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# **CHAPTER-7**

## **Summary and Conclusions**

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## CHAPTER-1: GENERAL INTRODUCTION

In this chapter, we present a comprehensive overview of our research project, which focuses on the mitigation and quantification of toxic heavy metal impurities in both pharmaceuticals and food products. We underscore the critical importance of addressing toxic heavy metal contamination in these indispensable commodities, specifically in the context of medicines such as Losartan and Cholic acid, as well as food items originating from the North Gujarat region, including potatoes and coffee. One of the central features of our research is the innovative utilization of synthesized SNPs as adsorbents, a novel approach that sets our work apart from previous studies. This chapter serves as the foundation for the ensuing chapters, underscoring the significance and novelty of our research. Subsequent chapters will delve into the synthesis and characterization of various types of SNPs intended for use as adsorbents in the removal of heavy metals. We will elaborate on the methodologies employed for synthesizing SNPs with diverse properties and surface functionalities. Furthermore, we will present in-depth characterization techniques, such as SEM, TEM, XRD, and FTIR, which are instrumental in assessing the structural and surface attributes of the synthesized SNPs. This chapter lays a robust foundation for the forthcoming chapters, assuring the quality and suitability of the adsorbents for the remediation of toxic heavy metals.

Our initial two chapters are dedicated to the precise quantification of toxic heavy metal impurities in pharmaceuticals, with a specific focus on Losartan and Cholic acid. We will elucidate the analytical techniques employed, including ICP-MS, for the accurate measurement and quantification of toxic heavy metal concentrations. These chapters will also encompass a comparative analysis of toxic heavy metal contents in various samples, shedding light on the potential health risks associated with these impurities. In our subsequent two chapters, we will investigate the effectiveness of various types of synthesized SNPs as adsorbents for the removal of toxic heavy metals from food products. Our specific focus will be on the application of SNPs in the purification of potatoes from the North Gujarat region and coffee samples. We will rigorously evaluate the adsorption capacity, efficiency, and selectivity of these SNPs in the context of toxic heavy metals removal. These chapters will offer valuable insights into the distinctive capacity of SNPs to diminish toxic heavy metal impurities in food products, thus ensuring consumer safety and product quality.

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In addition, we will put forth recommendations for future research directions to further enhance the utilization of synthesized SNPs as adsorbents and to broaden their application in diverse realms of toxic heavy metal remediation

