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APPLICATION OF CRAB SHELLS (*Portunus pelagicus Linn.*) AS RAW MATERIAL FOR SELECTIVE ADSORBENT OF Cu(II) AND Cd(II) IN SEAWATER

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ABSTRACT

Crab shell (*Portunus pelagicus Linn*.) waste, a by-product of the seafood industry, was utilised as a raw material to produce chitosan and its derivatives for selective adsorption of Cu(II) and Cd(II) in seawater. The preparation process involved deproteinization, demineralisation, and deacetylation, followed by structural modifications to obtain beads, phosphate, thiocyanate, molybdate, sulfate, glutaraldehyde-crosslinked, and thiourea chitosan. Characterisation using FTIR and XRD confirmed functional group transformation and crystallinity reduction associated with deacetylation and chemical modifications. The degree of deacetylation (DD) was significantly influenced by NaOH concentration, temperature, reflux time, and HCl concentration, with values reaching up to 96%. Adsorption studies revealed that higher DD correlated with improved Cu(II) binding, particularly at pH 5–6. Chitosan beads exhibited enhanced swelling and nearly doubled Cu(II) adsorption compared to powder. Among the derivatives, glutaraldehyde-crosslinked and thiourea-modified chitosan showed the highest adsorption efficiencies (60.7% and 69.5%, respectively), indicating strong interactions with metal ions. These findings highlight the potential of crab shell waste as a sustainable source of selective adsorbents for heavy metal removal in marine environments.

Keywords: Cd(II) Adsorption; Chitosan; Crab Shell; Cu(II) Adsorption; Seawater Treatment; Solid-Phase Extraction

RASĀYAN *J. Chem.*, Vol. 19, No.1 2026

INTRODUCTION

Indonesia, with the world's second-longest coastline, has great potential to benefit from marine resources.[1,2] Besides fish, crab (*Portunus pelagicus Linn.*) is an important protein source, but its growing consumption leaves behind large amounts of shell waste, up to 52.59% of total biomass.[3] This waste is usually discarded, harming the environment while neglecting its potential value. Crab shells, which make up 20–30% of dry weight, contain chitin and chitosan, two biopolymers with wide applications in industries such as pharmaceuticals, agriculture, food, wastewater treatment, and textiles.[4] In Japan alone, about 700 tons of shells are processed annually, with 500 tons used as flocculants in wastewater treatment and the rest for cosmetics, food, and other applications.[5] Chitin has acetamide (-$NHCOCH_3$) groups that can bind heavy metals, and under alkaline treatment, it converts into chitosan, exposing amino (-NH_2) groups that act as stronger donors. This makes chitosan more effective as an adsorbent for metals like Cu(II), Ni(II), Cd(II), Zn(II), Pb(II), and Cr(III).[6-8] The degree of deacetylation (DD) is key: higher DD means more amino groups, thus higher adsorption capacity, though conventional methods usually yield only 70–80%.[9]

Optimising NaOH concentration, temperature, and reflux time can raise DD up to 85–90%.[10] Chitosan has also been developed into gels, membranes, and beads that improve swelling ability and surface area, achieving >90% removal of Pb, Cr, and As in certain forms.[11]Chemical modifications—such as grafting with PEI/PVA, sulfonation, or cross-linking-further enhance adsorption, with capacities reported up to 445.29 mg/g for Cr(VI) and 211–357 mg/g for tetracycline.[12] Functional group modifications also align with Pearson's HSAB theory, where thiol-modified chitosan effectively adsorbs soft acid metals like Hg(II) (833.33 mg/g), while amino-modified chitosan performs better with hard acid metals like Cu(II) (348 mg/g).[13] Additional groups, such as sulfate or humate, introduced by immobilisation, hybridisation, or impregnation, further expand removal efficiency across multiple metals, including Cu(II), Ni(II), Cd(II), Zn(II), Pb(II), and Cr(III).[14,15] Still, conventional production methods remain resource-intensive, prompting the need for simplified approaches such as skipping deproteinization. This study, therefore, focuses on optimising production parameters and chemical modifications to transform crab shell waste into efficient, selective adsorbents for environmental remediation.

