

INTRODUCTION

1. Significance of the study

In the recent years, although the topic of treating heavy metals in aqueous solution has attracted the vast interest of research groups, the study in this section still need more in-depth works to meet the requirements for dealing with the risk of increasing environmental pollution around the world. Our group has chosen to synthesis and screening of potential absorbent materials which can be used as a replacement with higher performance, low cost and better diversification for treating heavy metals in aqueous solution.

There are many applying techniques to isolate the metal ions from aqueous solution, one of present practical study trend is using conducting polymer with heavy metal absorbing purpose. Some of research has been performed on the adsorb capability of heavy metal of Polyaniline-based (PANi-based) material by blending Polyaniline (PANi) with other agriculture by-product like sawdust, peanut shells, pea seedpods, etc...

Downy rose myrtle (*Rhodomyrtus tomentosa* Wight) (*Myrtus Tomentosa* Ait., *Myrtus canescens* Lour.), tea plant (*Camellia sinensis* (L.) O.Kuntze) are both native to Asia. According to these above-mentioned study, the using of both separatly PANi-based materials or some natural plant products on metal ions adsorption results in comparatively outcome. Thus, the new approach has been chosen on using polyaniline modified with rose myrtle, tea plant products to adsopt the metal ions in aqueous solution. We proceed to implement the thesis "*Modified Polyaniline materials on tea residue and rose myrtle, aiming at treatment of aqueous metallic ions*".

2. Research Objectives

- Synthesis and evaluation the adsorption capacity of PANi-based materials modified with rose myrtle and tea residue products on aqueous As(V), Cu(II), Pb(II), Cr(VI) ions.

- Implement study, selection and propose the practical method for using obtained PANi-based materials on treatment of several metallic ions in aqueous solution.

3. Scope of study

- Collect and processing raw plant materials from rose myrtle and tea leaves. Preliminary processing is conducted with several other method such as water extract; ethanol extract; pulverization; modification with KOH/H₃PO₄.

- Synthesis the PANi-based materials modified with the previous raw/modified plant products.

- Study and evaluate the structural characteristics of obtained PANi materials by FTIR spectroscopy, surface morphology by SEM/TEM and specific surface area BET.

- Evaluate the adsorption capacity on aqueous As(V), Cu(II), Pb(II), Cr(VI) ions of the above PANi materials.

- Select the materials with high adsorption capacity, apply in different effect condition and propose the application method for selected PANi-based materials modified with plant products on treatment of several metallic ions in aqueous solution.

4. New contributions of the thesis

- Performing the modification of rose myrtle product and tea residue on KOH, H₃PO₄ solution, which gave the much higher adsorption capacity of aqueous Pb(II), Cr(VI) ions comparison with the raw plant products.

- 14 new PANi-based materials has been successfully chemical synthesised with tea and rose myrtle product, including 08 types from PANi modified with rose myrtle product (PAN-S1÷PANi-S8) and 06 types from tea residue (PANi-C1÷PANi-C6). The structure and properties of selected materials have been analysed via IR, SEM, TEM, BET.

- Performing the study, evaluation on adsorption capacity of PANi-based materials modified with rose myrtle leaves and tea residue products on aqueous As(V), Cu(II), Pb(II), Cr(VI) ions over time. In conclusion, the high results of adsorption capacity of aqueous Pb(II), Cr(VI) ions in room temperature,

neutral medium have been found from PANi-C5, PANi-C6, PANi-S7, PANi-S8.

- The impact of other factors such as initial concentration, quantity of adsorbent, pH on adsorption efficiency have been studied on 04 types of selected materials (PANi, PANi-S1, PANi-S8, PANi-C6) for Pb(II), Cr(VI) ions. The adsorption model following the Langmuir isotherm.

- Propose a preliminary application plan with 02 new PANi-based materials (PANi-S8, PANi-C6) on treatment of aqueous Pb(II), Cr(VI).

5. Thesis structure

The main content of the thesis consists of 136 pages divided into sections: Introduction: 2 pages; Chapter 1. Overview: 40 pages; Chapter 2. Experimental and research methods: 22 pages; Chapter 3. Results and discussion: 56 pages; Conclusion: 2 pages; New points of the thesis: 1 page; Published works related to the thesis: 1 page; References: 12 pages. The thesis includes 23 tables, 85 figures and 114 references.

CONTENT OF THE THESIS

CHAPTER 1. OVERVIEW

The review was studied in 114 references.

1.1. Overview of Polyanilin (PANi)

1.2. Overview of plant objects used in environmental treatment.

1.3. Overview of research metal objects.

CHAPTER 2. EXPERIMENT AND RESEARCH METHOD

2.1. Research subjects

The research objects mentioned in the thesis include:

- Anions and metal cations: As(V), Cu(II), Pb(II), Cr(VI).
- Plant preparations: tea residue (C1÷C6) and rose myrtle branches (S1÷S8).

- The original PANi materials combine tea residue and rose myrtle branches: the materials are synthesized from ANi combined with plant

products by chemical method in 1M H₂SO₄ acid. Material samples are denoted as shown in Table 2.1 following.

Table 2.1. Samples of PANi combined with plant products

STT	Tên vật liệu	Ký hiệu
I. The original PANi material combined with tea residue		
1	PANi combined with tea residue	PANi-C1
2	PANi combined with activated tea residue in KOH solution	PANi-C2
3	PANi combined with activated tea residue in H ₃ PO ₄ solution	PANi-C3
4	PANi combined with fine tea residue	PANi-C4
5	PANi combined with activated fine tea residue in KOH solution	PANi-C5
6	PANi combined with activated fine tea residue in H ₃ PO ₄ solution	PANi-C6
II. The original PANi material combined with rose myrtle branches		
7	PANi combined with water extract of rose myrtle	PANi-S1
8	PANi combined with alcohol of rose myrtle	PANi-S2
9	PANi combined with residue of rose myrtle	PANi-S3
10	PANi combined with residue of activated rose myrtle in KOH solution	PANi-S4
11	PANi combined with residue of activated rose myrtle in H ₃ PO ₄ solution	PANi-S5
12	PANi combined with powder of rose myrtle	PANi-S6
13	PANi combined with powder of activated rose myrtle in KOH solution	PANi-S7
14	PANi combined with powder of activated rose myrtle in H ₃ PO ₄ solution	PANi-S8

2.2. Chemicals, tools and equipment

2.3. Experiment

2.3.1. Synthesis of original PANi materials combining plant products

2.3.1.1. Extraction and activation of plant samples

a) Activating tea residue

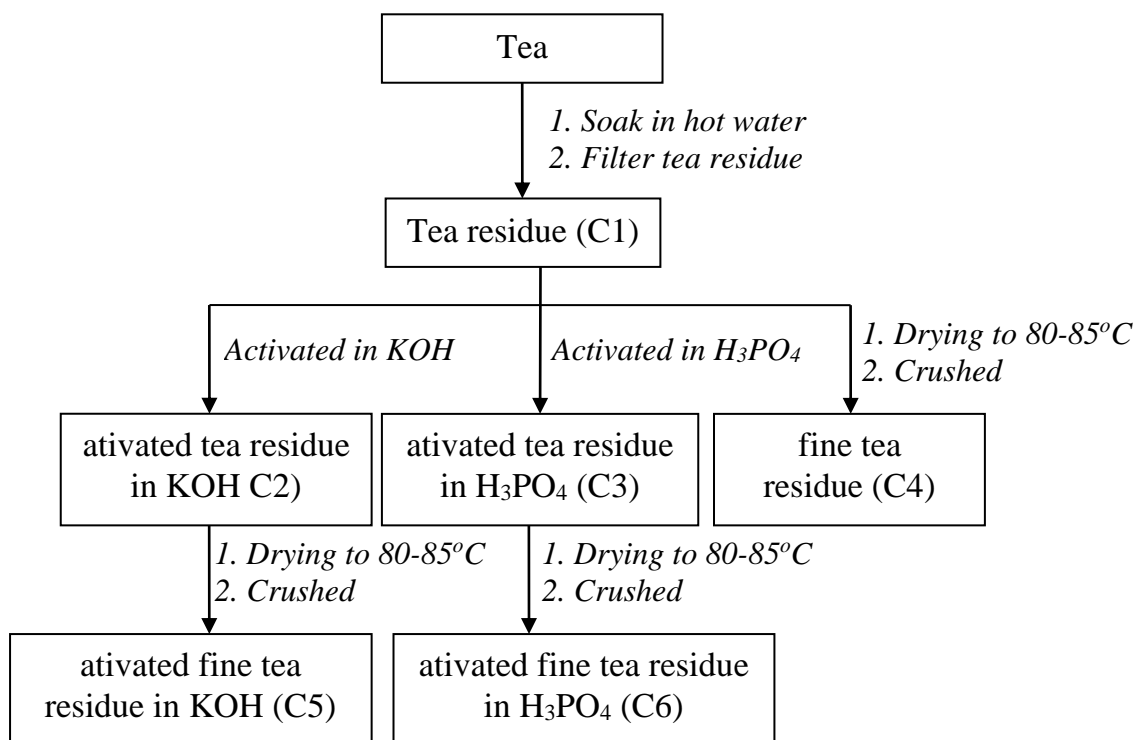


Diagram 2. 1. Activation process of tea residue

b) Extraction and activation of rose myrtle samples

+ *Extraction of rose myrtle samples:*

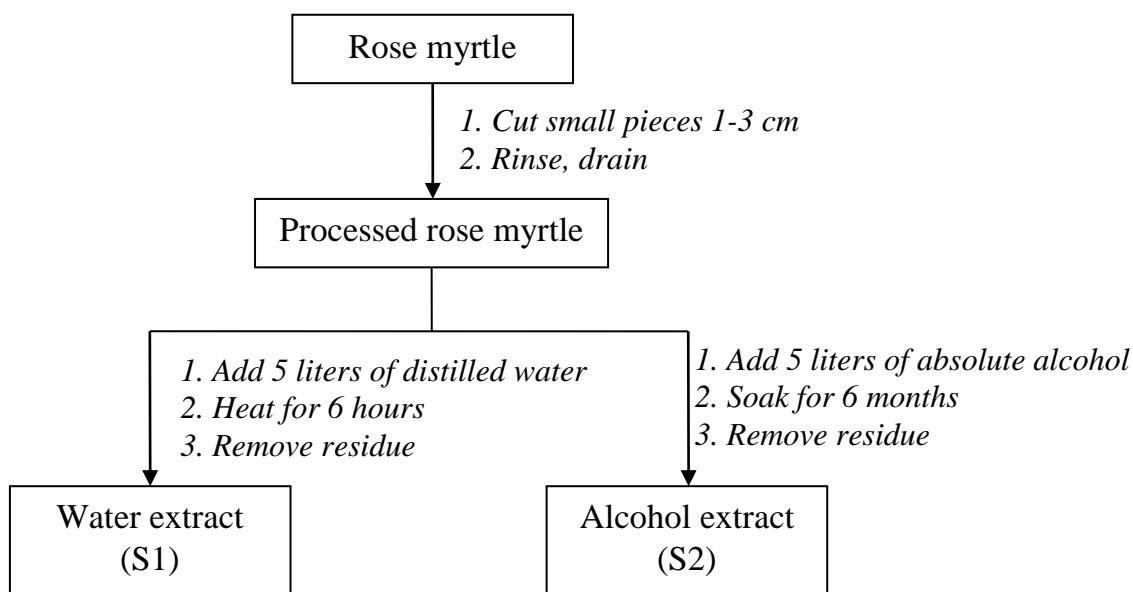


Diagram 2. 2. Extraction of rose myrtle samples

+ *Activation of rose myrtle samples:*

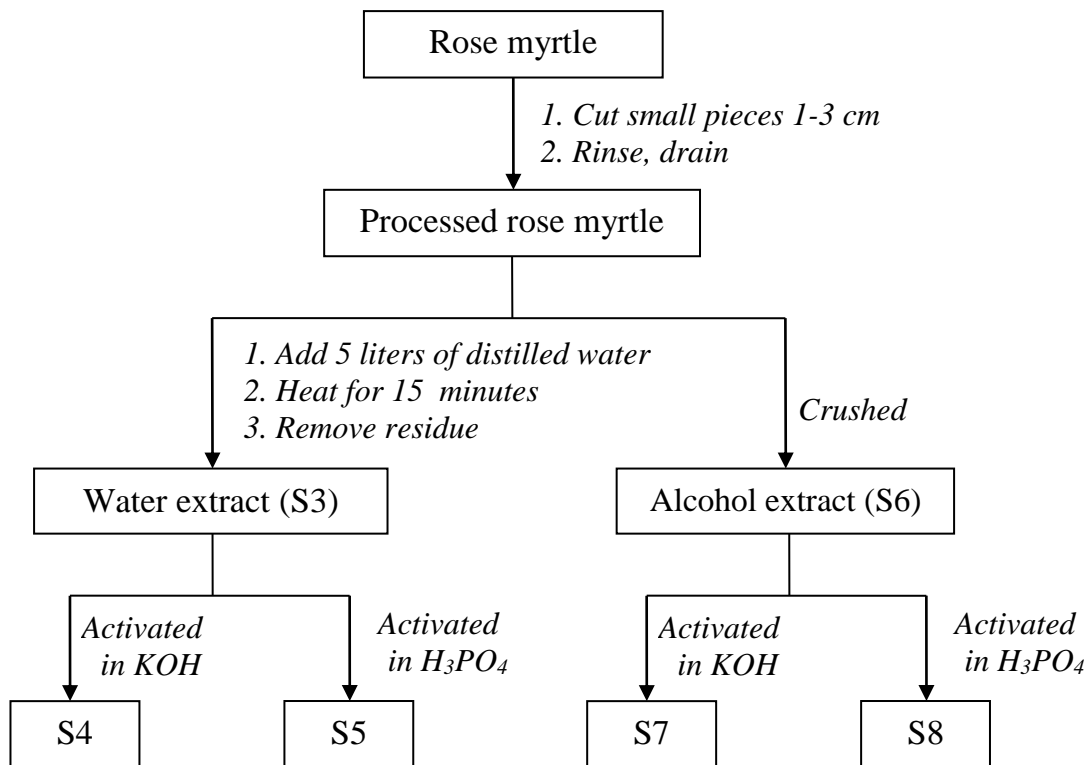


Diagram 2. 3. Activation of rose myrtle samples

2.3.1.2. Synthetic polyanilin conductive polymer

Polyanilin is synthesized by chemical method in 1M H₂SO₄ solution medium as follows:

