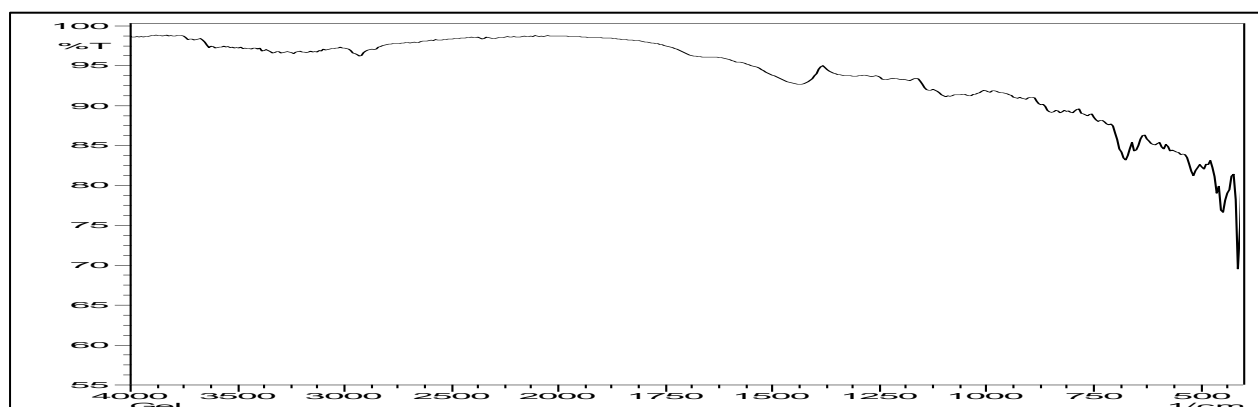
1.	1%	5%	68.56%	75.67%
2.	3%		61.77%	65.36%
3.	5%		54.23%	60.45%
4.	7%		50.33%	51.16%

Table 1: Yield of lignin – PVA hydrogel showing the effect of lignin concentration on hydrogel yield.

From the table 1 it is evident that as the lignin concentration increases the hydrogel yield decreases. This is due to leaching of lignin from hydrogel. This fact may be investigated to understand that with increasing lignin concentration, not enough crosslinking was done with PVA by ECH that produces unbound lignin. This trend is same in both NOSL and CL. However, yield is more with NOSL as compared to CL. This may be due to the presence of unmodified functional group on NOSL.

3.2 Characterization of hydrogel

Hydrogel was synthesized using PVA, lignin, and ECH. To understand the crosslinking between polymers via ECH, FTIR was carried out. OH tensile vibration and CH_2 bending was observed by intense peak at 3331 cm^{-1} . The characteristic peak confirming the hydrogel formation was peak observation at 1514 and 1588 cm^{-1} . The cross linking between lignin and PVA can be confirmed by peak at 1588 cm^{-1} . The peaks at 1060 cm^{-1} and 1082 cm^{-1} confirms C-O stretching showing the formation of ethereal bonds confirming that ECH caused crosslinking between the OH of lignin and PVA. These peaks confirm the successful synthesis of lignin-PVA hydrogel.



Graph 1: FTIR of lignin – PVA hydrogel

The FESEM images were developed of dried hydrogel to understand its morphology. The images shows a tightening structure which is densely meshed. This tightening can be attributed to the presence of lignin in hydrogel. Due to mesh like



appearance the hydrogel shows great porosity that affects the high absorbing nature of our hydrogel. These characterizations confirms the development of crosslinked lignin-PVA hydrogel with high porosity.