EXPERIMENTAL

Materials and Equipment

Crab shells were used as the raw material, while NaOH, HCl, acetic acid, glutaraldehyde, thiourea, thiocyanate, ammonium phosphate, ammonium molybdate, and ammonium sulfate were used as reagents. Instruments included FTIR, XRD, UV-Vis spectrophotometer, AAS, pH meter, drying oven, hotplate stirrer, and orbital shaker.

Conversion of Chitin to Chitosan

Crab shells were washed, dried, and ground to 100 mesh before undergoing deproteinization (3.5% NaOH, 65℃, 2 h), demineralisation (1 M HCl, 30 min, room temperature), and deacetylation (50% NaOH, 100℃, 30 min). Process order variations and HCl concentrations (0.2–1 M) were tested, followed by optimisation of deacetylation using NaOH (50–70%) at 100–120℃ for durations ranging from 30 to 1380 min.[16]

Conversion of Chitosan into Derivatives

Chitosan was modified into several derivatives, including beads (via NaOH precipitation), phosphate, thiocyanate, molybdate, sulfate, glutaraldehyde-crosslinked, and thiourea forms. Each product was obtained as powder or beads and subjected to further evaluation.[17]

Characterisation of Chitin, Chitosan, and Derivatives

All products were analysed by FTIR to determine functional groups and degree of deacetylation (DD), and by XRD to evaluate crystallinity. Post-adsorption characterisation was also carried out to confirm metal–adsorbent interactions.

Adsorption and Solid Phase Extraction Tests

Adsorption studies were conducted using Cu(II) and Cd(II) solutions under controlled conditions. Experimental parameters included contact time (0–1380 min), pH (2–10), and metal ion concentration (50–1000 ppm).[18] Adsorption efficiency was determined by measuring concentration changes using UV-Vis and AAS. Kinetics, isotherms (Langmuir and Freundlich), selectivity, and mechanism analyses (entrapment, ion exchange, hydrogen bonding, and complexation) were performed to evaluate the adsorption behaviour.

RESULTS AND DISCUSSION

Transformation of Chitin into Chitosan

The preparation of chitosan from crab shells involved sequential deproteinization, demineralization, and deacetylation, which progressively removed proteins, minerals, and acetyl groups. IR spectra confirmed structural changes, including the weakening of carbonyl bands and persistence of carbonate and silicon impurities, while XRD patterns indicated higher crystallinity in demineralized shells compared to deacetylated ones, reflecting successful conversion steps.[19,20] The degree of deacetylation (DD), summarised in Table-1, increased after NaOH treatment, reaching values above 70%, confirming chitosan

formation.[21,22] Nitrogen content also rose, consistent with greater exposure of amine groups after acetyl removal. Factors such as treatment sequence, HCl concentration, NaOH strength, temperature, and reflux time strongly influenced DD, with optimal conditions producing DD values up to 96%, as verified by IR band intensity changes.[23-25] Adsorption studies revealed that higher DD correlates with improved Cu(II) uptake, as more $–NH_2$ groups enhance chelation. For instance, adsorption increased from 11.4% at DD 47% to 56.3% at DD 96%, confirming DD as a critical parameter for performance.[26] Additionally, solution pH influenced adsorption behaviour, with maximum uptake observed at pH 5–6, where reduced protonation of amine groups favours metal binding.[27]

Table-1: DD Value of Crab Shell Before and After Preparation

Formula	Degree of Deacetylation (%)		
	Crab Shell	Demineralized Crab Shell	Deacetylated Crab Shell
1	53.31	53.13	70.00
2	81.08	70.63	82.63
3	28.70	68.88	57.56
4	71.15	53.03	72.62
5	46.07	52.03	68.57
6	78.16	69.30	79.10
7	17.60	31.12	51.82
8	66.68	53.03	67.80

Conversion of Chitosan into Chitosan Derivatives and Their Adsorption Properties

Conversion of chitosan into beads and derivatives caused notable structural changes confirmed by IR spectra, with decreased intensity of amide and amine bands and the appearance of new functional groups (Fig.-1). These modifications enhanced adsorption, where beads (Kb) showed nearly double the Cu(II) uptake compared to powder (Ks).[28] pH also influenced adsorption behavior, with low pH reducing metal binding due to amine protonation.[29,30]