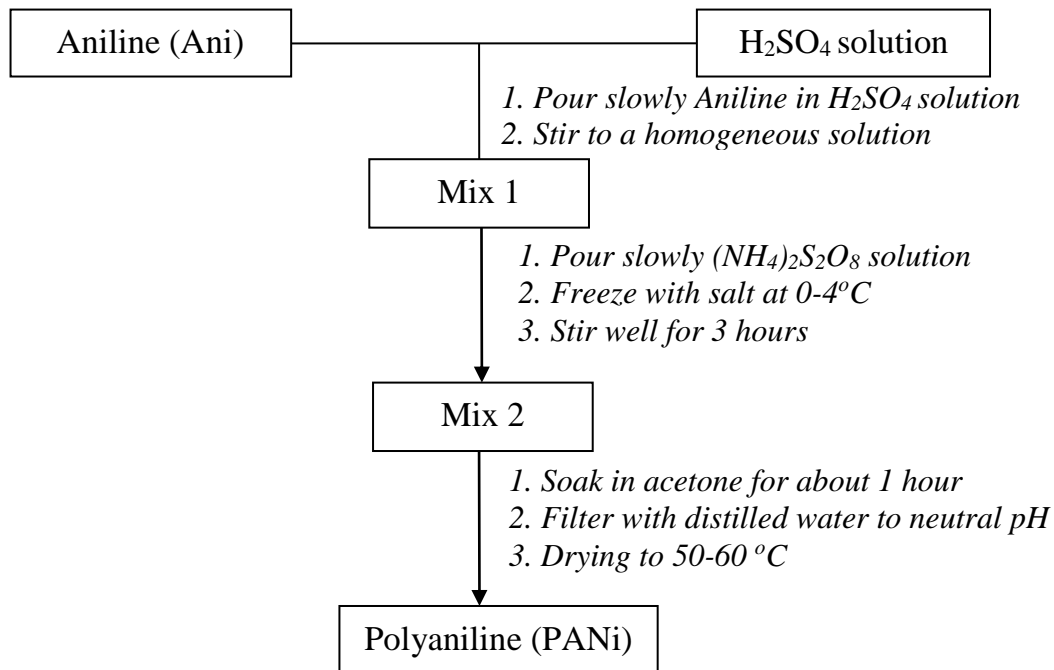


Diagram 2.4. Synthesizing PANi by chemical method

2.3.1.3. Synthesis of original PANi materials combining plant samples

Synthesis of PANi materials combined with plant sample includes steps:

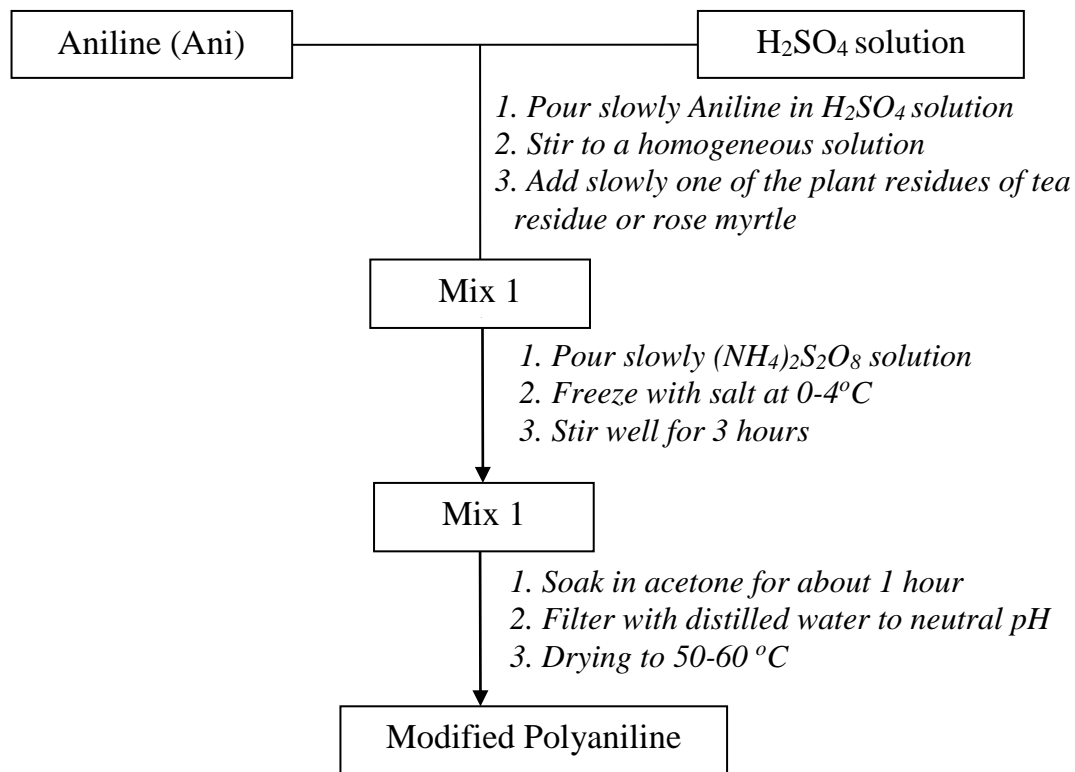


Diagram 2.5. Synthesized PANi materials combining plant samples

2.3.2. Study the adsorption capability of PANi-based materials

The implementation process include the following studies:

- Survey, evaluate the adsorption capacity of As (V), Cu (II), Pb (II) and Cr (VI) of each material over time (t = 5ph, 15ph, 30ph, 60ph, 120ph, 240ph , 360ph). The solutions of As (V), Cu (II), Pb (II) and Cr (VI) were conducted during the experiment at concentrations of 50 mg/l and 100 mg/l.

- Surveying on some experimental conditions such as initial concentration C₀ ranges from 100 mg/l ÷ 2000 mg/l, pH environment ranges from 1.5 ÷ 7, volume of adsorbent m = 0,5 g ÷ 5 g for selected materials and metals.

- Study of adsorption isotherm models of typical materials.

2.4. Research methods

2.4.1. Adsorption method

2.4.2. Infrared spectrum method (IR)

2.4.3. Scanning electron microscope method (SEM)

2.4.4. Transmission electron microscope method (TEM)

2.4.5. Method of specific surface area (BET)

2.4.6. Atomic absorption spectroscopy method (AAS)

CHƯƠNG 3. RESULTS AND DISCUSSION

3.1. Synthesis of polyanilin original materials (PANi)

3.1.1. Aniline conversion efficiency

The aniline conversion efficiency of the material samples is similar and fluctuates in the range of 75 ÷ 83%. Metabolic performance is quite high indicating that the selection of processes and conditions for synthesizing polyanilin materials in combination with plant preparations is appropriate.

3.1.2. Characteristics of material structure

3.1.2.1. Research results of infrared spectra (IR)

a) Infrared spectrum of PANi

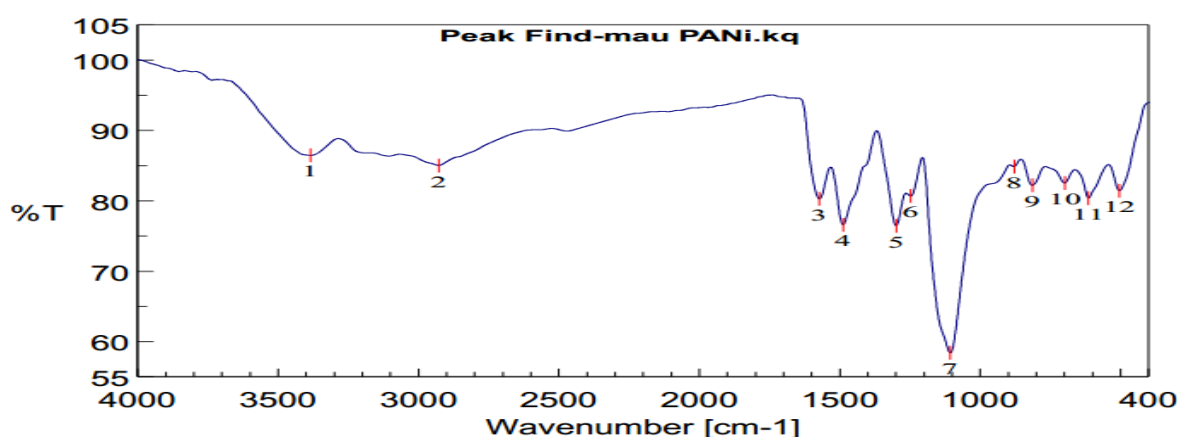


Figure 3. 1. Infrared spectrum of polyanilin (PANi)

In the graph spectrum of the PANi sample, we see the signal of functional groups in the synthesized PANi such as benzoite ring at 1572 cm⁻¹

and characteristic for quinone ring type at oscillation of 1487 cm^{-1} , oscillations chemotherapy of the second N-H amine group at 3385 cm^{-1} , the valence oscillations of C-H benzene ring at 2927 cm^{-1} , oscillation of the double nitrogen atom associated with the quinone ring ($\text{N} = \text{quinone} = \text{N}$) at 1298 cm^{-1} , bonding between carbon atoms in aromatic rings with nitrogen atoms of diamine form at 1246 cm^{-1} and valence oscillation of C-N⁺ groups at 1105 cm^{-1} .

b) Infrared spectrum of tea residue

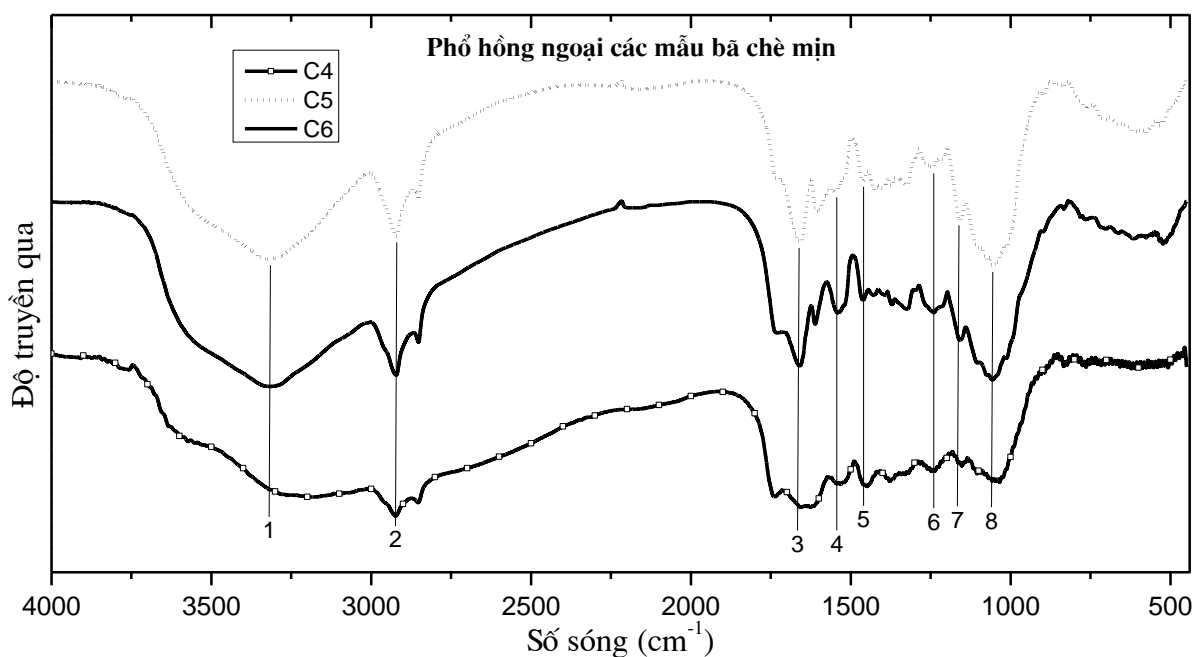


Figure 3. 2. IR spectrum of smooth tea residues C4, C5, C6

The IR spectrum shows that the presence of specific frequencies of functional groups in fine tea residues activated by KOH or H₃PO₄ is similar. Based on infrared spectra, it can be seen that some functional groups characterize the compounds of fine tea residues such as: -OH group, specific for alcoholic, phenol at the position of 3000- 3300 cm⁻¹ with broad peak characteristics as for carboxylic compounds or anhydrides, oscillations are characteristic of C-H no symmetry groups at about 2930 cm⁻¹, C-H groups are asymmetric at 2850 cm⁻¹, oscillating at position 1660 and 1730 cm⁻¹ represent carbonyl group > C = O of carboxylic acid or aromatic aldehydes and ketones. The characteristic pic for quadratic amine group -NH- or vibrating oscillation of group -C = C - in aromatic nuclei also appears at weak intensity at 1540

cm⁻¹. In tea bags, there are many asymmetric CH₃ groups that appear in the range of 1450-1470 cm⁻¹ and symmetrically at 1360-1380 cm⁻¹ and C-O bonds are full in about 1160 cm⁻¹ [109, 110] .

c) *Infrared spectrum of the PANi material combines smooth tea residue*

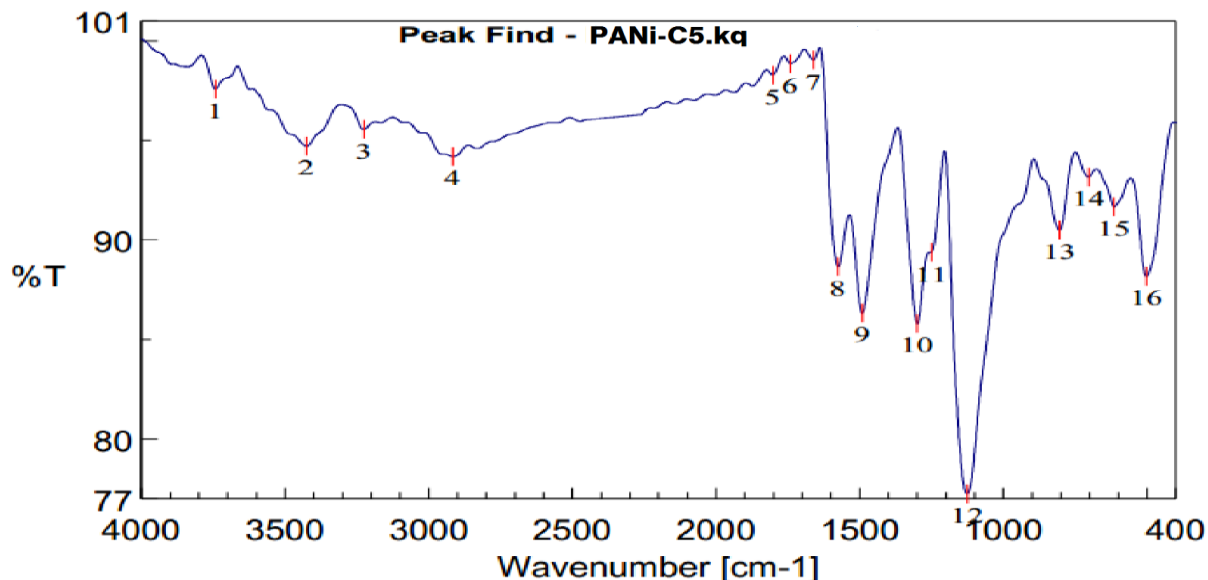


Figure 3. 3. Infrared spectrum of PANi-C5 material model

The results of infrared spectra analysis showed that PANi-C5 has shown that the functional groups for both PANi and tea residue are smooth activated KOH, PANi-C6 material also has characteristic peaks shown for functional groups are present in both PANi and fine tea residues activated by H₃PO₄ when combined. On the spectrum there is a decrease and variation compared to the initial pic of PANi or samples of smooth activated tea residues are explained by the interaction and mutual masking of some functional groups but it can be concluded that the total Successfully combining PANi root material in combination with activated tea residue by KOH/H₃PO₄. Based on the infrared spectrum of the combined material PANi-C5, it can be seen that the characteristic characteristic of the -OH group has strongly decreased in intensity, the characteristic peaks in the spectrum have high similarity with the infrared spectrum of PANi can be covered by PANi material on the surface of tea residue material.