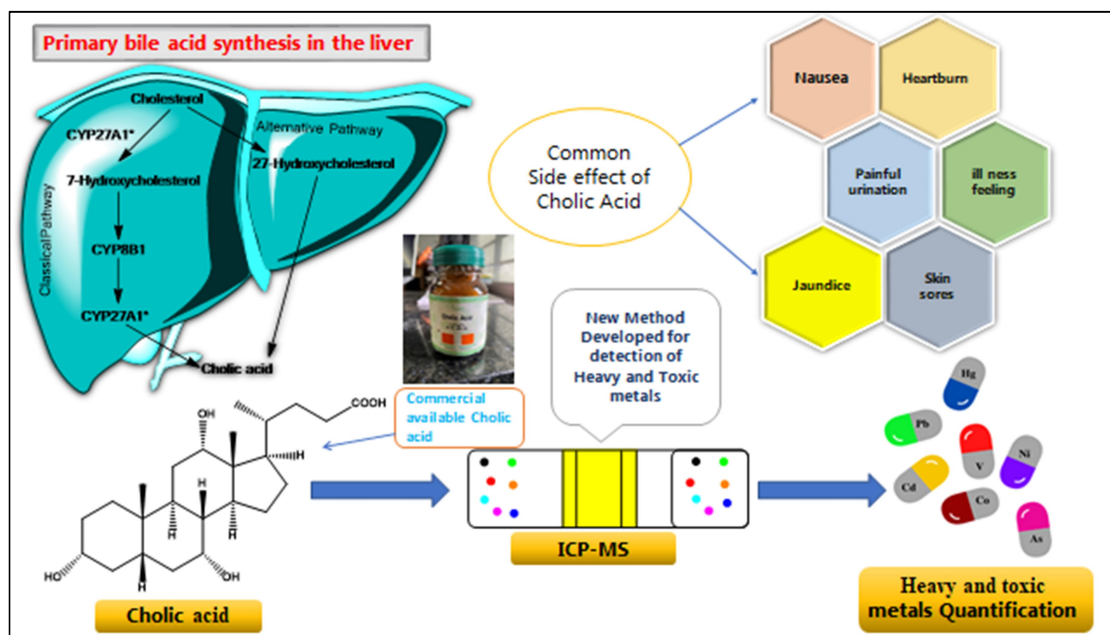
## **CHAPTER-2: MATERIALS, EXPERIMENTAL DESIGN AND CHARACTERIZATION METHODS**

This thesis revolves around a critical theme: the removal of toxic heavy metal contaminants from pharmaceutical and food products using various types of RH. The study employed RH sourced from the 'GNR-3 (Gujarat Navsari Rice – 3)' rice variety, which was obtained from Navsari Agricultural University, Navsari. Coffee samples were sourced from the Kerala region, specifically the "Robusta" variety. Analytical reagent-grade HCl with a concentration of 35-38% was provided by SDFCL, and deionized water was consistently used throughout the experiments. This chapter provides a comprehensive overview of the primary characterization techniques that were employed, including TGA, FT IR, SEM, EDX, DLS, TEM, AFM, XRD, and ICP-MS. It delves into the experimental design used in the research, along with the underlying theoretical framework. Furthermore, the chapter elucidates the various methods and procedures implemented during the study, substantiated by a detailed evaluation of pertinent data parameters. By illuminating these methodologies, this research aims to contribute valuable insights into effective strategies for eliminating toxic heavy metals contamination from pharmaceutical and food products, with the ultimate goal of ensuring the safety and purity of consumer goods.

## **CHAPTER-3: QUADRUPOLE INDUCTIVELY COUPLED PLASMA MASS SPECTROMETRY FOR THE DETERMINATION OF HEAVY METAL CONCENTRATIONS IN CHOLIC ACID**

In this study, a microwave-assisted acid digestion approach combined with a Q-ICP-MS analysis successfully validated the measurement of toxic heavy metals in Cholic acid. For the analysis of trace metals in Cholic acid, this method is considered to be efficient, reliable, and quick. In terms of accuracy, which is defined as a range of 30–150% of the target value, and precision, which is expressed as  $n=6$ , the suggested technique successfully met the standards given by WHO, AOAC, USFDA, ICH, and USP 232/233. For routine analysis of toxic heavy metals in Cholic acid, the proposed method has been successfully implemented. All of the

following parameters LOD, LOQ, linearity, repeatability, reproducibility, accuracy, and precision) were successfully tested using the validated technique. The validated method given is ideal for the quantification of these potentially harmful metals in regular laboratory analysis because it is exceedingly simple, quick, basic, and economical. Scheme of Quantification of Toxic Heavy Metals present in Cholic Acid by Q-ICP-MS in Fig. 1.