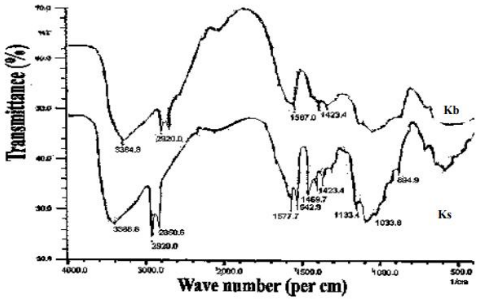

Fig.-1: IR Spectra of Ks and Kb Adsorbents

Further derivatisation, including phosphate, molybdate, and thiocyanate groups, altered crystallinity and introduced new sites for coordination. As shown in Table-2, derivatives like KMb and KFb demonstrated higher Cu(II) adsorption than Ks, while some reduced Cr(VI) uptake. IR shifts after adsorption confirmed interactions between metal ions and both native $–NH_2$ and newly formed groups.[31]

Table-2: Adsorption of Cu(II) and Cr(VI) by Various Chitosan-Derived Adsorbents

Adsorbent	Adsorption Capacity (%)	
	Cu(II)	Cr(IV)
KFb	57.60	0.00
KFs	49.20	0.00
KTn	33.30	19.20
KMb	60.00	10.58
KMs	48.90	0.00
Ks	32.00	18.52

Covalently crosslinked derivatives such as KGb and KTb achieved the best performance, with Cu(II)

adsorption up to 69.5% (Table-3). XRD patterns indicated amorphisation in these structures, reflecting increased active site accessibility. Adsorption was also pH-dependent, with Cu(II) showing stronger affinity than Cd(II) at pH 4–6, consistent with HSAB principles and functional group contributions.[32,33]

Table-3: Adsorption of Cu(II) by KSb, KGb, and KTb Adsorbents

Adsorbent	Adsorption Capacity of Cu(II) (%)
KSb	39.90
KGb	60.70
KTb	69.50
Ks	32.00

Adsorption Characteristics of Chitosan and Its Derivatives toward Heavy Metals

The adsorption of metal ions on chitosan-based adsorbents (Ks, Kb, KFs, KFb, KMb, KGb, and KTb) showed a similar kinetic profile: rapid uptake in the early stage due to abundant active sites, followed by slower adsorption until equilibrium was reached (Fig.-2). This behaviour highlights the significance of surface functionalization in improving adsorption efficiency and suggests that the process can be well-described by pseudo-first-order and pseudo-second-order models.