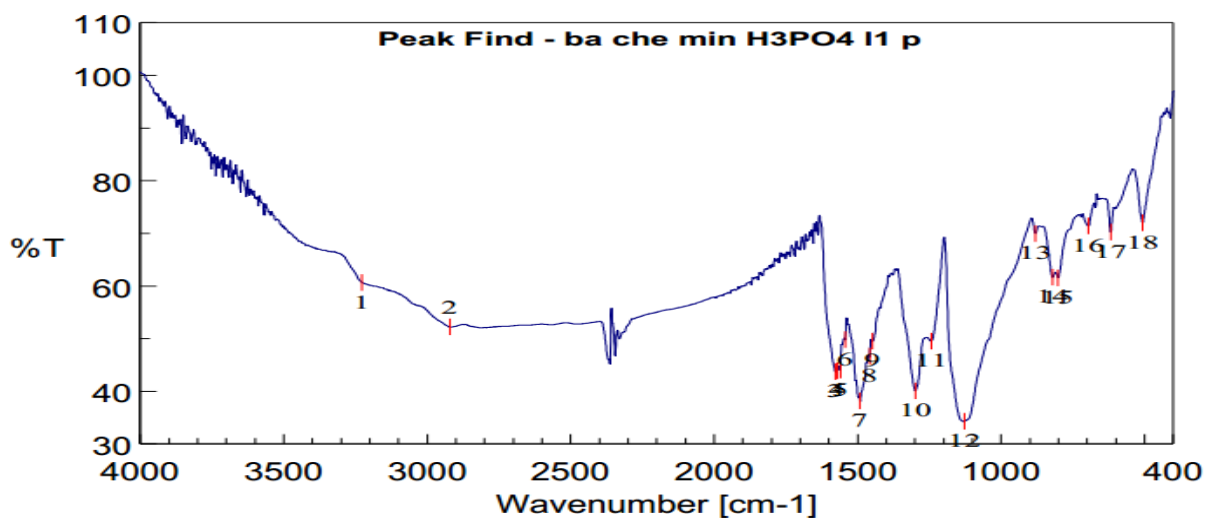


Figure 3. 4. IR spectrum of PANi-C6 material sample

d) Infrared spectrum of rose myrtle branches

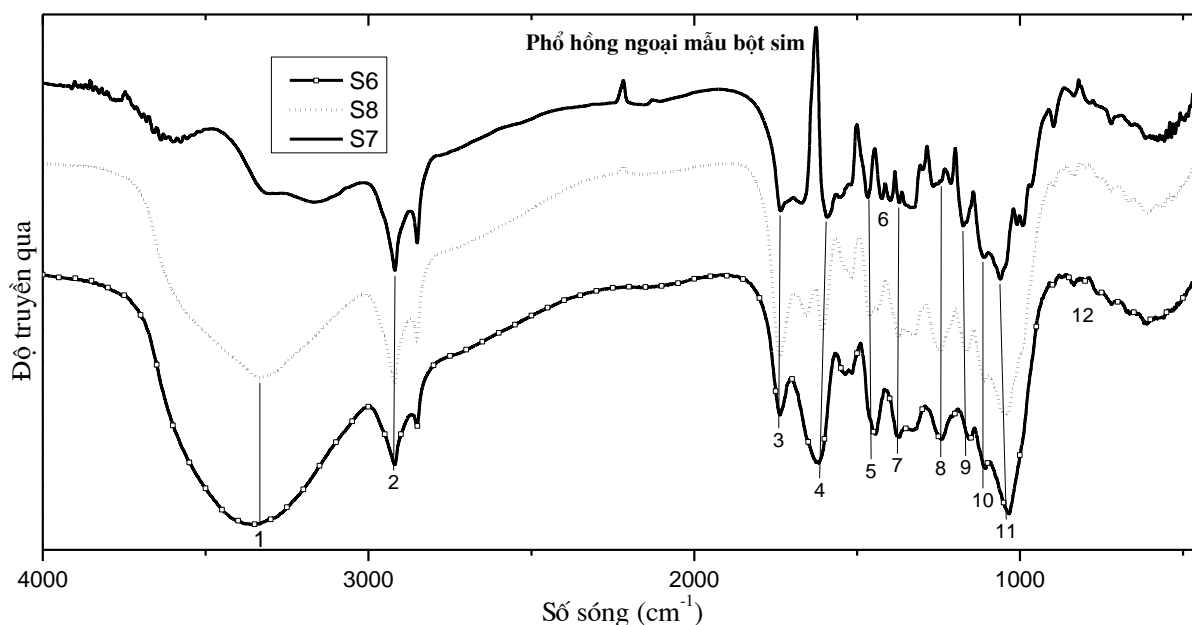


Figure 3. 5. IR spectrum of of rose myrtle branches (S6, S7, S8)

The sample of foliage foliar material activated with H_3PO_4 (S8) also appeared typical frequency for functional groups in the sample of activated rose myrtle powder material with KOH (S6). In the infrared spectrum of the dendritic foliage plants have shown functional groups such as: OH group specific to anco, phenol at positions 3321, 1398 and 1335 cm^{-1} , oscillation of functional group C = O (carbonyl and carboxyl) at positions 1735 and 1669 cm^{-1} , at the position of 1591 cm^{-1} characterizing the functional group -NH- of the order of amine 2. C-O – C ether oscillation occurs at position 1230- 1260

cm⁻¹, oscillation of C-OH bond at position 1174 and 1116 cm⁻¹, methoxium O-CH₃ group appears at position 1444 and 1060 cm⁻¹ and benzene ring oscillator signal at 897 cm⁻¹ first. In the infrared spectrum of rose myrtle powder samples, there are representative oscillations of lignin and cellulose.

e) *Infrared spectrum of material PANi combined with rose myrtle powder*

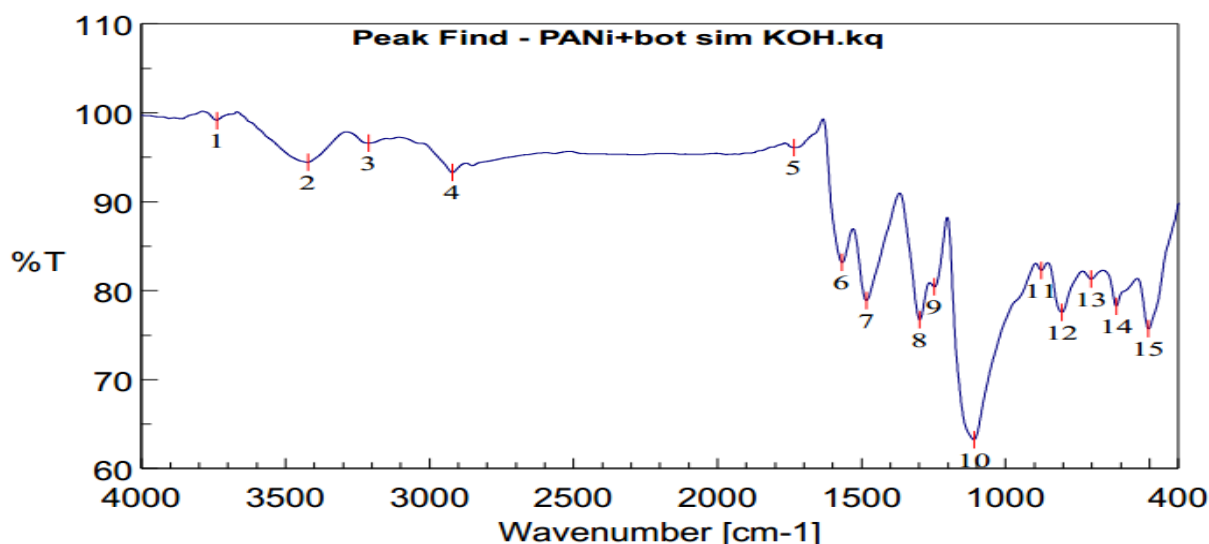


Figure 3. 6. IR spectrum of PANi-S7 material

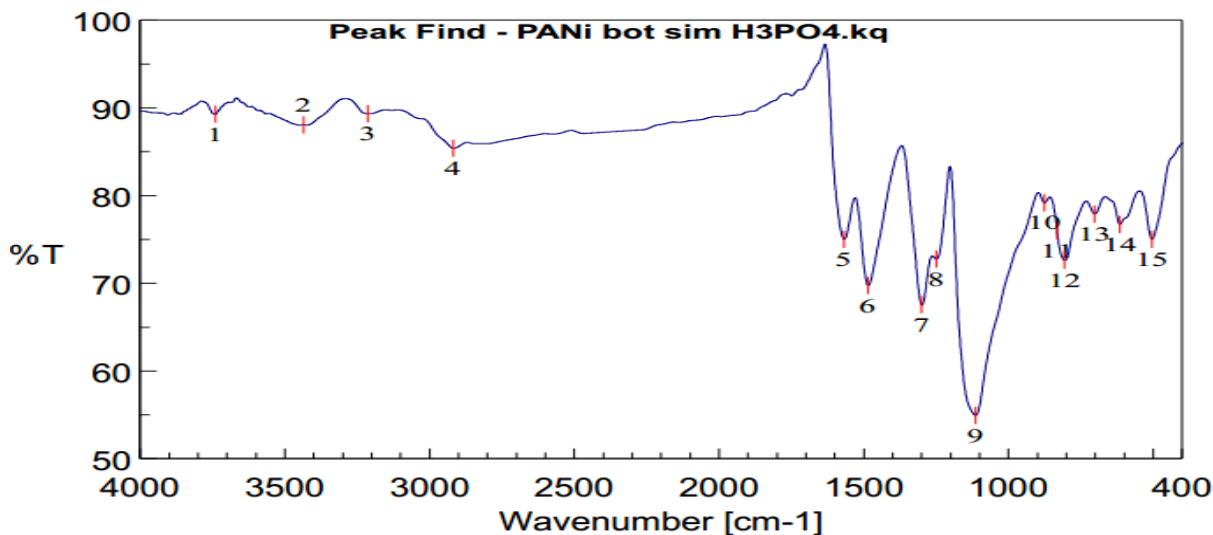


Figure 3. 7. IR spectrum of PANi-S8 material

The PANi-S7 material has shown the functional groups specific to both PANi and KOH activated plant powder, PANi-S8 material also appears characteristic peaks for functional groups in both PANi materials and rose myrtle powder activated by H₃PO₄ when combined. On the spectrum there is a decrease and variation compared to the initial pic of PANi or the activated

rose myrtle powder is explained by the interaction and mutual masking of some functional groups but it can be concluded successfully synthesized PANi original materials in combination with rose myrtle powder activated with KOH/H₃PO₄. The similarity between IR spectrum of PANi-S7 and PANi-S8 materials with PANi's spectrum shows that the surface of the material contains mostly PANi. This may suggest that the PANi material has been covered on the surface of the rose myrtle branches.

3.1.2.2. Results of research on material structure

a) Analysis of SEM images of tea residue

- SEM image of tea residue: SEM images of tea residue samples showed that these materials are sheets and plates, protruding, rugged, less uniform surface. Image of surface structure of tea residue material after being activated by KOH (C2) or H₃PO₄ (C3) has some changes in form compared to inactive sample (C1).

- SEM image of fine tea residue: The material of tea residue after finely ground (C4) and tea residue smooth activated KOH (C5) or H₃PO₄ (C6) with material size from 50 ÷ 70 µm, number of pores strongly increased compared to non-crushed materials and uneven size in the range of 100 ÷ 200 nm. The material of fine tea residues activated by KOH or H₃PO₄ changes a lot on the surface, forming cavities, slits with a distance of 0.5 ÷ 1 µm, strongly dissected to form a structure with large pores (macropore) and small pores. The surface appears to have particles of 200 nm in size, interwoven in blocks, branched and short fibers, combined together into areas separated by gaps, local pores evenly and the size is in the range of 100 ÷ 150 nm.

- SEM image of the original PANi material combines fine tea residue: The synthetic PANi material is dark green, in the form of fine powder. SEM images of materials show grain texture in coral form, particle size of 90 ÷ 100 nm. The particles associated with each other form branches in the larger network with the number of large formed holes. The distance between the network branches is relatively uniform, forming holes reaching 200 nm. The

surface of PANi root material in combination with smooth activated tea residue in KOH (PANi-C5), PANi combined with activated smooth tea residue in H₃PO₄ (PANi-C6) has larger particle size from 100 ÷ 200 nm, The particles are rod-like, combining together into corals. The size of the hole between the particles increases, the porous network forms with a large oscillation size in the range of 100 nm ÷ 400 nm. When comparing and comparing SEM images of original materials PANi, C5, C6 and materials after combining PANi-C5, PANi-C6, it shows that the surface changes big, the size of the material particles after coordination Case increases compared to the original materials. This allows for the guessing of PANi material after covering the outside of fine tea residues has increased the size of branches, while keeping the main properties such as foam, fibers and forming the spatial structure.

b) SEM images of rose myrtle branches

- SEM image of rose myrtle branches: The material of rose myrtle branches before and after activating with KOH or H₃PO₄ has a big difference. Samples of materials S3 have a fiber-like surface of the sheet and slabs specific to raw materials from plants with cellulose fibers and ribbed networks. The size of each cellulose fiber reaches 500 nm, the plates are much larger, covering the yarn. After activation with KOH, the surface of the sample (S4) changes drastically, the slab is no longer available, becoming a completely fibrous form, the holes are formed with very large sizes from 0.5 ÷ 1 µm. With S5 materials, the surface is completely changed to form long strands of chaotic distribution.

- SEM image of rose myrtle powder: The material of rose myrtle powder after smooth grinding (S6) is from 50 ÷ 70 µm, the surface of the material is in the form of slabs, scales with many layers are stacked, the size of the plates is from 0.5 ÷ 3 µm. Similar to tea residue, after activating with KOH or H₃PO₄, the material surface changes drastically. With the S7 sample, the surface is converted into a fully fibrous form, the scales are smaller in number and formed with smaller sizes, reaching 0.5 ÷ 1 µm. The S8 material has a surface in the form of fibers, combined into bundles of fibers separated by

similar areas by grooves, the size of each fiber reaches $200 \div 250$ nm. The holes and cavities are therefore formed in large and heterogeneous sizes, distributed from $0.2 \div 2$ μm .

- SEM image of the PANi material combined with rose myrtle powder: SEM image of the original PANi materials combined with activated rose myrtle powder in KOH solution (PANi-S7) and PANi combined with activated rose myrtle powder H_3PO_4 solution (PANi-S8) shows that these materials are fibrous, the fibers formed from PANi are bigger, longer and more oriented. The surface of PANi-S7 material forms long strands of rod-shaped fibers, facing outward to the surface, each fiber size reaches 200 nm. Between the bundles of fibers forming gaps, pores due to the interweaving of the bundles of fibers, the size of these slots is relatively equal to $150 \div 300$ nm. With PANi-S8 material, the fibers on the material surface are shorter, the volume is stronger and the size of each fiber is smaller, reaching only 50 nm. Porosity and openings formed on uneven surfaces, divided into spatial porosity consisting of large pores formed between smaller blocks of material and pores right on the material, smaller pore size reached 100 nm. When comparing and comparing the SEM image of the original materials PANi, S7 and the material after combining PANi-S7, the surface with the flake shape of S7 is replaced by the surface with fibers similar to that of PANi material, however, the size of these fibers increased sharply compared to PANi. Similar to S8 and PANi-S8, materials after combining large differences of original materials. This allows for the assumption of PANi after polymerization to cover the outside of the rose myrtle powder, increasing the size of the fibers, while keeping the characteristics of porosity, fibrous structure, branching and texture. structure space.

c) TEM image analysis of plant preparations

When studying TEM images of two samples of original PANi materials combined with activated tea residue H_3PO_4 and rose myrtle branches (PANi-C6, PANi-S8), all materials have spatial structure with holes, gaps formed during material synthesis. Due to the hollow structure and the gap between the blocks of material, it forms a capillary network on the surface. The outer

surface layer and the material are homogeneous blocks, not doped. Samples of PANi root materials in combination with finely activated tea residues H_3PO_4 exist uniformly covered materials. The structure of the material in the form of stacked and multi-directional growth pathways should create a lot of space, the size of the material sprouts on the surface is approximately 50nm. With the formation of a porous surface with a multidimensional structure, it is possible that the synthetic material retains its specific surface area characteristics. Similarly, we can see that the original PANi material combined with activated rose myrtle branches in H_3PO_4 acid has a multi-branched coral form, the branches are alternating and overlapping, creating many holes and porosity on the surface. Branch sizes range from 20 nm ÷ 100 nm. The material is highly homogeneous, forming a skeleton in space with a porous structure and forming capillaries on the surface.