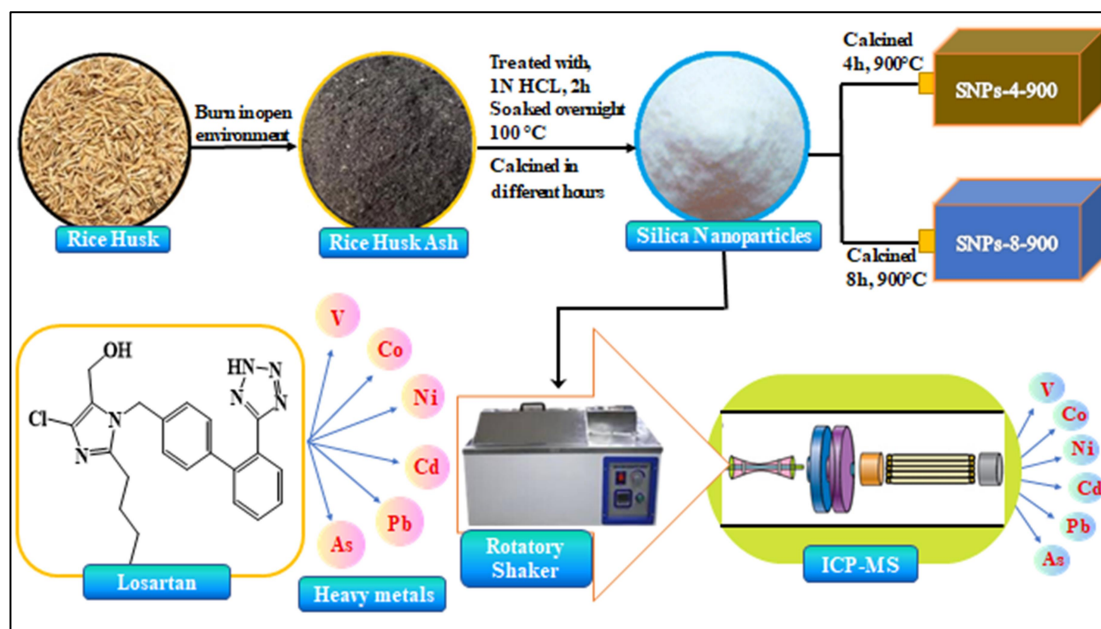
**Fig. 1:** Scheme of Quantification of Toxic Heavy Metals present in Cholic Acid

In this investigation, employing a microwave-aided acid digestion procedure and Q-ICP-MS analysis, the quantification of toxic heavy metals in Cholic acid was validated to a satisfactory level. It is regarded as a good, reliable, and speedy method for analysing trace metals in Cholic acid, among other applications. The proposed method's accuracy (30-150% of the target value) and precision value (n=6) satisfactorily meet the standards set by the WHO, AOAC, USFDA, ICH, and USP 232/233. The recommended method for analysing the Cholic acid sample was carried out with success. The LOD, LOQ, linearity, repeatability, reproducibility, accuracy, and precision of the validated method have all been assessed and determined to be within acceptable ranges. The validated approach that was presented is extremely straightforward, speedy, uncomplicated, and inexpensive; in addition, it is dependable, which makes it an excellent choice for the quantification of these potentially dangerous metals in routine laboratory analysis.

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## **CHAPTER-4: GREEN REMOVAL OF TOXIC HEAVY METALS FROM LOSARTAN USING RHA DERIVED SNPs**

Toxic Heavy Metal contamination in medication, food, the environment, water, etc. is one of the most important problems we are currently facing. They are hazardous even in extremely low quantities. Numerous of them result in birth defects and are carcinogenic. Consequently, we must reduce or eliminate the toxicity of heavy metals in medicine. Adsorption is one such technique that not only gets rid of toxic heavy metals from medication but is also environmentally benign and has a little effect on the environment. Even while adsorbents like activated carbon are frequently used, their high cost restricts their availability. Because of this, it's critical to think about long-term fixes that also take the problem's broader effects into account. Biochar, industrial waste, and agricultural waste are all examples of low-cost adsorbents that can be used to help remove toxic heavy metals from water at a cheap cost. These adsorbents are easy to make, and their fundamental constituents are readily available. It is therefore an environmentally friendly technique that greatly enhances the toxic heavy metals removal procedure in the medical field. More research into creating affordable adsorbents could help in the removal of toxic heavy metals. In order to test their efficacy as a toxic heavy metal ion adsorbent, we effectively synthesised SNPs from a variety of biomasses. Adsorption experiments showed a high capacity for the removal of toxic heavy metal ions including V, Co, Ni, Cd, Hg, Pb, and As. We were able to reduce V (78.76%), Co (68.21%), Ni (82.87%), Cd (69.03%), Pb (80.34%), and As (84.45%) using SNPs-4-900 and SNPs-8-900. Thus, based on this finding, we may say that SNPs-8-900 is more effective than SNPs-4-900 at adsorbing hazardous and toxic heavy metals from the medication losartan potassium. Materials with excellent adsorption capability, reusability, and high regeneration capacity, as well as simple materials, are the main advantages of this work. As a result, the study opens up new avenues for putting environmental clean-up into practise in real life. Scheme of removal of Toxic Heavy Metals present in Losartan (API) by using RHA synthesized SNPs in Fig. 2.