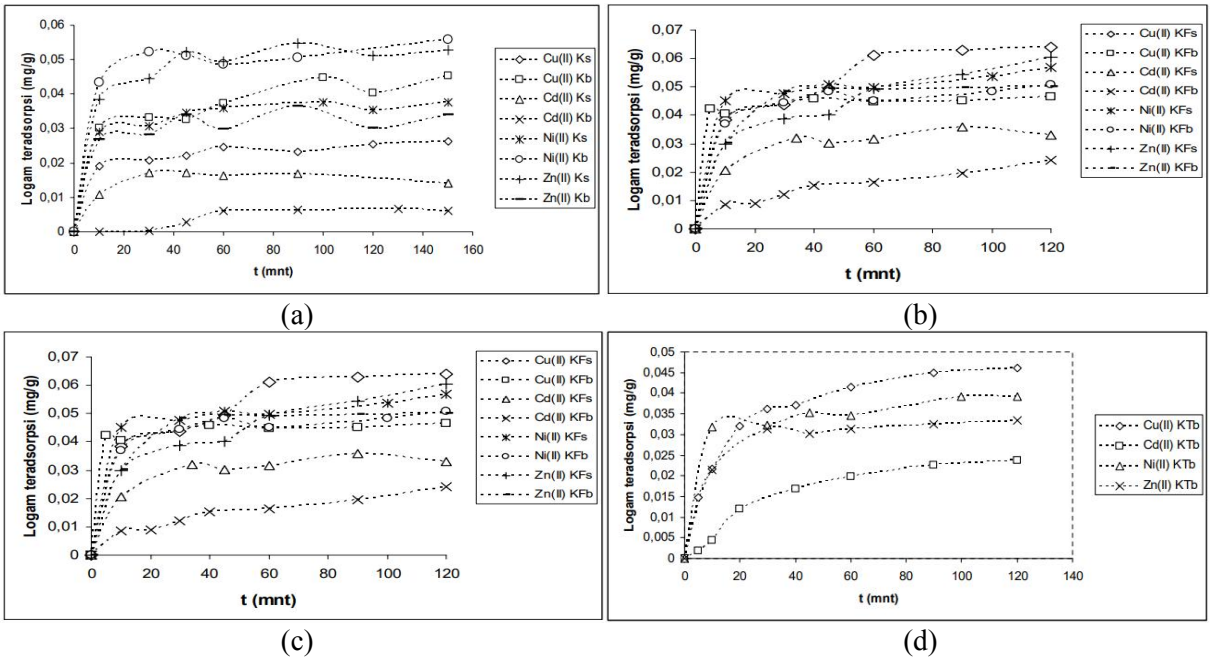

Fig.-2: Effect of Contact Time on Metal Ion Adsorption: (a) Adsorption on Ks and Kb; (b) Adsorption on KFs and KFb; (c) Adsorption on KMb and KGb; (d) Adsorption on KTb

Variation in initial metal concentration revealed that adsorption capacity increased with concentration until reaching saturation, consistent with the Langmuir isotherm. Cu(II) on Ks and Kb, and Cd(II) on KFs, showed clear monolayer adsorption, whereas other systems suggested multilayer adsorption, aligning with the Freundlich model. Selectivity patterns were further explained by the Hard-Soft Acid-Base (HSAB) principle: Cd(II), a soft acid, preferred sulfur-containing sites in KTb, while Cu(II), a borderline acid, interacted more strongly with harder base groups (Fig.-3).

Environmental conditions also played an important role. Chitosan derivatives maintained effective Cu(II) and Cd(II) adsorption across a wide pH range, with surface conditioning ensuring compatibility with metal ions (Fig.-4).[34] Desorption studies confirmed that hydrogen bonding dominated the mechanism, as HCl efficiently released Cu(II) (>99% for KFb and KTb), while Na-EDTA was less effective. Modified adsorbents such as KFs, KFb, and KTb demonstrated stronger desorption and higher stability than parent chitosan, while KGb showed enhanced complexation due to its crosslinked structure.[14,35] In both single- and multi-metal systems, Cu(II) adsorption remained highly selective, reinforcing hydrogen bonding as the dominant adsorption mechanism (Fig.-5).