3.1.2.3. Research results determined specific surface area

a) The specific surface area of the PANi material combines tea residue

From the results of measuring the specific surface area of fine tea residues (C4), activated activated tea residues of KOH (C5), activated fine tea residues H_3PO_4 (C6) and PANi original materials combined with fine tea residues Currently Table 3.10 we can see: the specific surface area of the fine activated tea residue after activation is higher than that of the inactive tea residue. The specific surface area of the C6 sample is higher than that of the C5 sample, the combination of the active pulp residue of H_3PO_4 (PANi-C6) is higher than the specific surface area of PANi combined residue. KOH smooth tea (PANi-C5). However, the specific surface area of the PANi material samples combined with fine tea residues (PANi-C5 and PANi-C6) decreased compared to the specific surface area of PANi material, and increased compared to the specific surface area. of models C5 and C6. This can be explained as follows, when combining two types of PANi materials with a large specific surface area and fine tea residue compositions with many smaller smaller surface areas have caused the combined effect, PANi layer synthesized

into the ability to only cover the outside of the fine tea residue, resulting in a change of the specific surface area index of the product, lower than that of materials with large and increased index compared to materials. have lower index. The capillary hole size of PANi base materials ranges from 27,677 nm ÷ 39,932 nm to the average capillary material (Mesopore) with a capillary diameter of 2-50 nm, suitable for use as a material adsorbent.

b) The specific surface area of the PANi material combined with the rose myrtle branches

From the results of measuring the specific surface area of rose myrtle branches (S6), activated rose myrtle branches in KOH solution (7), activated rose myrtle branches in H₃PO₄ acid (S8) and PANi base materials fine tea residue combination is shown in table 3.11, we can see: the specific surface area of the S7 and S8 samples is higher than that of the S6 model. The specific surface area of the sample S8 is higher than that of S7, the sample of PANi combined with the rose myrtle powder in H₃PO₄ acid (PANi-S8) is higher than the specific surface area of the original material PANi combined with spike activated branches in KOH solution (PANi-S7). However, the specific surface area of PANi-S7 and PANi-S8 samples decreased compared to the specific surface area of PANi material, and increased compared to the specific surface area of S7 and S8 samples. This can be explained similarly to the above-mentioned PANi sample of fine tea residues. When combining these two materials, one has a large specific surface area and the other has a much smaller surface area that has a combined effect, the resulting product has a surface area index. separately changed, decreased compared to materials with large index and increased compared to lower index materials. The capillary hole size of PANi base materials ranges from 27,677 nm ÷ 39,932 nm to the average capillary material (Mesopore) with a capillary diameter of 2-50 nm, suitable for use as a material adsorbent.

- From the results of analyzing the characteristics and structure of some samples of tea residue, residue/pulp and leaves, the original material of PANi combined with tea residue, rose myrtle braches shoots in KOH solution or

H₃PO₄ by infrared spectroscopy (IR) method, scanning electron microscope (SEM), transmission electron microscope (TEM), and specific surface area (BET) can be found that:

- Successfully synthesized samples of PANi original materials in combination with plant products of tea residue/rose myrtle powder.

- Samples of materials are quite homogeneous, fiber-like, uniform pores and overlapping in size about 50 - 200 nm. When combined with the results of infrared spectroscopy analysis, SEM imaging, TEM and specific surface area results (BET) can be affirmed more assertively for the assumption that PANi material is fully covered. the dark part is the tea residue rose myrtle powder, the PANi material after the polymerization process has covered the outside of the rose myrtle powder, increasing the size of the fibers, while keeping the characteristics of degree porous, fibrous, branched and spatial structures.

- There is an average capillary (Mesopore) with a capillary diameter of 2-50nm, suitable for the purpose of use as adsorbent.

3.2. Survey, evaluate the ability of adsorption of anions and metal ions

3.2.1. Study the adsorption capacity of As (V) according time

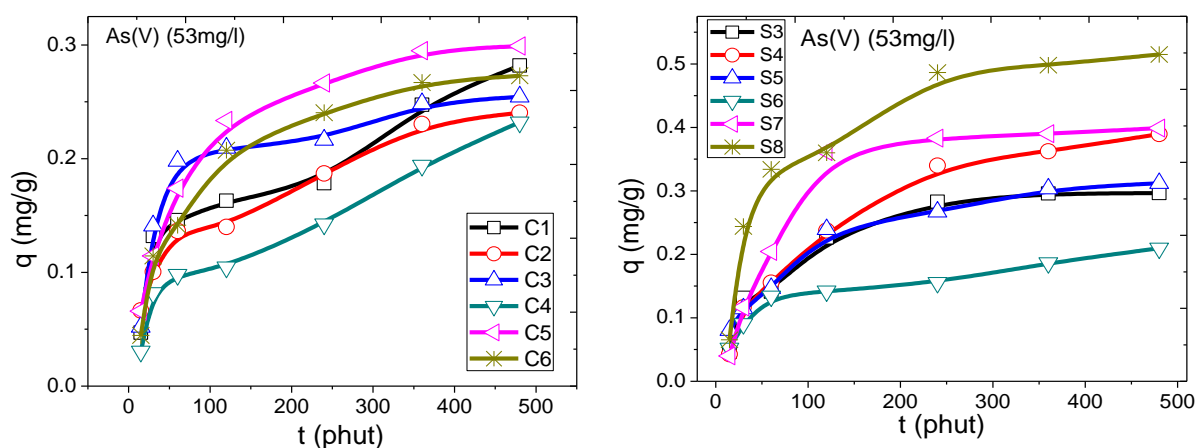


Figure 3.8. As (V) adsorption capacity of plant preparations

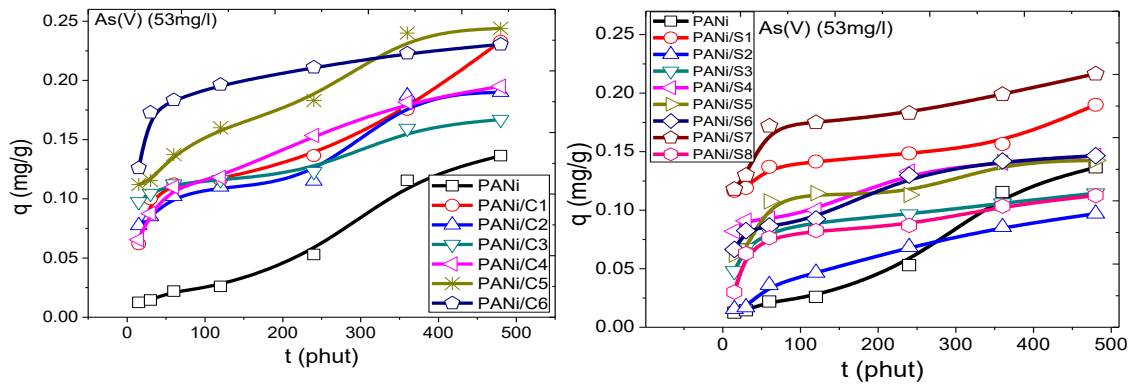


Figure 3.9. The capacity of As (V) adsorption

Adsorption capacity over time shows the adsorption capacity of As (V) of tea residue plant products, rose myrtle branches and 14 samples of PANi root materials combined with tea residue preparations, rose myrtle branches are very low. After the period $t = 360 \div 480$ minutes, the experiment of adsorption process appears to be balanced. The adsorption capacity of the sample groups ranged from 0.013-0.515 mg/g.

3.2.2. Study on Cr (VI) adsorption capacity according time

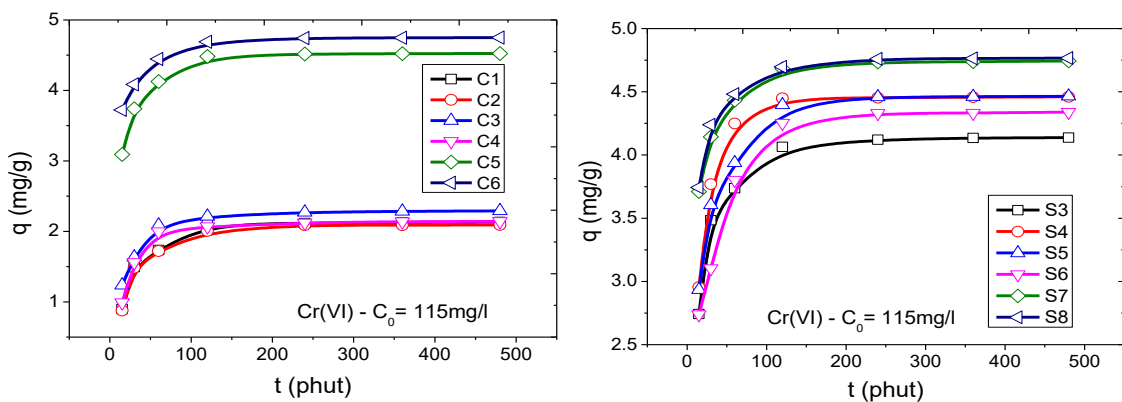


Figure 3.10. Cr (VI) adsorption capacity of plant preparations

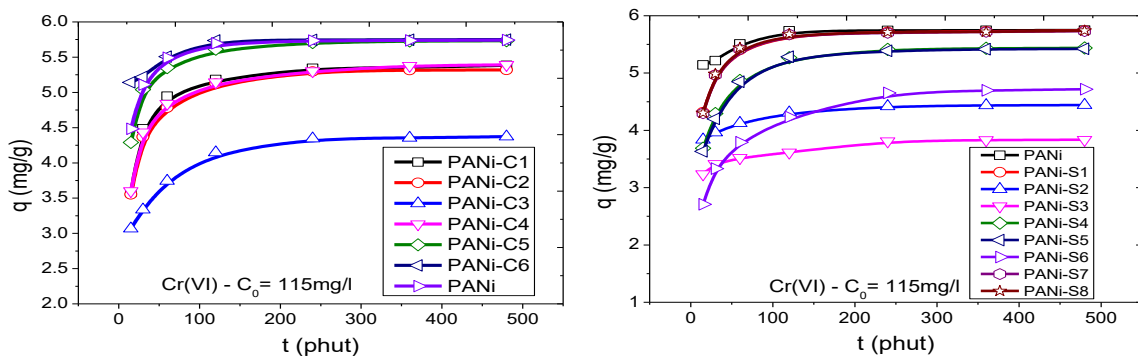


Figure 3.11. Cr (VI) adsorption capacity of PANi - plant

The tea residue, rose myrtle branches after denaturing by finely ground or activated in KOH, H₃PO₄ solution have higher adsorption capacity than non-denatured plant preparations. Smooth modified tea residues C5, C6 have the highest adsorption capacity of tea residues, at t = 480 minutes their adsorption capacity is q = 4.53 mg / g and 4, respectively. 75 mg / g. S7, S8 modified rose myrtle powder has the highest adsorption capacity among plant compositions of sim leaves, at t = 480 minutes their adsorption capacity is q = 4.74 mg/g and 4 respectively 77 mg/g.

Samples of PANi materials combined with plant preparations have higher adsorption capacity than plant samples. The Cr (VI) adsorption capacity of the original PANi samples combined with the tea residue plant composition, activated rose myrtle branches is many times higher than the adsorption capacity of As (V). PANi with activated modified tea residue PANi-C5, PANi-C6 has the highest adsorption capacity among PANi materials - tea residue, at t = 480 minutes, their adsorption capacity in turn is q = 5,73 mg/g and 5,74 mg/g. PANi combined modified rose myrtle powder PANi-S7, PANi-S8 has the highest adsorption capacity among PANi materials - rose myrtle branches and leaves, at t = 480 minutes of their adsorption capacity. As with fine tea residues, q = 5,73 mg/g and 5,74 mg/g are equivalent to adsorption efficiency of over 99%.

Time appears the equilibrium trend of most groups of materials determined at t = 120 minutes (2 hours).

3.2.3. Study on Cu (II) adsorption capacity according time

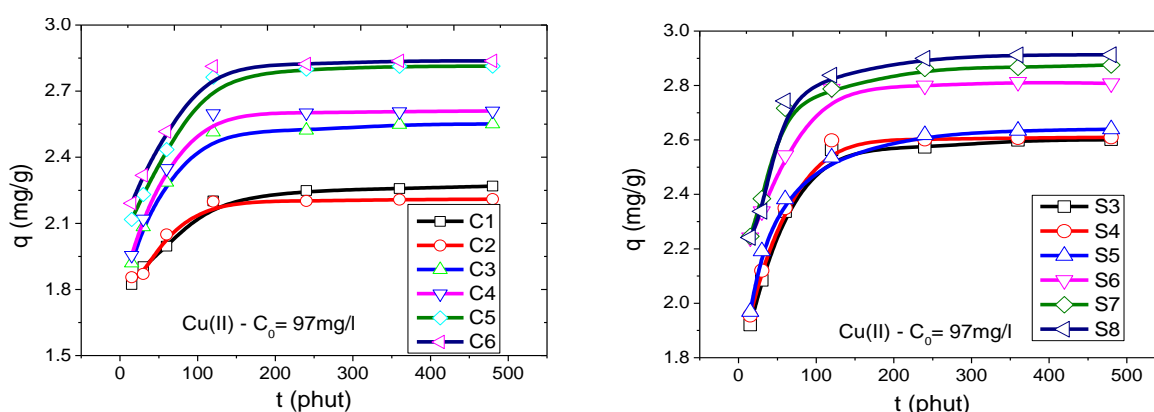


Figure 3.12. Adsorption capacity of Cu (II) of plant preparations

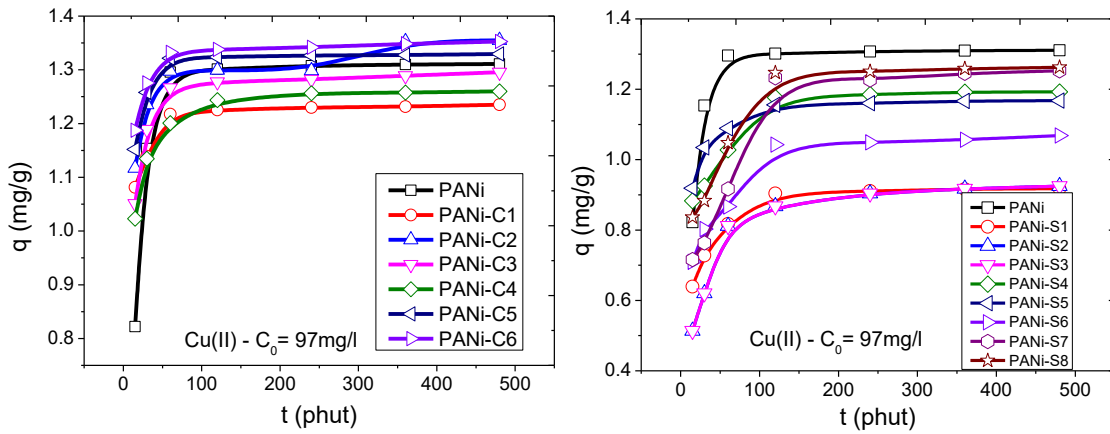


Figure 3.13. Adsorption capacity of Cu (II) of PANi - plant products

The ability to adsorb metal ions of Cu (II) of tea residue plants, rose myrtle branches and 14 samples of PANi root materials combined with tea residue preparations, sim leaves are very low. After a period of $t = 120$ minutes, the adsorption process appears to be balanced. Adsorption capacity of the material groups ranged from $0.5135 \div 2.913$ mg / g.