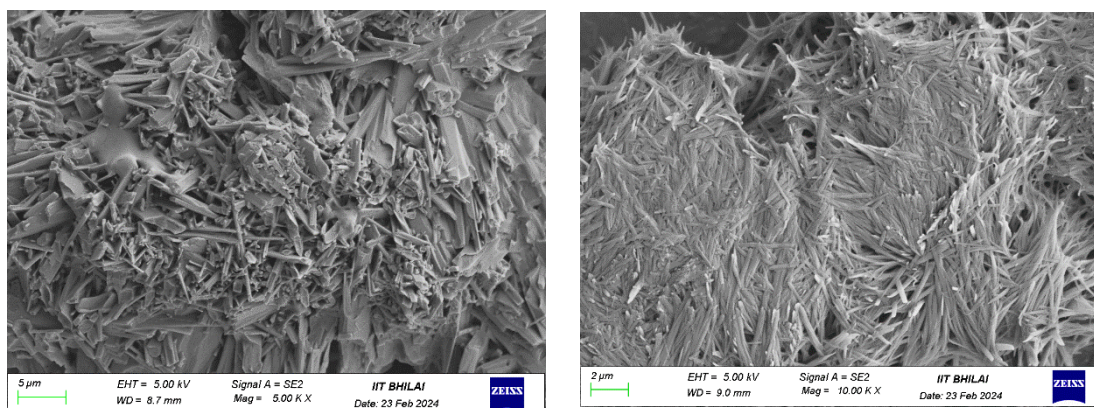
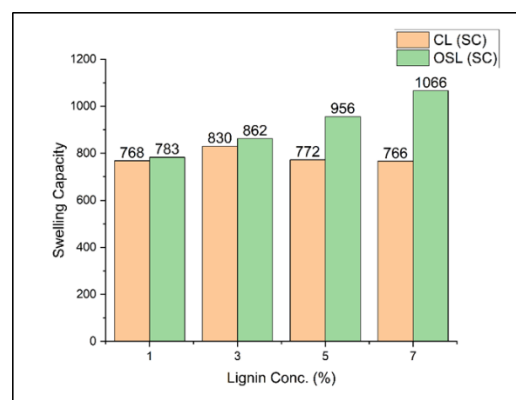


Fig 1: FE-SEM Image of Hydrogel prepared from NOSL – PVA Hydrogel

3.3 Swelling behavior

Hydrogels were prepared having 5% concentration of PVA and 0.15 ml of ECH. Four different concentration of lignin was added to understand the effect of lignin in swelling capacity of hydrogel. The prepared hydrogels were immersed in distilled water having 7pH. Effect of lignin on swelling capacity can be seen in graph 2. The least swelling of 768% and 783% was found at 1% lignin concentration for both CL and NOSL respectively. In case of CL-PVA hydrogel, the highest swelling capacity was 830% at 3% lignin concentration. For NOSL-PVA hydrogel, a swelling capacity of 1066% was attained at 7% concentration. At 3%, NOSL-PVA showed 862%, slightly above CL-PVA swelling capacity. At 5% lignin concentration, NOSL-PVA showed 956% swelling capacity, whereas CL-PVA hydrogel swelling capacity was 772%. A decrease in swelling capacity of CL-PVA hydrogel can be seen beyond 3% lignin concentration. Whereas in case of NOSL-PVA hydrogel, with increasing concentration of lignin, swelling capacity also increased. From this result we conclude that CL-PVA hydrogel showed a linear behavior in water absorption suggesting no effect of lignin in swelling capacity. NOSL lignin on the other hand remains unmodified therefore has a good concentration of OH group in its structure. These OH groups may be available in hydrogel, thus enhancing swelling capacity. FESEM images also indicated high porosity, thus supporting high absorption capacity. Therefore the hydrogels prepared by the above mentioned method shows supra-absorbability.



Graph 2: Bar graph showing the effect of lignin concentration on the swelling capacity of CL-PVA and NOSL-PVA hydrogels