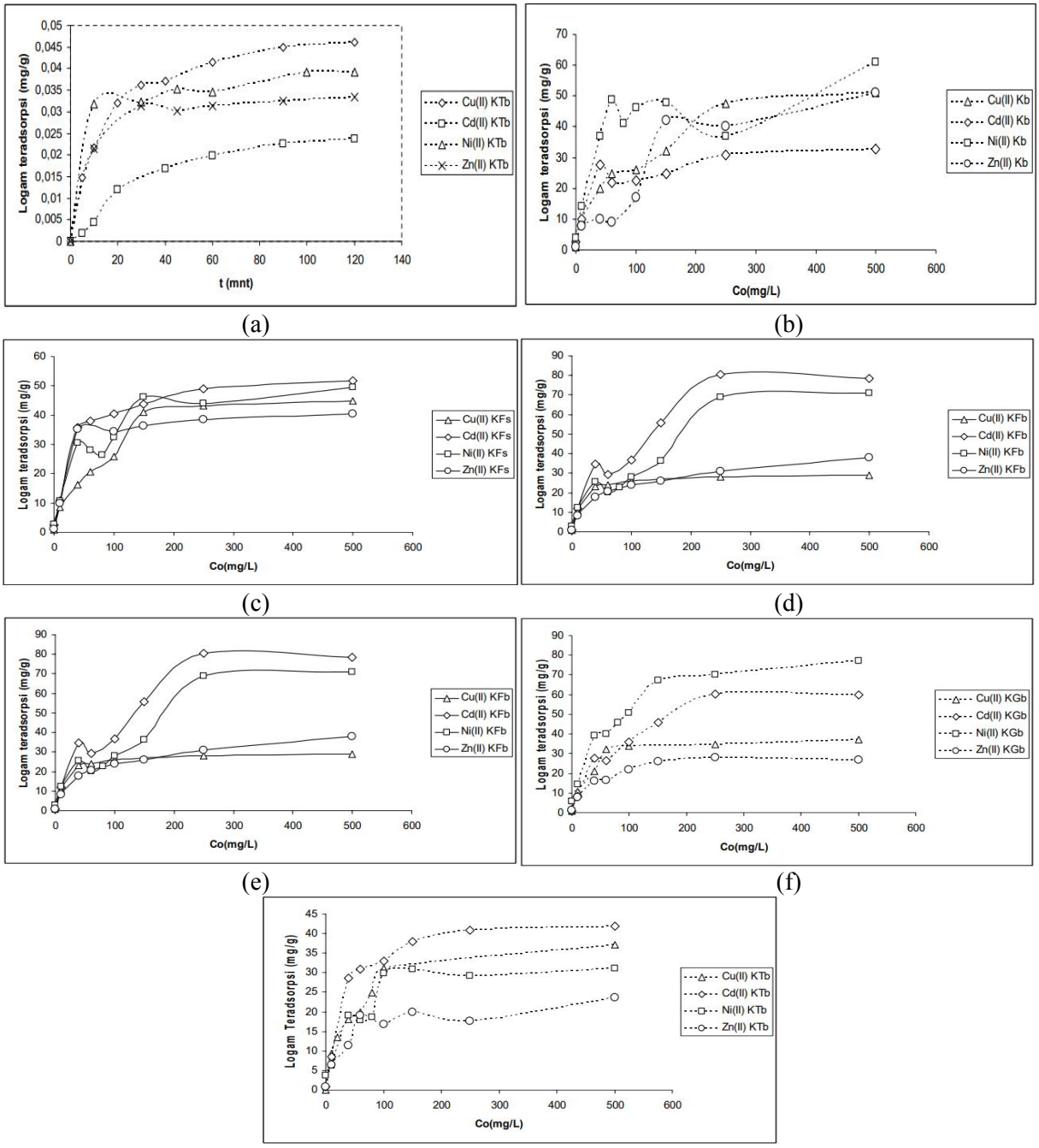

Fig.-3: Effect of Initial Concentration on Cu(II), Cd(II), Ni(II), and Zn(II) Adsorption by: (a) Ks; (b) Kb; (c) KFs; (d) KFb; (e) KMb; (f) KGb; (g) KTb

Solid Phase Extraction (SPE) Using Chitosan and Its Derivatives

The effect of flow rate on Cu(II) adsorption using SPE (Fig.-6(a)) showed that chitosan derivatives (KFb, KTb, KGb) achieved optimal adsorption efficiency at 0.5 mL/min, while native chitosan (Ks) performed less effectively. This improvement is attributed to newly formed active sites that have a stronger affinity compared to the original ones.[36] In the desorption step, 0.5 M HCl provided effective recovery of Cu(II) (Fig.-6(b)), while higher concentrations produced abnormal values (>100%) due to adsorbent degradation and contaminant release, which interfered with measurement accuracy.