Through the above drawings, it is also possible to see that the plant specimens have higher adsorption capacity than the samples of PANi materials combined with plant preparations and tea residue plant compositions, rose myrtle branches after denatured by fine grinding or activating in KOH, H_3PO_4 solution has higher adsorption capacity than non-denatured plant preparations.

3.2.4. Study on Pb (II) adsorption capacity according time

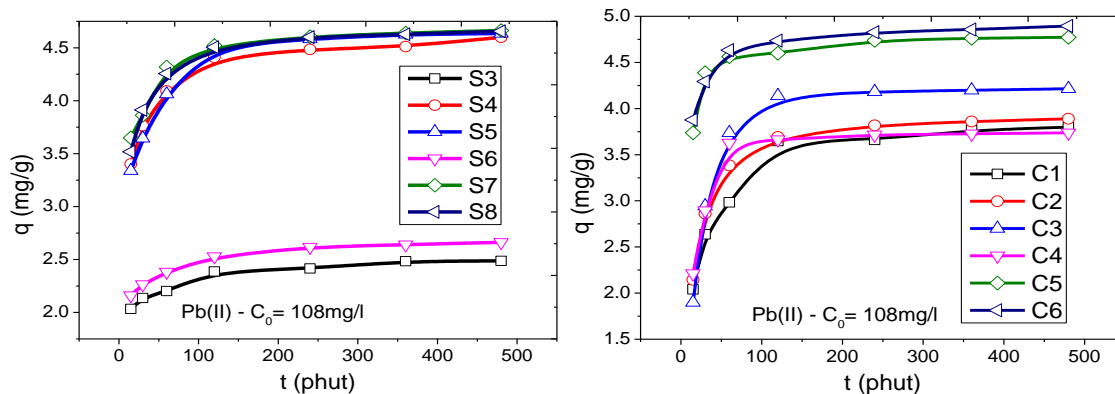


Figure 3.14. Adsorption capacity of Pb (II) of plant preparations

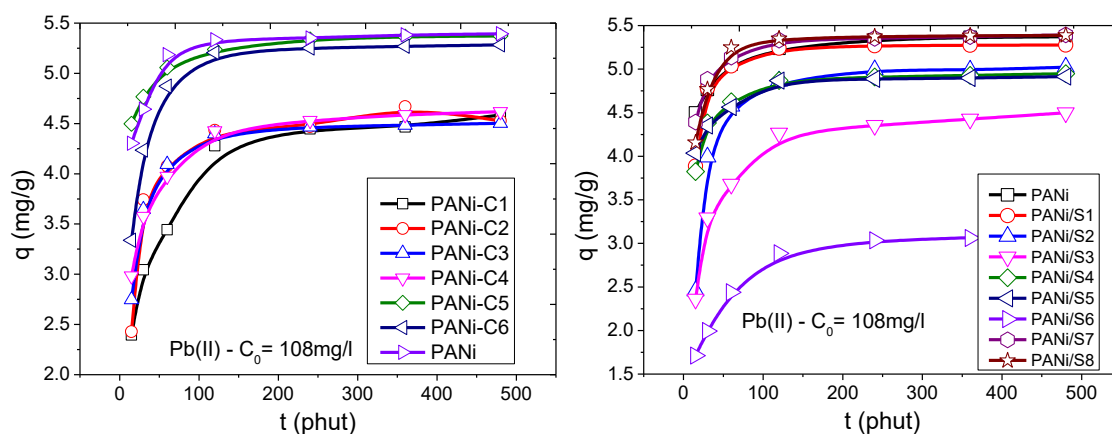


Figure 3.15. Adsorption capacity of Pb (II) of PANi - plant preparations

The tea residue, rose myrtle branches after denaturing by finely ground or activated in KOH, H_3PO_4 solution have higher adsorption capacity than non-denatured plant preparations. Modified smooth tea residues C5, C6 have the highest adsorption capacity of tea residues, at $t = 480$ minutes their adsorption capacity is $q = 4.78 \text{ mg/g}$ and 4 respectively 90 mg/g . rose myrtle powder S4, S5, S7, S8 have the highest adsorption capacity among the rose myrtle branches of leafy plants. At $t = 480$ minutes their adsorption capacity is $q = 4.60 \text{ mg/g}$ respectively 4.63 mg/g ; 4.67 mg/g and 4.66 mg/g .

The original PANi material incorporates plant preparations with higher adsorption capacity than plant preparations. Adsorption capacity of Pb (II) of samples of original materials PANi combined with tea residue plant, activated rose myrtle branches is similar to Cr (VI) adsorption capacity and many times higher than adsorption capacity of As (V), Cu (II). PANi with activated modified tea residue PANi-C5, PANi-C6 has the highest adsorption capacity among PANi materials - tea residue, at $t = 480$ minutes, their adsorption capacity in turn $q = 5.28 \text{ mg/g}$ and 5.39 mg/g . PANi combines modified rose myrtle powder of PANi-S7, PANi-S8 with the highest adsorption capacity among PANi materials - rose myrtle branches. At $t = 480$ minutes, their adsorption capacity is similar to that of fine tea residues $q = 5.28 \text{ mg/g}$ and 5.39 mg/g , equivalent to the adsorption efficiency of over 99%. Time appears the equilibrium trend of most groups of materials determined at $t = 120$ minutes (2 hours).

Thus, through the research results, it can be seen that:

➤ The activation process of tea residue and residue, rose myrtle powder has increased the surface area of the material, created many cavities and the adsorption efficiency of the material increased due to the porous, hollow structure increased in the whole block of materials. When denaturing plant residues of tea residue, rose myrtle branches by mechanical grinding, this process breaks down the raw structure of the material into many finer particles. After activating in a solution of KOH or H₃PO₄, the surface of the particles becomes porous, more porous, so the surface porosity of the material increases, but still retains the cellulose fiber structure. The hydroxyl groups on cellulose play an important role in the ion exchange capacity of lignoxene cellulose. In addition, the process of denaturing tea residue, residue/foliar pulp has led to the disruption of ligin bonds in plant activating, increasing the interaction of cellulose and hemicellulose in materials for steps. Further processing, the porosity of the material increases after the lye breaks the cross-links, releasing from the material of carboxylic acid salts and alcohol. According to the research on the structure of materials based on IR spectrum, SEM and TEM surface morphological analysis results can predict the material of tea residue and rose myrtle powder to act as a carrier. The polymer PANi layer in the synthesis process will cover the outer surface of the bearing material is tea residue, rose myrtle branches, the process is simulated as follows:

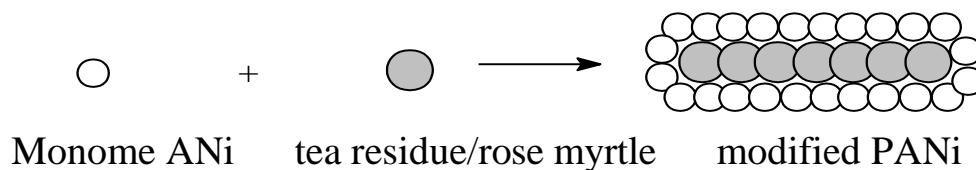


Figure 3.16. The process of forming PANi and tea residue/rose myrtle

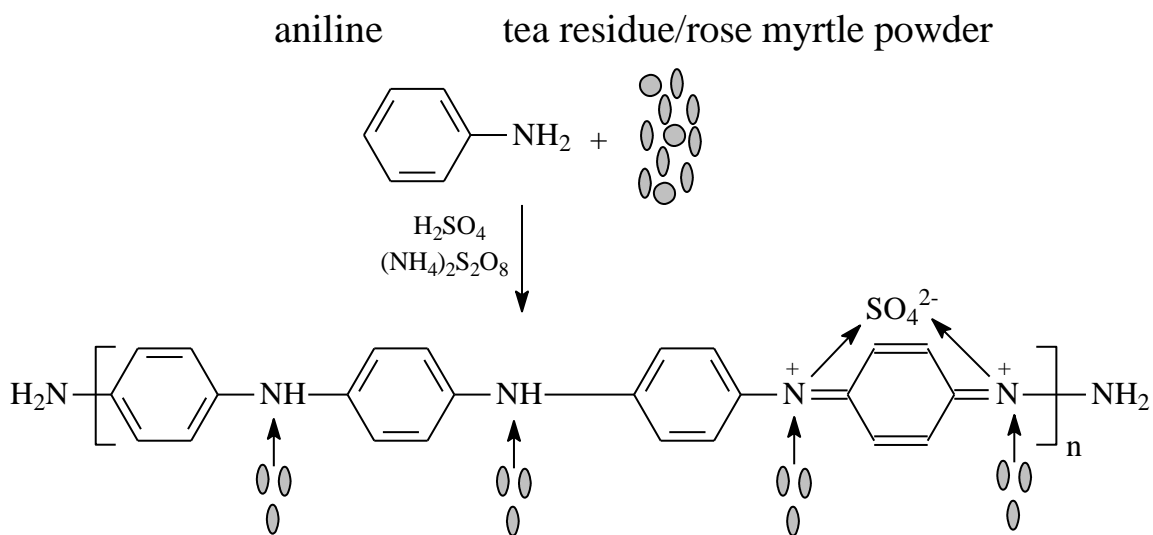


Figure 3.17. PANi combines tea residue or rose myrtle powder

➤ PANi base materials combined with plant residues after being synthesized are brought to neutral pH so the main form of PANi is ES (Emeradin salt) green and dark green. Mechanism of adsorption of anions and heavy metal ions on the surface of the original material PANi combined with tea residue, rose myrtle branches in neutral environment is proposed as follows:

- For As (V), Cr (VI): the process of physical adsorption of electrostatic interaction between the AsO_4^{3-} , CrO_4^{2-} , $\text{Cr}_2\text{O}_7^{2-}$ and PANi anions occurs. AsO_4^{3-} , CrO_4^{2-} , $\text{Cr}_2\text{O}_7^{2-}$ anions will compete instead of SO_4^{2-} in $\text{PANi}^{2+}/\text{SO}_4^{2-}$ salts.

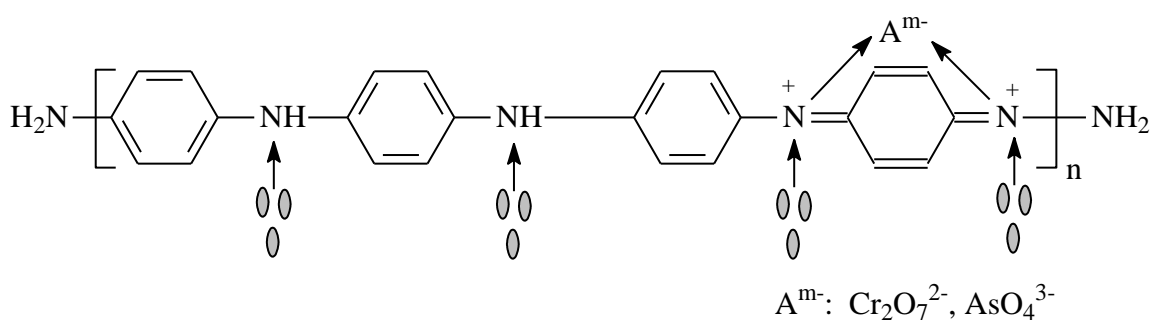


Figure 3.18. Mechanism of adsorption of As (V) and Cr (VI)

- For Cu (II), Pb (II): the process of physical adsorption of electrostatic interaction between metal ions of Cu (II), Pb (II) occurs with PANi. Metal ions will create crab complexes of metal ions with imine ($-\text{N}=\text{C}$) in PANi molecule.

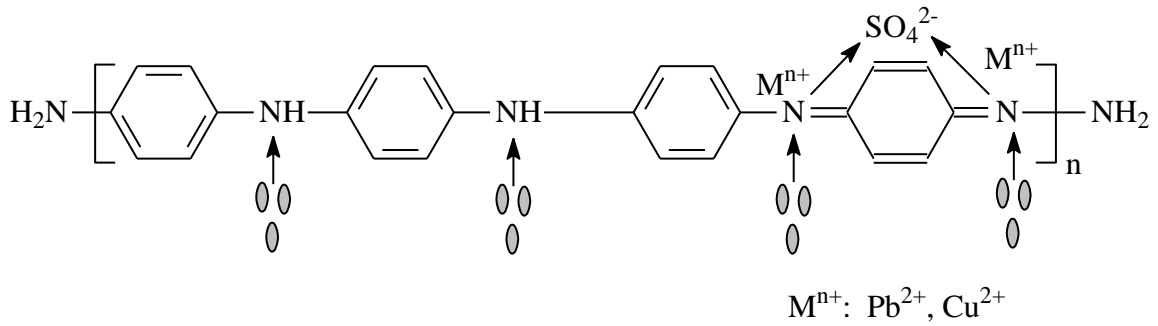


Figure 3.19. Mechanism of adsorption of metal ions Cu (II) and Pb (II)

➤ Survey results, evaluation of adsorption capacity of As (V), Cr (VI), Cu (II), Pb (II) according to the above time shows: at experimental conditions at room temperature $t = 250C$, neutral medium, volume of adsorbent $m = 2g$ in 100ml solution, adsorption capacity for Cr (VI), Pb (II) of the original PANi material samples combined with residual plant products tea, sim leaves are quite high, many times higher than adsorption capacity of As (V) and Cu (II). Therefore, Cr (VI) and Pb (II) were selected for the survey in higher concentration conditions to study applications for treatment of them in water environment.