3.4 Effect of pH on swelling capacity

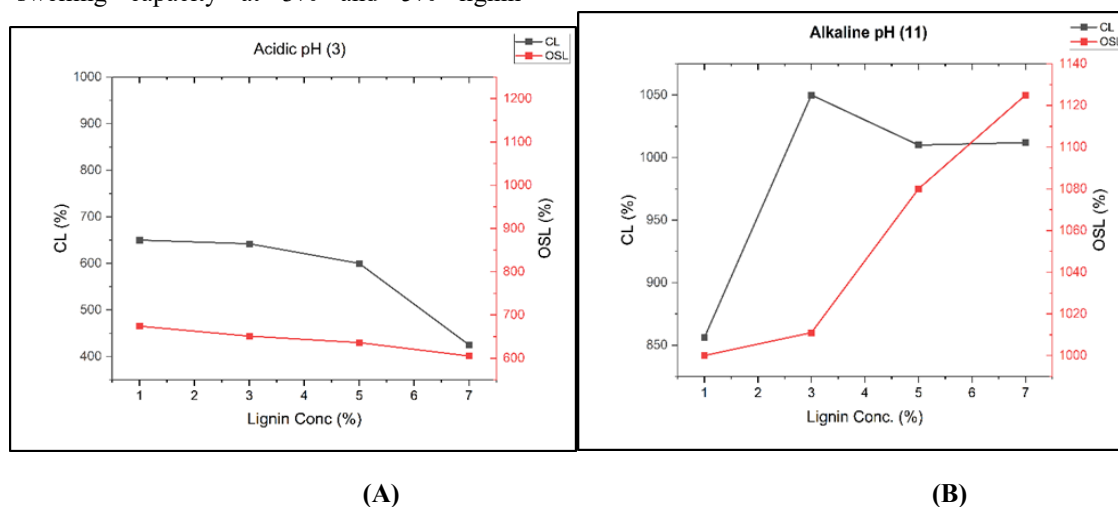
To study the effect of alkalinity and acidity on hydrogels swelling behavior, hydrogels were immersed in water having pH 3 (for effect of acidity) and 11 (for effect of alkalinity). In acidic condition CL-PVA hydrogel at 3% concentration showed swelling capacity of 942%. Looking at its trend CL-PVA hydrogel swelling capacity decreases at 5% and 7% lignin concentration. As evident from earlier swelling capacity in CL-PVA hydrogel, the swelling behavior was dependent on PVA and CL-lignin pose no significant effect on water uptake. Studies have indicated that the functional groups like -COOH present in PVA remains in protonated condition at lower pH thus



cannot bind effectively with water molecules (Khoerunnisa et al., 2021; Taleb et al., 2009). Also, as lignin concentration increases, number of PVA molecules in hydrogel matrix decreases, hence an overall reduced swelling capacity can be observed in CL-PVA hydrogel.

In contrast, NOSL-PVA hydrogel shows 1011% and 1175% swelling capacity at 3% and 5% lignin

concentration. However, this trend is absent at 7% lignin concentration. The condition of protonation of NOSL-lignin is same as of CL, therefore the swelling capacity is not significantly affected by lignin concentration (Avasi & Chattopadhyay, 2019; Kim et al., 2024). Moreover, a stable absorption capacity unaffected by lignin can be observed. However, NOSL-PVA as compared to CL-PVA showed better swelling capacity.



Graph 3: Line graphs showing the effect of acidic and alkaline pH on hydrogel swelling capacity. Graph A and B shows the effect of acidic condition and alkaline condition on swelling capacity

3.5). Heavy metal removal

Hydrogel was tested for the removal of Pb from synthetic water. Hydrogels were immersed in synthetic waste water and Pb adsorption was measured using ICPMS. The best absorbing CL-PVA and OSL-PVA hydrogel was used for this assessment. As evident 7% NOSL-PVA hydrogel and 5% CL-PVA hydrogel having maximum absorption capacity was taken for study. As more absorption will cause more intake of Pb ions. From table 2, it is clear that NOSL-PVA hydrogel has more Pb adsorption from synthetic waste water as compared to CL-PVA hydrogel. The initial concentration of Pb in water was 1000 ppm or 1 ppb. NOSL-PVA treated water showed 72.5% removal of Pb ions, as the concentration of Pb ions decreased to 275 ppm.

Lignin Type	Concentration	Initial Pb Conc.	Pb conc. after hydrogel treatment
CL	5%	1000 ppm	513 ppm
NOSL	7%	1000 ppm	275 ppm

	(in ppm)	(in ppm)
CL	5%	513 ppm
NOSL	7%	275 ppm

Table 2: Hydrogel prepared from CL and OSL shows difference in heavy metal adsorption capacity.