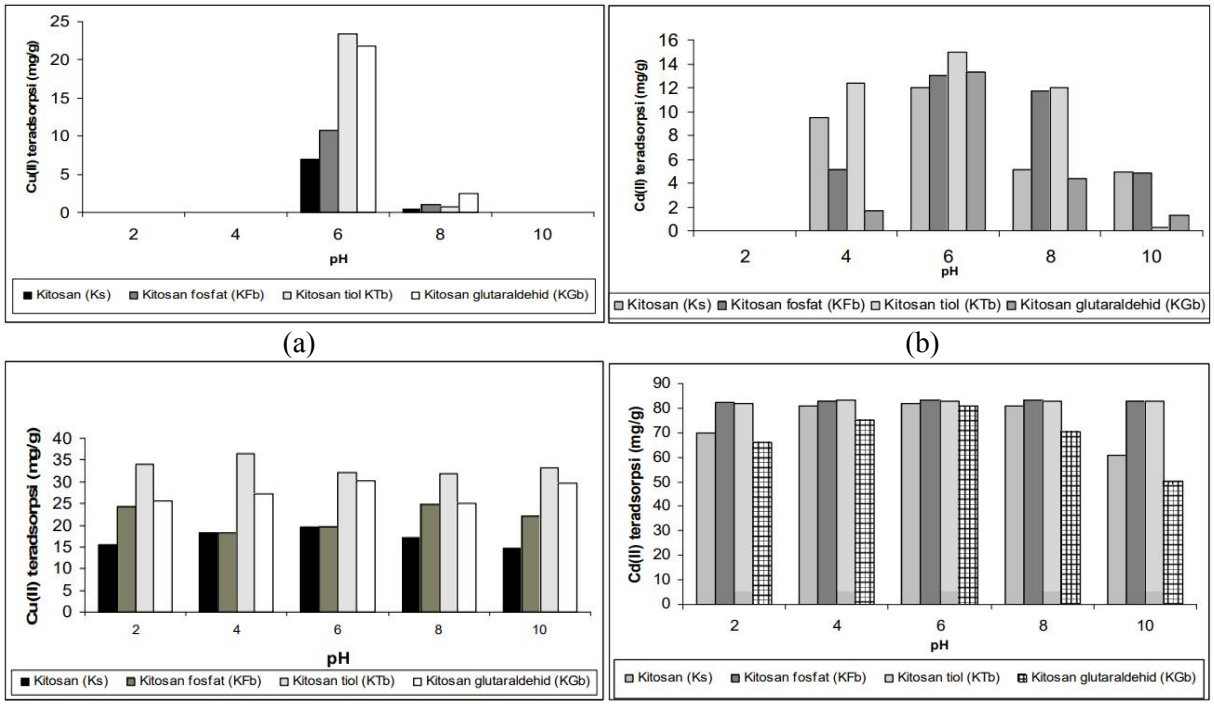

Fig.-4: Adsorption Performance of Chitosan and its Derivatives after Pretreatment at different Immersion Ph Conditions (pH 2–10): (a) Cu(II) Adsorption at Equilibrium pH 2–10; (b) Cd(II) Adsorption at Equilibrium pH 2–10; (c) Cu(II) Adsorption at Equilibrium pH 6; (d) Cd(II) Adsorption at Equilibrium pH 6

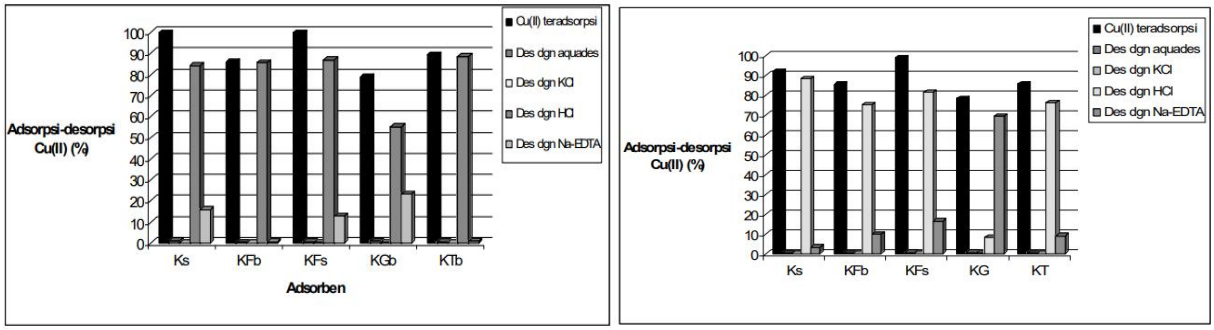

Fig.-5: Adsorption Mechanisms of Cu(II): (a) in a Single-Metal System; (b) in a Multi-Metal System Containing Cu, Cd, Ni, and Zn.