3.3. Study on application of treatment of Cr (VI) and Pb (II) in water

3.3.1. Adsorption capacity of Cr (VI) and Pb (II) at high concentrations

3.3.1.1. For Cr (VI)

a) At the initial concentration $C_0 = 525 \text{ mg/l}$

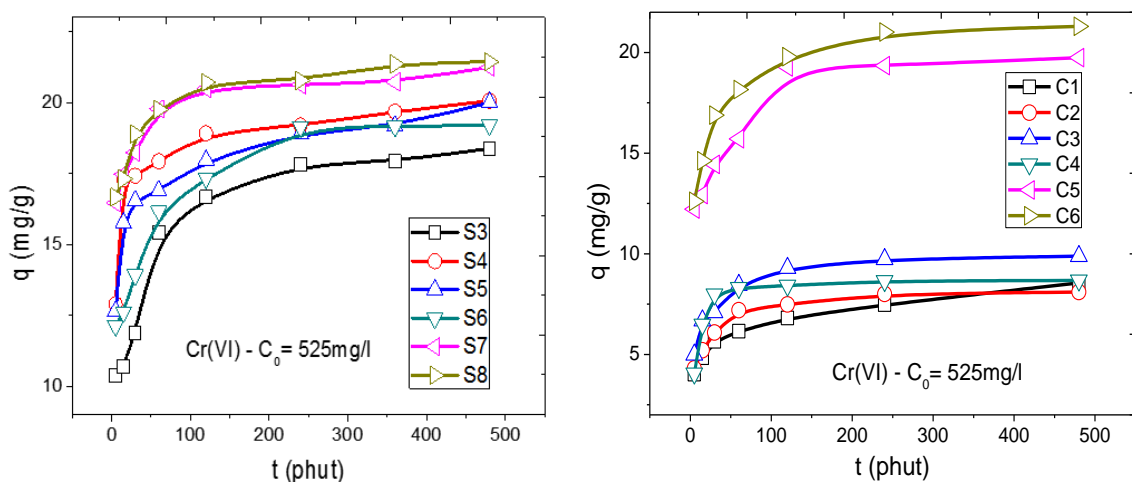


Figure 3.20. Cr (VI) adsorption capacity ($C_0 = 525 \text{ mg/l}$)

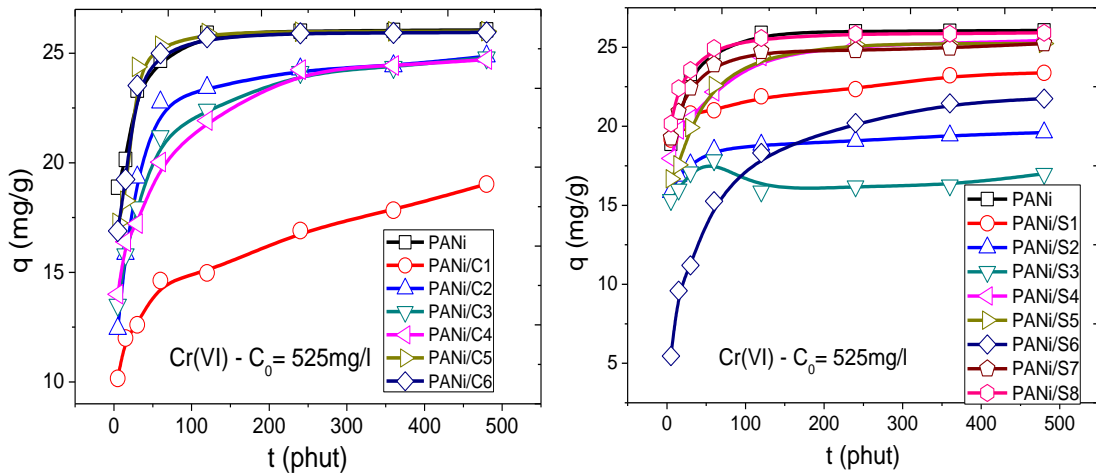


Figure 3.20. Cr (VI) adsorption capacity ($C_0 = 525 \text{ mg/l}$)

Cr(VI) adsorption capacity of 14 original PANi materials combined with tea residue, rose myrtle powder is much higher than that of raw material samples made from tea residue, rose myrtle powder. 04 samples of original PANi materials combined with tea residue preparations, rose myrtle powder in KOH/ H_3PO_4 solution give the highest efficiency and capacity of adsorption. Samples of PANi-C5, PANi-C6, PANi-S7, PANi-S8 have adsorption capacity at $t = 480$ minutes respectively 25.90 mg/g ; 26.12 mg/g ; 25.24 mg/g and 25.92 mg/g (equivalent to a performance of over 99%). Time appears the equilibrium trend of most groups of materials determined at $t = 120$ minutes (2 hours).

b) At the initial concentration $C_0 = 1075 \text{ mg/l}$

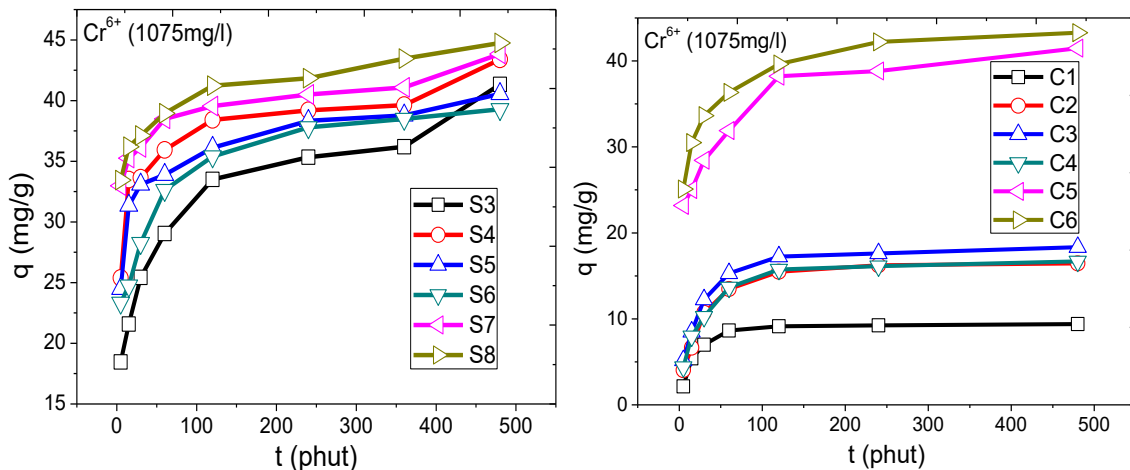


Figure 3.22. Cr (VI) adsorption capacity ($C_0 = 1075 \text{ mg/l}$)

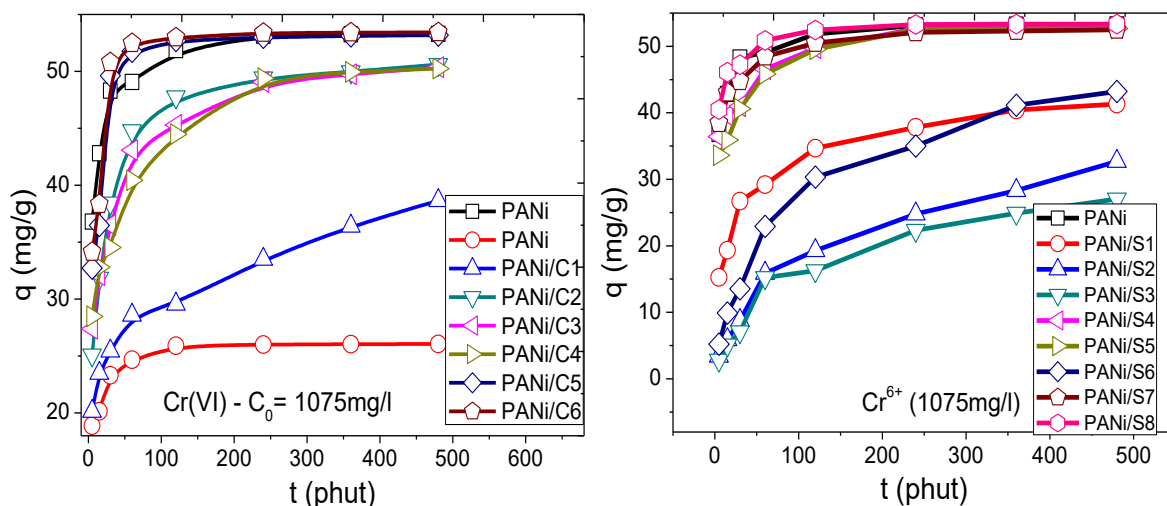


Figure 3.23. Cr (VI) adsorption capacity of PANi - plants ($C_0 = 1075$ mg/l)

The original PANi material combined with tea residue, sim leaves is much higher than the group of raw materials made from tea residue, sim leaves. 04 samples of original PANi materials combined with tea residue preparations, rose myrtle powder in KOH/H₃PO₄ solution give the highest efficiency and capacity of adsorption. Samples of PANi-C5, PANi-C6, PANi-S7, PANi-S8 have adsorption capacity at $t = 480$ minutes, respectively 53.18 mg/g; 53.43 mg/g; 52.47 mg/g and 53.35 mg/g (equivalent to an efficiency of over 99%). Time appears the equilibrium trend of most groups of materials determined at $t = 120$ minutes (2 hours).

3.3.1.2. For Pb(II)

a) At the initial concentration $C_0 = 553$ mg/l

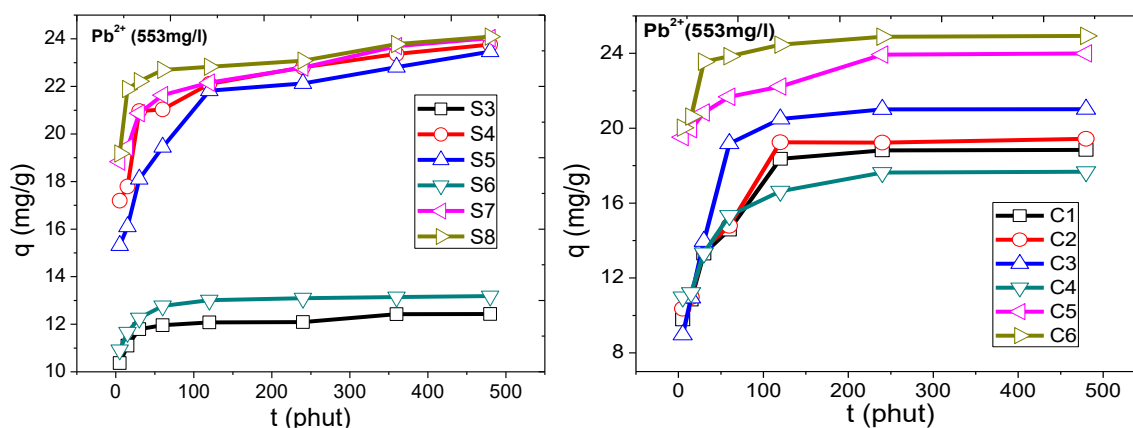


Figure 3.24. Adsorption capacity of Pb (II) ($C_0 = 553$ mg/l)

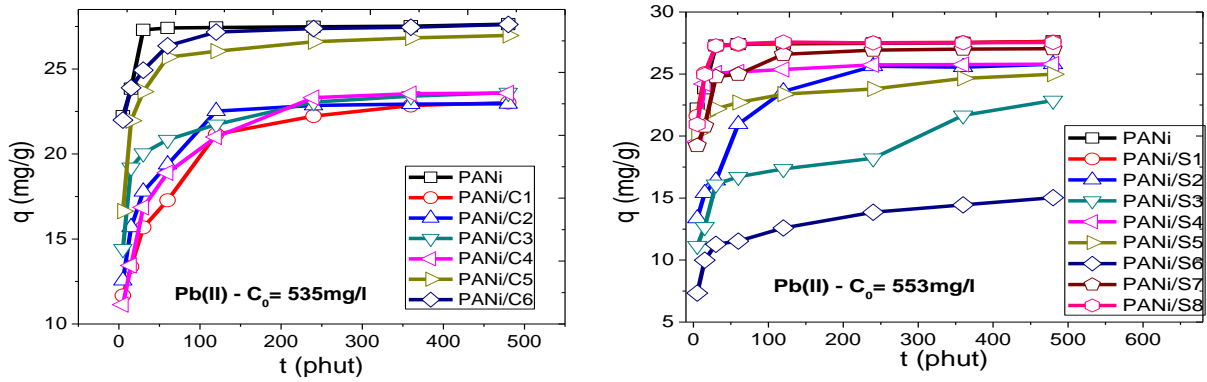


Figure 3.25. Adsorption capacity of Pb (II) ($C_0 = 553 \text{ mg / l}$)

The ability to adsorb metal ions Pb (II) of 14 original materials PANi combined with tea residue, rose myrtle branches are much higher than that of samples of raw materials made from tea residue, rose myrtle. 04 samples of original PANi materials combined with tea residue preparations, rose myrtle in KOH/H₃PO₄ solution give the highest efficiency and capacity of adsorption. Samples of PANi-C5, PANi-C6, PANi-S7, PANi-S8 have adsorption capacity at t = 480 minutes, respectively 26.98 mg/g; 27,62 mg/g; 27.05 mg/g and 27.54 mg/g (equivalent to a performance of over 99%). Time appears the equilibrium trend of most groups of materials determined at t = 120 minutes (2 hours).

b) At the initial concentration $C_0 = 996 \text{ mg/l}$

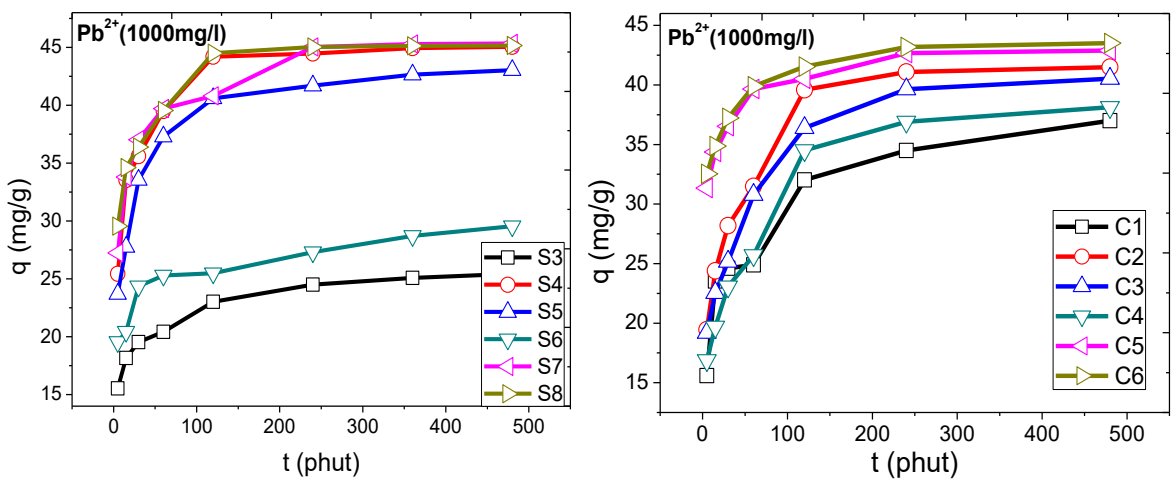


Figure 3.26. Adsorption capacity of Pb (II) ($C_0 = 996 \text{ mg/l}$)

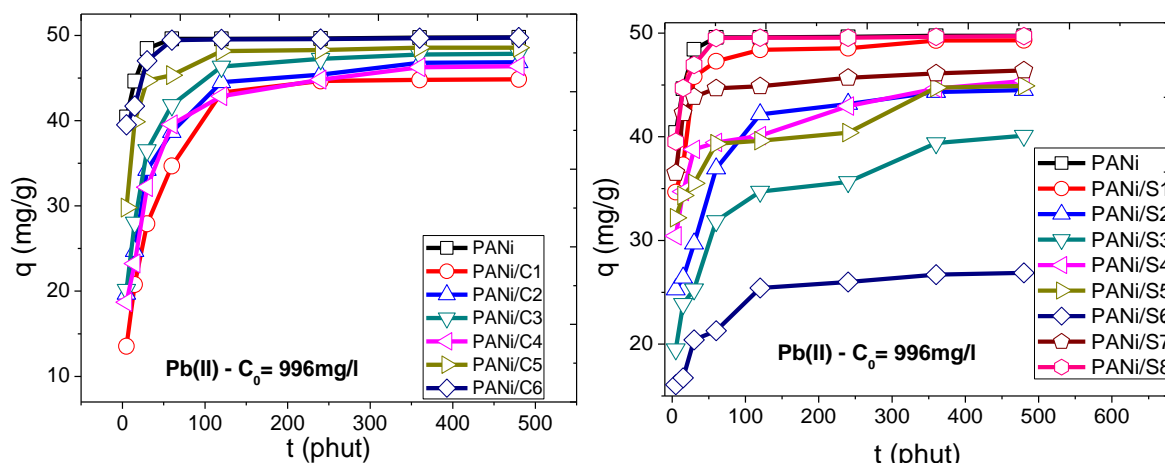


Figure 3.27. Adsorption capacity of Pb (II) ($C_0 = 996 \text{ mg/l}$)

The ability to adsorb metal ions Pb (II) of 14 original materials PANi combined with tea residue, rose myrtle branches are much higher than that of samples of raw materials made from tea residue, rose myrtle branches. 04 samples of PANi original materials combined with tea residue preparations, modified rose myrtle branches in KOH/ H_3PO_4 solution for the highest efficiency and adsorption capacity. Samples of PANi-C5, PANi-C6, PANi-S7, PANi-S8 have adsorption capacity at $t = 480$ minutes respectively 48.55 mg/g ; 49.75 mg/g ; 46.40 mg/g and 49.71 mg/g (equivalent to performance of over 99%). Time to reach the equilibrium trend of most materials is determined at $t = 120$ minutes.