**Fig. 2:** Scheme of removal Toxic Heavy Metals present in Losartan by SNPs

Toxic heavy metal contamination in medication, food, the environment, water, etc. is one of the most important problems we are currently facing. They are toxic heavy metals even in extremely low quantities. Numerous of them result in birth defects and are carcinogenic. Consequently, we must reduce or eliminate the toxicity of toxic heavy metals in medicine. Adsorption is one such technique that not only gets rid of toxic heavy metals from medication but is also environmentally benign and has a little effect on the environment. Even while adsorbents like activated carbon are frequently used, their high cost restricts their availability. Because of this, it's critical to think about long-term fixes that also take the problem's broader effects into account. Biochar, industrial waste, and agricultural waste are all examples of low-cost adsorbents that can be used to help remove toxic heavy metals from water at a cheap cost. These adsorbents are easy to make, and their fundamental constituents are readily available. It is therefore an environmentally friendly technique that greatly enhances the toxic heavy metals removal procedure in the medical field. More research into creating affordable adsorbents could help in the removal of toxic heavy metals. In order to test their efficacy as a toxic heavy metal ion adsorbent, we effectively synthesised SNPs from a variety of biomasses. Adsorption experiments showed a high capacity for the removal of toxic heavy metal ions including V, Co, Ni, Cd, Hg, Pb, and As. We were able to reduce V (78.76%), Co (68.21%), Ni (82.87%), Cd (69.03%), Pb (80.34%), and As (84.45%) using SNPs-4-900 and SNPs-8-900. Thus, based on this finding, we may say that SNPs-8-900 is more effective than

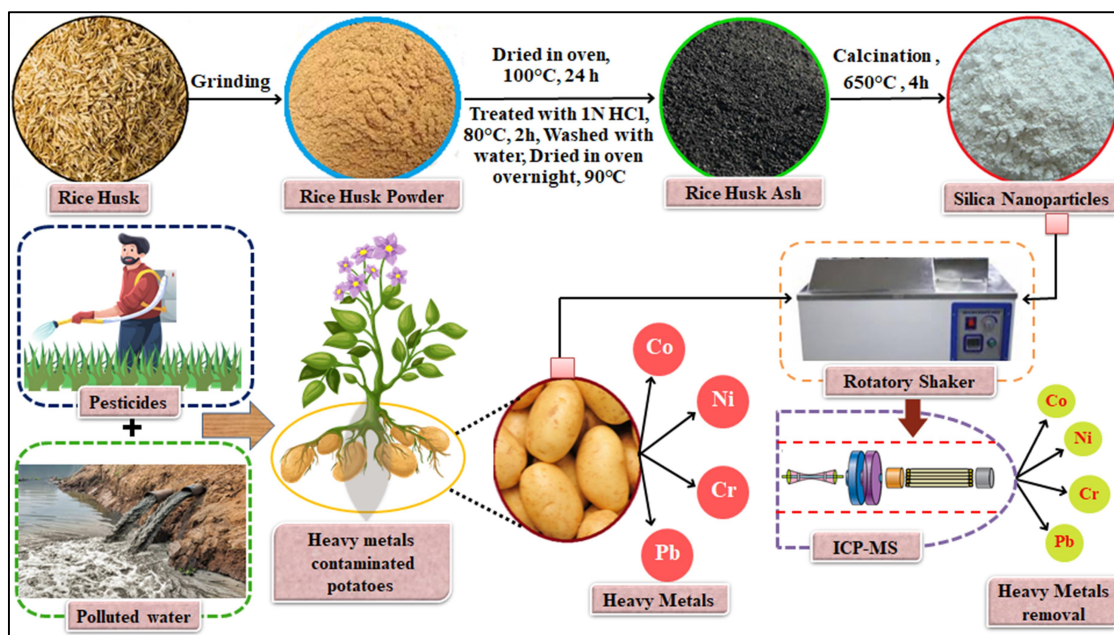
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## **CHAPTER-5: NANO-AGRI: RH SNPs FOR HEAVY METAL REMEDIATION IN POTATOES FROM NORTH GUJARAT**