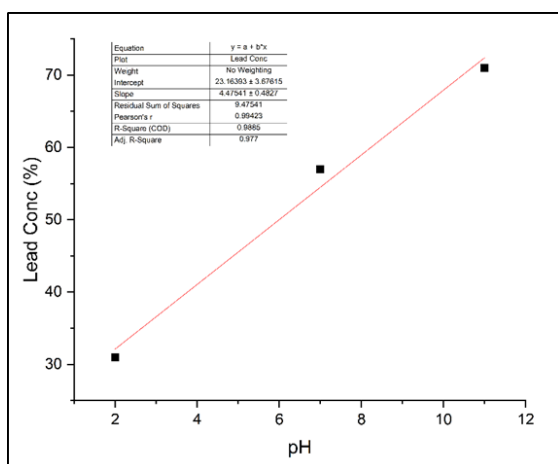
On the other hand, CL-PVA hydrogel could remove only 48.7% of Pb ions, that is the Pb ion concentration in water after hydrogel treatment was measured to be 513 ppm. High removal of Pb by NOSL-PVA hydrogel can be understood by the fact that it contains high amount of phenolic group that can act as a chelating agent that form stable complexes with Pb ions (Du et al., 2023, 2024). Also high porous nature of NOSL-PVA hydrogel allows excess seepage of Pb ions in hydrogel structure and thus giving room for making complexes with lignin structure



(Nair et al., 2023). CL-PVA though have sulfonate groups that could effectively bind to Pb ions but found to be less efficient than NOSL-PVA hydrogel.

3.6). Effect of pH on heavy metal removal

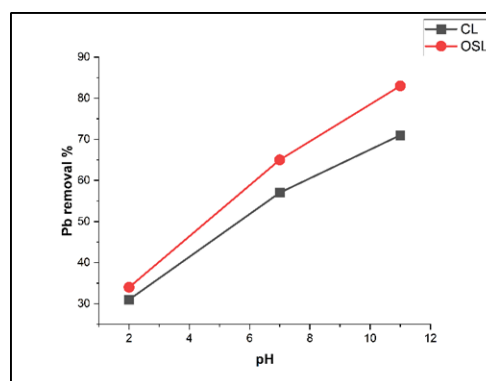
To study the effect of pH on hydrogel for lead adsorption was studied by UV-visible spectrophotometer. A graph was plot between pH versus lead ion adsorption. From graph 4 it is evident that as pH increases the adsorption of Pb ions also increases. pH invariably affects the adsorption of Pb ions. At lower pH (acidic condition), the concentration of hydrogen ion is enough to compete with binding sites present on the hydrogel. Therefore, less amount of Pb ions can bind to hydrogel. Increasing the pH from pH 2, the concentration of H⁺ ions decreases and hence the binding sites for Pb ions are not competitively saturated. At pH <7, less than 50% of Pb ions were adsorbed. On the other hand increasing pH up to 7, hydrogel decreased more than half concentration of Pb ions in water. This trend continues as pH goes beyond 7 and more binding sites are available for Pb ion binding. The removal capacity for hydrogel increases to >70% at alkaline pH. Therefore, through our studies we conclude that the heavy metal removal capacity of hydrogel is pH dependent. Hence it is suggested to use hydrogel for effluent treatment in alkaline condition for effective removal of heavy metals.



Graph 4: Effect of pH on Pb ion removal by hydrogel.

Now if we compare Pb ion adsorption capacity of CL-PVA and NOSL-PVA hydrogel, the later shows better adsorption capacity. From graph 5, it was observed that in alkaline condition OSL-PVA hydrogel can remove

>80% of Pb ions from water, whereas CL-PVA hydrogel can remove upto 70% of Pb ions. This may be due to the presence of unmodified functional groups on NOSL, that provides efficiency to hydrogel for Pb⁺ removal. Therefore it can be suggested that NOSL based hydrogels are efficient adsorber of heavy metal.



Graph 5: Effect of pH on lead ion removal from water by NOSL-PVA and CL-PVA hydrogel.

4). Conclusion

In conclusion, the study on lignin-PVA hydrogels highlights their significant potential for effectively addressing lead contamination in wastewater. The addition of lignin, a natural biopolymer, enhances the hydrogel's ability to adsorb lead ions due to its abundant functional groups that form stable complexes with these ions, establishing them as an environmentally friendly alternative for heavy metal remediation. This research emphasizes the urgent need for sustainable methods to combat water pollution, especially since heavy metals pose serious risks to public health. The use of biopolymer-based hydrogels not only underlines the importance of renewable resources in environmental applications but also offers a practical solution for improving water quality. Future research should focus on optimizing hydrogel formulations and exploring their potential to remove various contaminants, which could lead to broader implications for water treatment technologies and environmental sustainability.

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