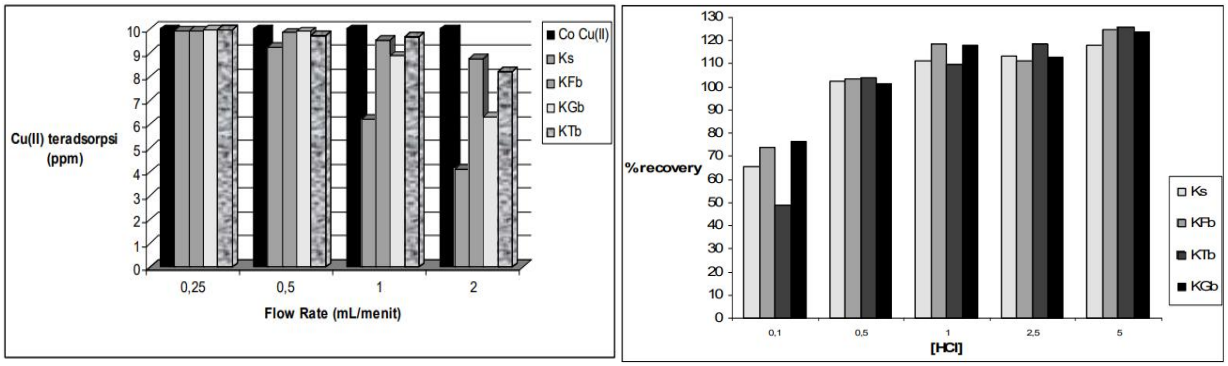

Fig.-6: (a) Effect of Flow Rate on Cu(II) Adsorption Using Chitosan-Based Adsorbents; (b) Effect of HCl Concentration on % Recovery of Cu(II) using the SPE Technique

Further testing demonstrated that chitosan-based adsorbents maintained selectivity toward Cu(II) even in saline and artificial seawater conditions, indicating their suitability for marine applications. The presence of Na(I) or other major seawater cations did not significantly reduce Cu(II) recovery, confirming that competition effects were minimal. These findings support previous reports showing the ability of chitosan materials to adsorb Cu(II) in seawater with high selectivity.[37] In mixed-metal systems, Cu(II) and Cd(II) were more effectively adsorbed compared to Ni(II) and Zn(II), which was influenced by ion size and electronegativity differences.[38,39] Preconcentration studies (Fig.-7) further highlighted that both Cu(II) and Cd(II) could be enriched from ppb to ppm levels, with chitosan derivatives consistently outperforming native chitosan.[40] This confirms the critical role of modified active sites in enhancing adsorption, desorption, and preconcentration efficiency, especially for detecting trace heavy metals in seawater.

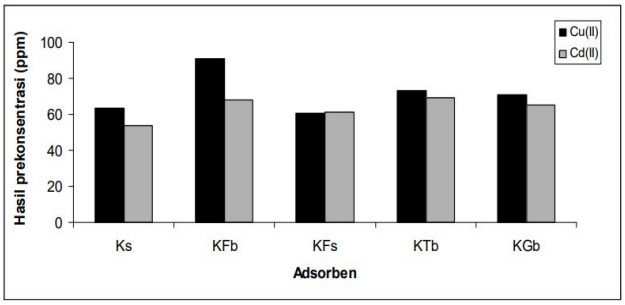

Fig.-7: Preconcentration Results of Cu(II) and Cd(II) from an Initial Concentration of 1 ppb

CONCLUSION

This study confirms that crab shell waste from Portunus pelagicus Linn. can be effectively converted into chitosan and its derivatives as selective adsorbents for heavy metal removal. Optimization of deacetylation produced a high degree of deacetylation (up to 96%) under NaOH 60% at 120 °C for 6 h, which significantly enhanced the adsorption capacity. Chitosan beads (Ks) showed almost 2-fold higher Cu(II) adsorption (from 28.4% to 55.6%) compared to chitosan powder, due to their higher swelling capacity. Among the derivatives, glutaraldehyde-crosslinked chitosan (KGb) and thiourea-modified chitosan (KTb) exhibited the best performance, with Cu(II) adsorption reaching 60.7% and 69.5%, respectively. Adsorption was strongly pH-dependent, with maximum uptake observed at pH 5–6, and competitive adsorption tests showed that Cu(II) was preferentially adsorbed over Cd(II), Ni(II), and Zn(II). These findings demonstrate that crab shell waste is not only a sustainable raw material but also provides highly efficient adsorbents for heavy metal remediation in marine environments.

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CONFLICT OF INTERESTS

The authors declare that there is no conflict of interest.

AUTHOR CONTRIBUTIONS

The present work is related to *SDG-6: Clean Water and Sanitation*. All the authors contributed significantly to this manuscript, participated in reviewing/editing and approved the final draft for publication. The research profile of the authors can be verified from their ORCID ids, given below:

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[RJC-9586/2025]