Because eating polluted plants, like potatoes, might expose one to health concerns, metal poisoning in agricultural soils is a significant problem nowadays. Industrialization, mining, and agricultural practises are just a few of the human activities that can cause toxic heavy metal concentrations in the environment to surpass safe limits. Consequently, harmful toxic heavy metals like Co, Ni, Pb, Cd, and Cr may be present in potatoes and other crops. Techniques for effective removal are required to reduce these threats to human health and food safety. This study describes a low-cost, straight forward green synthesis method that removes toxic heavy metal contamination from North Gujarat region Banaskantha district's special variety potatoes, known as "KURFI BADASHAH" by extracting mesoporous SNPs from Navsari region "GNR-3 (Gujarat Navsari Rice – 3)" rice using a bottom-up approach. The EDX analysis led to the production of a high-purity SNPs by calcining the RH. To remove biological waste and toxic heavy metals impurities, RHA was calcined in a box furnace at 650°C for 4 h. This work discusses how to successfully decrease the quantity of dangerous toxic heavy metals contamination in potatoes by employing SNPs as a biomass adsorbent. All the basics of defining SNPs are included as well. The absence of sharp peaks in the XRD pattern, along with the presence of a broad peak at 22.1°, demonstrates that the GNR-3 variety rice SNPs are amorphous. SEM showed clusters of consistently formed, spherical-shaped SNP aggregates, while TEM revealed an average particle size of less than 50 nm. The two peaks at 1083.29  $\text{cm}^{-1}$  and 795.48  $\text{cm}^{-1}$  in the Fourier transform infrared spectra were found to correspond to the O-Si-O symmetric stretching vibration and the O-Si-O asymmetric stretching vibration. The BET study found that the average pore size was 8.5 nm, the specific surface area was 300.2015  $\text{m}^2/\text{g}$ , and the pore volume was 0.659078  $\text{cm}^3/\text{g}$ . To sum up, SNPs made from agricultural waste present a strong case for removing hazardous toxic heavy metals from potatoes. This method is simple since it uses easily accessible agricultural waste materials and requires little processing. Scheme of removal of Toxic Heavy Metals present in Potatoes by using RHA synthesized SNPs in Fig. 3.





**Fig. 3:** Scheme of removal Toxic Heavy Metals present in Potatoes by SNPs

In conclusion, the comprehensive analysis of the data presented in Table 2 reveals a reduction of toxic heavy metal contaminants present in North Gujarat region Banaskantha district special variety named "Kufri Badshah" through the strategic utilisation of synthesised SNPs derived from Navsari region variety "GNR-3" RH. We learned this by using synthetic SNPs based on a Navsari region variety called "GNR-3." We used these SNPs at various potato-to-SNPs ratios, including 1:2, 1:5, and 1:10, to show their exceptional efficacy in cleaning up biomass, especially potatoes of the Kufri Badshah kind. We discovered that after six hours of interaction with SNPs, biomass contained considerably lower amounts of Co, Ni, Pb, and Cr across all tested concentration ranges. The element-specific clearance values confirmed this. The element Co was removed at rates of 1,2980.62, 2,0490.28, and 3,4120.87 at adsorbent doses of 1,2, and 5, respectively. Similarly, Ni removal rates were 3.3820.30, 4.1826.43, and 5.643.33, and Pb removal rates were 0.6550.22.30, 1.264.16, and 1.417.08. The exceptional ability of SNPs to successfully adsorb these potentially harmful toxic heavy metals from potato biomass was demonstrated by the removal of chromium, with values of 3.6230.25, 4.9641.21, and 8.7331.15 being recorded. What stands out as most intriguing about this experiment is the finding that SNPs are resistant even in the midst of increased quantities of potato. Because of the continuous reduction in hazardous toxic heavy metals over a variety of potato-to-SNPs ratios, it appears that SNPs have the ability to adsorb and immobilise dangerous heavy metals that are present in potatoes, regardless of the number of potatoes.