3.3.2. Affected factors for the adsorption capacity of Cr (VI), Pb (II)

3.3.2.1. Effect of concentration

a) For Cr(VI)

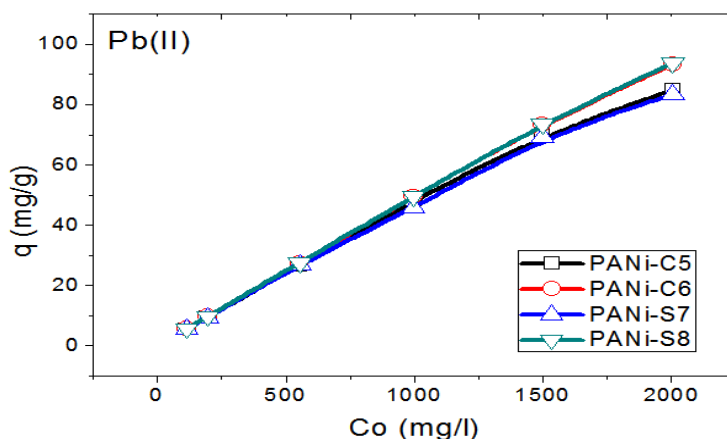


Figure 3.28. Effect of initial concentration on Cr (VI)

We found that the adsorption capacity of 04 samples of PANi original materials combined with tea residue, rose myrtle branches of leaves and leaves were increased when the initial concentration of Cr (VI) solution increased. By the concentration range of $C_0 = 2000 \text{ mg / l}$, the adsorption capacity tends to be stable and increase more slowly.

b) For Pb(II)

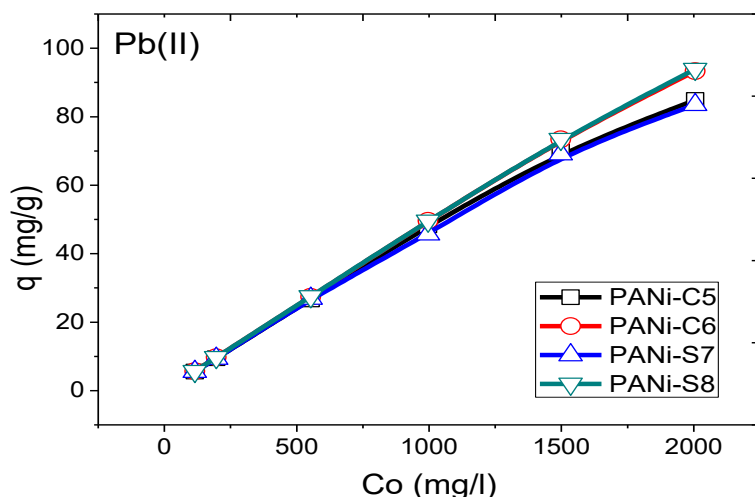


Figure 3.29. Effect of initial concentration on Pb(II) adsorption capacity

Similar to the case of Cr (VI), adsorption capacity of 04 samples of original PANi materials combined with tea residue, rose myrtle branches of the selected leaves increased when the initial concentration of Pb solution (II) increase. In the $C_0 = 2000 \text{ mg/l}$ concentration range, adsorption capacity tends to be stable and increase more slowly.

3.3.2.2. Effect of pH environment

- For Cr(VI)

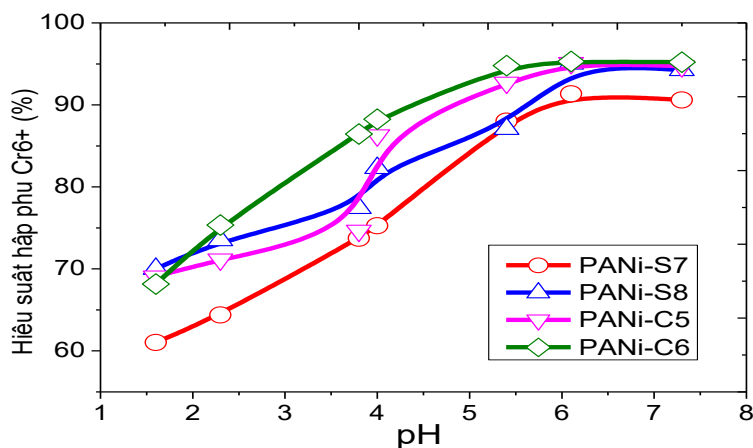


Figure 3.30. Effect of pH environment on adsorption capacity of Cr (VI)

- For Pb(II)

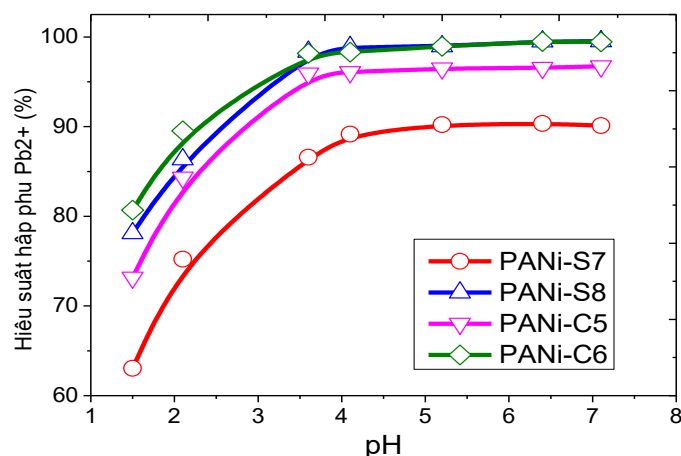
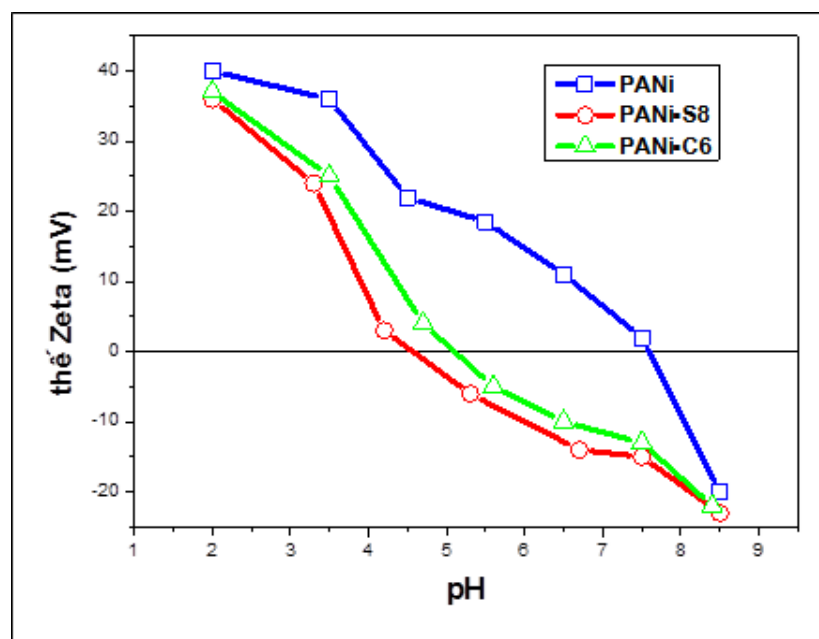


Figure 3.31. Effect of pH environment on adsorption capacity of Pb (II)

Based on the graph in Figure 3.30, it can be seen that the Cr (VI) adsorption efficiency increases with increasing pH. Adsorption efficiency reached the highest value at $\text{pH} = 5 \div 7.5$, with many materials, Cr (VI) adsorption efficiency reached over 95%. At $\text{pH} < 5$ in acidic environment, the adsorption efficiency is lower, only 60-70% for the adsorbent used. Similarly, Figure 3.31 can be seen that at the pH range of $4.5 \div 6.5$, the adsorption efficiency of Pb (II) reaches the highest value, when the pH is at the $\text{pH} < 4$ of strong acid environment, then poor adsorption performance.

According to the above argument, the adsorption mechanism of the original PANi materials combined with tea residue preparations, rose myrtle are thought to be electrostatic interaction so to investigate and explain the process of pH influence to The adsorption capacity of the material experiments determining the isoelectric point of the adsorbent by measuring the Zeta potential was carried out. The Zeta potential results determine the isoelectric point shown in Figure 3.40 below:



Hình 3.32. Đồ thị xác định điểm đẳng điện của vật liệu gốc PANI

From Figure 3.32, it can be seen that the isoelectric points of PANi-S8 and PANi-C6 adsorbents range from $4.6 \div 5.1$. Specifically, the isoelectric point of PANi-S8 material is $pH_{pzc} = 4.6$ and of PANi-C6 material is $pH_{pzc} = 5.1$. This shows that when $pH < pH_{pzc}$, the surface adsorption of positive charge, when $pH > pH_{pzc}$, the adsorption surface will be negatively charged. This is consistent with the results of surveying the effect of pH on Pb (II), Cr (VI) adsorption capacity of two samples of materials. When $pH < 4.5$ PANi base materials are positively charged so the adsorption efficiency is low and when the pH value increases the surface of synthetic materials PANi-S8, PANi-C6 negatively charged, gradually leading to increase adsorption effect.

So it can be concluded that the pH environment which be the most favorable for the adsorption capacity of Cr (VI) and Pb (II) of 04 samples of PANi materials combined with tea residue plant or rose myrtle branches is neutral and weak acids with a pH range between $5.5 \div 7.5$.

3.3.2.3. Effect of mass of adsorbent

a) For Cr(VI)

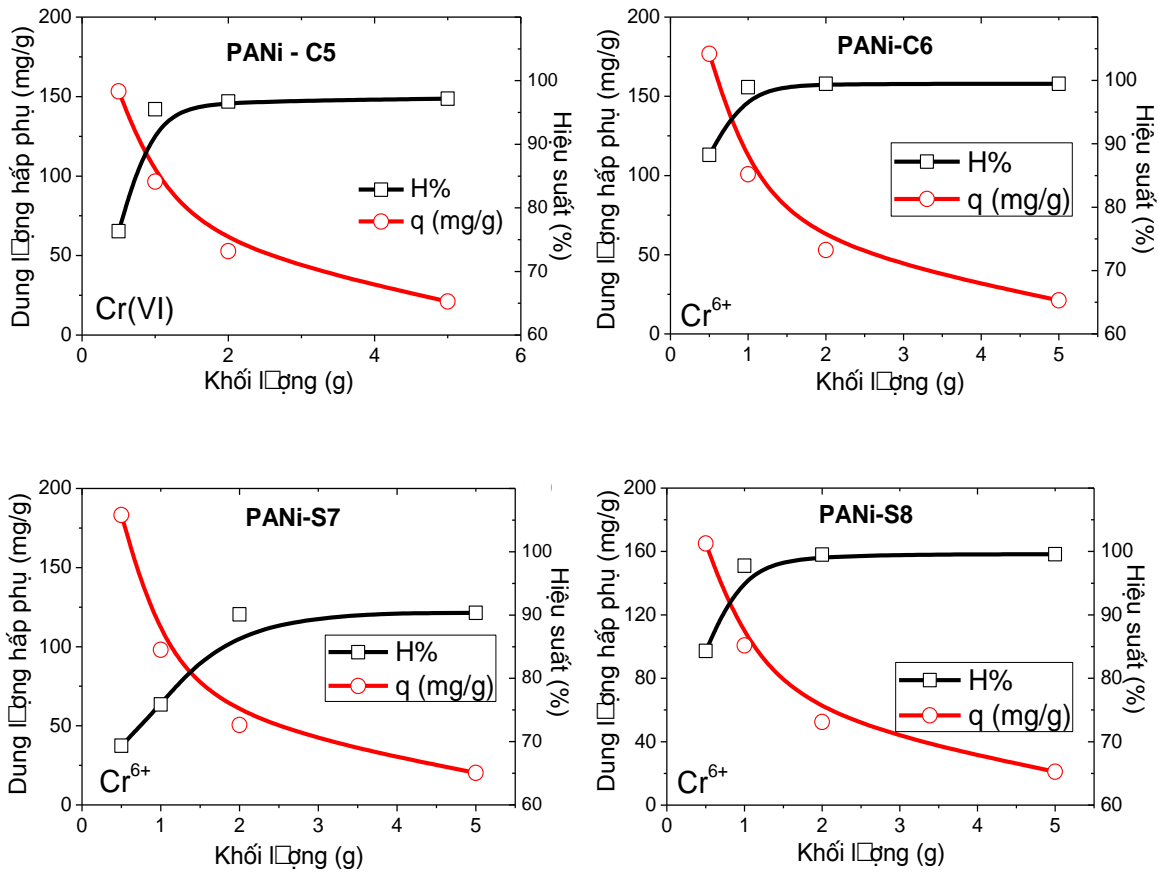


Figure 3.33. Effect of material volume on Cr (VI) adsorption capacity

Cr (VI) adsorption efficiency of 04 samples of PANi root materials increased with increasing of adsorbent volume from 0.5 g ÷ 2 g. As we continue to increase the mass of materials from 2 g ÷ 5 g we see that the adsorption efficiency does not increase much, which indicates that the adsorption process has reached equilibrium and when the mass of the material continues to increase, the efficiency of adsorption process will not increase much. Along with the increasing process of adsorption efficiency, we find that the adsorption capacity will be inversely proportional to the adsorption efficiency of the original PANi materials combined with the plant residue of tea residue or rose myrtle branches. The amount of material that fluctuates within $m = 1 \div 2$ g is the optimal choice to carry out the Cr (VI) adsorption process using the PANi materials in combination with the composition at the same conditions as the experimental conditions. experience.

b) For Pb(II)