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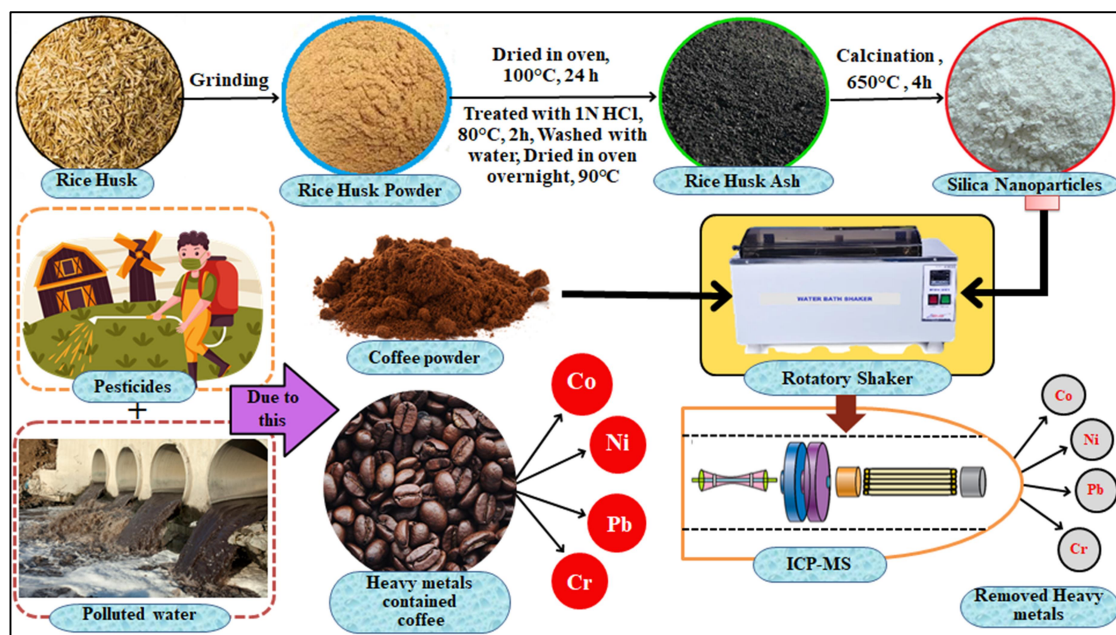
This not only proves the robustness of the SNPs, but it also implies that, at least up to greater concentrations of potato, they are highly effective in reducing the amount of harmful toxic heavy metal contamination in potato biomass. These results demonstrate the promise of SNPs synthesised from RH as a practical and long-term strategy for boosting the safety and quality of food crops like potatoes by lowering their exposure to harmful toxic heavy metals. Practical applications of these results to the problem of hazardous toxic heavy metal contamination in agricultural systems show great promise. More study and investigation are needed to fully realise the potential of SNPs in environmental remediation and agricultural sustainability. Due to the fact that basic foods make up such a large part of one's daily diet, food safety remains one of the most serious challenges influencing human health. Toxic heavy metals contamination of staple crops is a major problem, especially with potatoes, rice, and coffee. Controlling environmental toxic heavy metals levels is important because rice has a far higher potential than other crops do for build-up of dangerous toxic heavy metals. International bodies and national governments have established maximum allowable concentrations (MACs) for specific heavy metals in a wide range of foods. This work illustrates the cost-efficiency and environmental friendliness of using SNPs synthesised from RH for the elimination of harmful toxic heavy metals from food items. The NIFA (National Institute of Food and Agriculture) conducted the research. Using RH waste as a raw material for SNPs synthesis results in a by-product known as RHA, which has potential as a biomass resource for the elimination of toxic heavy metals in food. By employing rice husk waste as the raw material for SNP synthesis, not only is waste reduced efficiently, but toxic heavy metals are also eliminated. Raw material for SNPs production is discarded RH.

## **CHAPTER-6: REVOLUTIONARY ADSORPTION: RICE HUSK SNPs REMEDIATE HEAVY METALS IN COFFEE**

In this work, a unique biomass adsorption approach called Robusta is presented for the purpose of remediating hazardous heavy metals that are found in the Robusta kind of coffee that is farmed in Kerala. Utilising RHA synthesised SNPs as an efficient adsorbent is the strategy that will be used. However, the presence of dangerous toxic heavy metals in the soil significantly threatens both the quality of the crops and the health of the local population in the Kerala region, which is well-known for its coffee production. During the course of this study, RHA was collected from discarded agricultural products and then synthesised into SNPs in a manner that was under strict experimental control. After that, these nanoparticles

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were used as adsorbents for heavy and hazardous metals in coffee samples that were acquired from the region of Kerala. Adsorption efficiency was calculated by analysing toxic heavy metals concentrations before and after treatment using high-tech analytical tools and comparing the two sets of data. According to the findings, the SNPs that were synthesised using RHA had an exceptional ability for adsorption of hazardous toxic heavy metals, which successfully reduced the concentration of these metals in the coffee samples. Through this research, we were able to use SNPs to successfully lower the amount of harmful toxic heavy metals contamination found in coffee. In the research that we conducted, we found that utilising SNPs at different ratios (SNPs: Coffee) of 1:2, 1:5, and 1:10 was the most effective method for removing toxic heavy metal pollutants from coffee biomass. After agitation for a period of 6 h, the amount of Co that was removed was measured to be 2.083 0.15, 2.983 0.01, and 33.536 0.27 for the relevant ratios. The removal of Ni resulted in values of 2.786 0.60, 2.975 0.42, and 4.230 0.29, but the removal of Pb produced measurements of 1.192 0.15, 2.126 0.10, and 3.473 0.26. After elimination of Cr, the values were found to be 5.003 1.41, 3.346 0.43, and 1.418 0.35. Notably, the 1:10 ratio demonstrated a significant degree of efficiency in lowering the hazardous toxic heavy metal concentrations in coffee biomass. In addition, the SNPs-based adsorbents displayed good selectivity towards particular metals, which made it possible to remove specific contaminants from the coffee while still retaining the vital components of the beverage. When it comes to removing harmful toxic heavy metals from coffee manufacturing, this cutting-edge biomass adsorption approach provides a solution that is both environmentally friendly and economical. If this plan is put into action, the coffee grown in Kerala has a better chance of being of higher quality and posing less of a risk to consumers' health. Scheme of removal of Toxic Heavy Metals present in Coffee by using RHA synthesized SNPs in Fig. 4.



**Fig. 4:** Scheme of removal Toxic Heavy Metals present in Coffee by SNPs

In conclusion, we found that utilising SNPs at varied ratios (1:2, 1:5, and 1:10 SNPs: Coffee) was an effective method for lowering the toxic heavy metal contamination levels in coffee biomass. After shaking for 6 h, the ratio of 1:10 indicated the most significant elimination of harmful toxic heavy metals, including Co, Ni, Pb, and Cr. As a result of this, it appears that increasing the amount of SNPs beyond a ratio of 1:10 may not be essential, as SNPs have consistently demonstrated efficient removal of toxic heavy metals from coffee biomass. Our study's major aim was to look into claims that coffee is tainted with toxic heavy metals. Fe, Cu, Zn, Mn, Pb, Ni, and Cd concentrations were studied in detail. These results demonstrate the exciting potential of SNPs as a long-term, efficient technique for lowering toxic heavy metal contamination in coffee. Additionally, our study investigated potential variations in toxic heavy metal content across potato cultivars and discovered that different concentration ranges exist for each metals. Since staple foods constitute such a large portion of most people's diets, ensuring that they are safe to eat remains a top priority for public health. Even while MACs and MACs have been established by international organisations and governments, it is essential to acknowledge that concentrations even lower than these thresholds can be toxic harmful toxic heavy metals to one's health, particularly for populations who are more susceptible. Chronic exposure to even low amounts of some toxic heavy metals can result in a variety of ailments that are not caused by carcinogens, including cancer, hypertension, and neurological issues. In order to address the issue of toxic heavy

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metal contamination, our research investigated the potential for toxic heavy metal removal using modified potato biomass that has been treated with synthesised SNPs. This demonstrates the significance of developing optimised methods for the effective clean-up of contaminated environments of toxic heavy metals. Our findings shed light on the prevalence of toxic heavy metals in various potato varieties, demonstrating the need for stringent food safety regulations to lessen the risk of harmful health impacts.