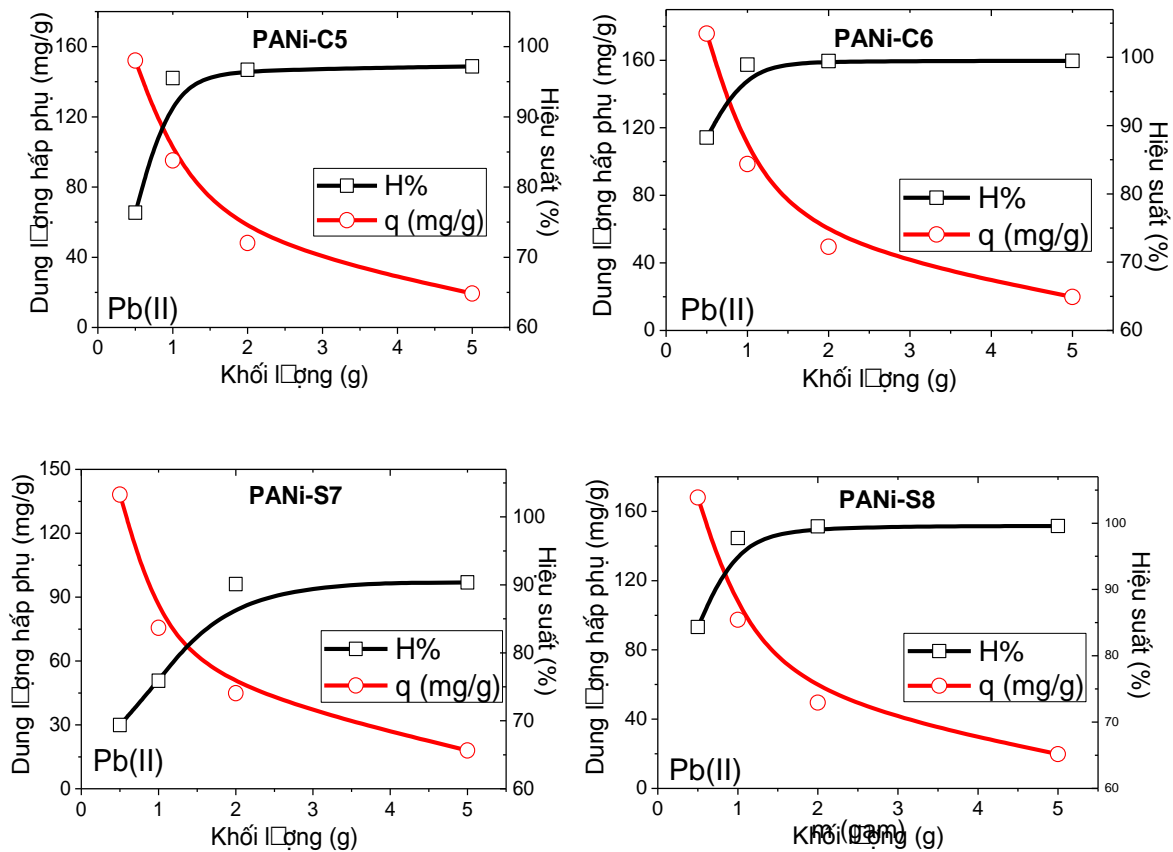


Figure 3.34. Effect of material weight on adsorption capacity of Pb (II)

Similar to Cr (VI), the adsorption efficiency of Pb (II) increases with increasing weight of adsorbent from 0.5 g ÷ 2 g. As we continue to increase the mass of materials from 2 g ÷ 5 g we see that the adsorption efficiency does not increase much, which indicates that the adsorption process has reached equilibrium and when the mass of the material continues to increase. adsorption, the efficiency of adsorption process will not increase much. The amount of material that fluctuates in the range of $m = 1 \div 2$ g is the optimal choice to carry out the adsorption process of Pb (II) using the materials PANi combined with the composition at the same condition as the experimental conditions. experience.

3.4. Study of isotherm adsorption models of selected materials

3.4.1. Langmuir isothermal model

a) For Cr(VI)

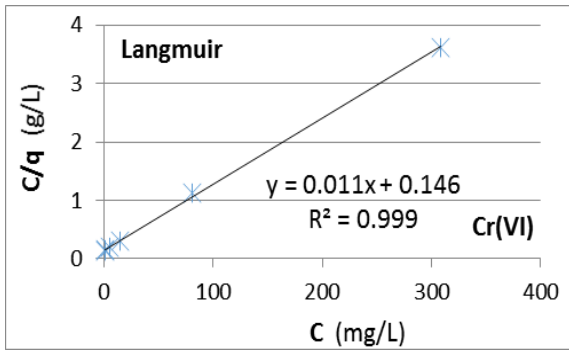


Figure 3.35. Graph of Langmuir isothermal adsorption model of PANi-C5

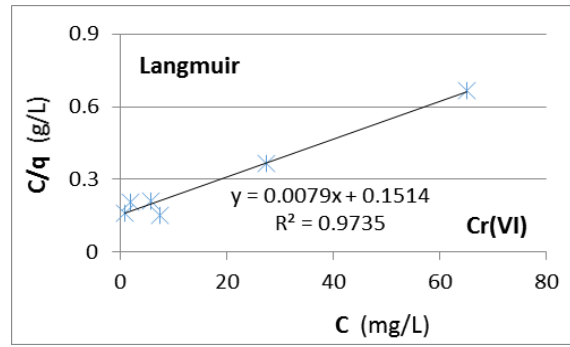


Figure 3.36. Graph of Langmuir isothermal adsorption model of PANi-C6

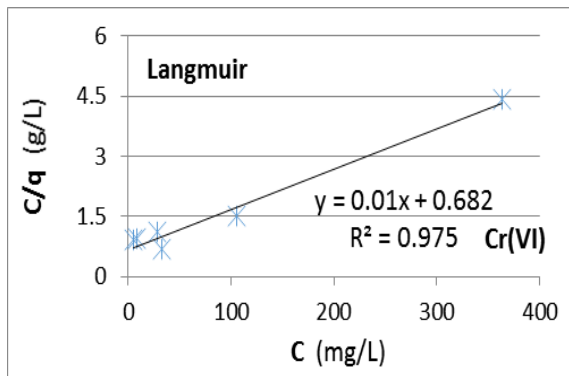


Figure 3.37. Graph of Langmuir isothermal adsorption model of PANi-S7

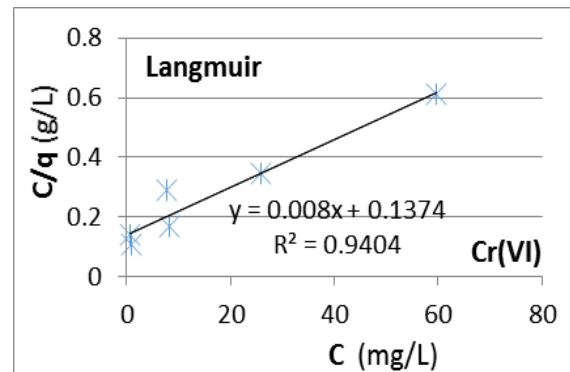


Figure 3.38. Graph of Langmuir isothermal adsorption model of PANi-S8

Table 3.1. Parameters of Langmuir isothermal adsorption model for Cr (VI)

Name	Linear equation	R ²	q _{max} (mg/g)	K _L (l/mg)	R _L
PANi-C5	y = 0,011x + 0,146	0,999	90,91	0,075	0,006÷0,1035
PANi-C6	y = 0,0079x + 0,1514	0,9735	126,58	0,052	0,0094÷0,1428
PANi-S7	y = 0,01x + 0,682	0,975	100,0	0,015	0,0322÷0,3728
PANi-S8	y = 0,008x + 0,1374	0,9404	125,0	0,058	0,0085÷0,1299

b) For Pb(II)

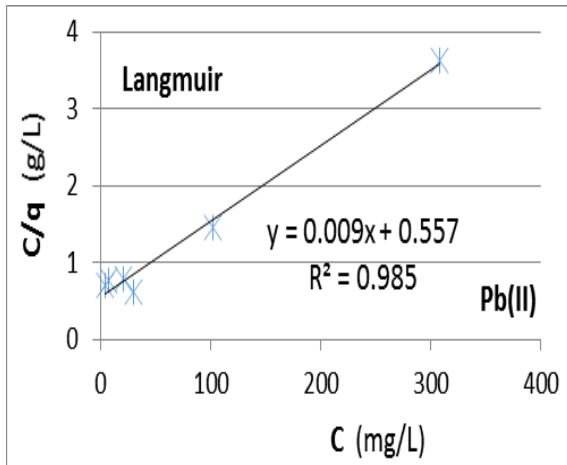


Figure 3.39. Graph of Langmuir isothermal adsorption model of PANi-C5

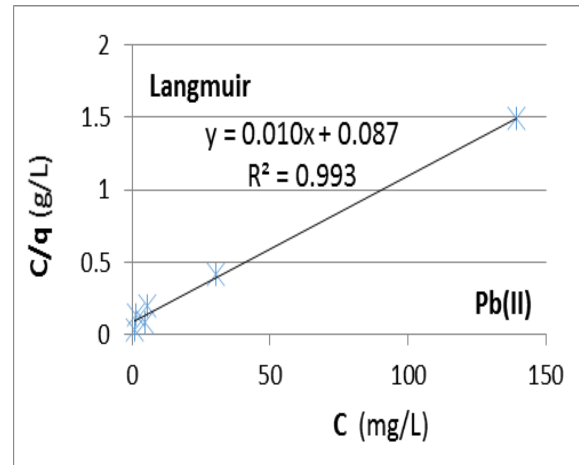


Figure 3.40. Graph of Langmuir isothermal adsorption model of PANi-C6

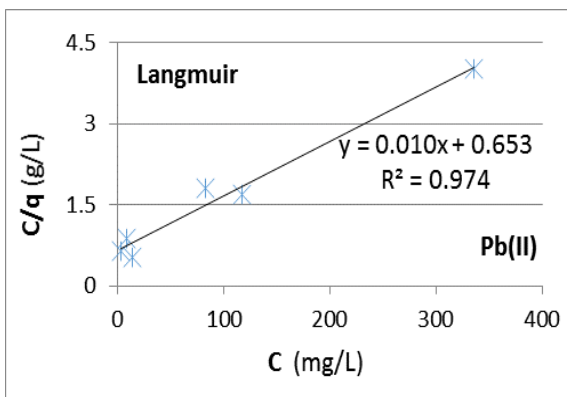


Figure 3.41. Graph of Langmuir isothermal adsorption model of PANi-S7

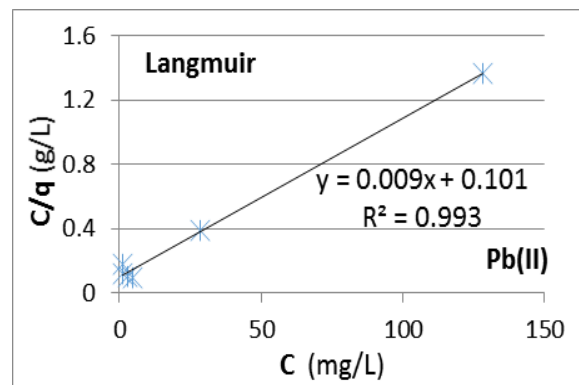


Figure 3.42. Graph of Langmuir isothermal adsorption model of PANi-S8

Table 3.2. Parameters of Langmuir isothermal adsorption model for Pb(II)

Name	Linear equation	R ²	q _{max} (mg/g)	K _L (L/mg)	R _L
PANi-C5	y = 0,009x + 0,557	0,985	111,11	0,016	0,0298÷0,349
PANi-C6	y = 0,010x + 0,087	0,993	100,0	0,115	0,0043÷0,0703
PANi-S7	y = 0,010x + 0,653	0,974	100,0	0,015	0,0314÷0,362
PANi-S8	y = 0,009x + 0,101	0,993	111,11	0,089	0,0055÷0,0889

3.4.2. Freundlich isothermal model

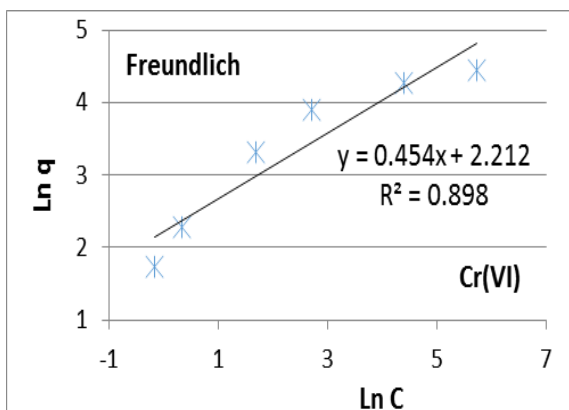


Figure 3.43. Graph of Freundlich isothermal adsorption model of PANi-C5

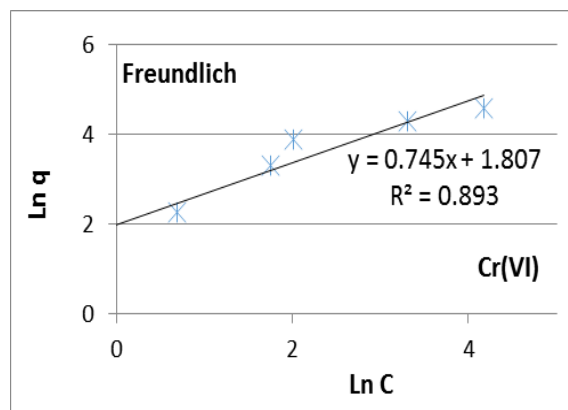


Figure 3.44. Graph of Freundlich isothermal adsorption model of PANi-C6

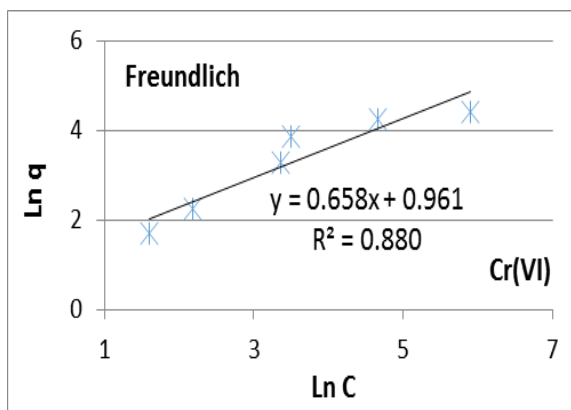


Figure 3.45. Graph of Freundlich isothermal adsorption model of PANi-S7

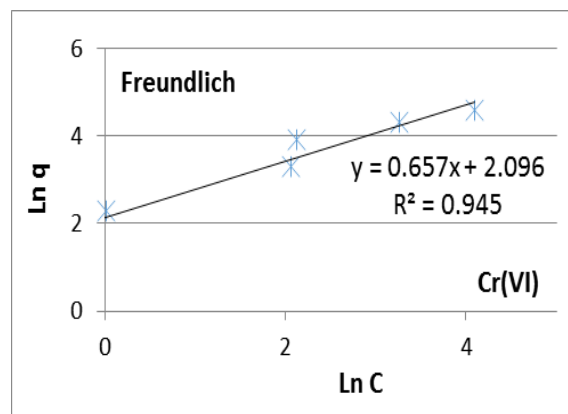


Figure 3.46. Graph of Freundlich isothermal adsorption model of PANi-S8

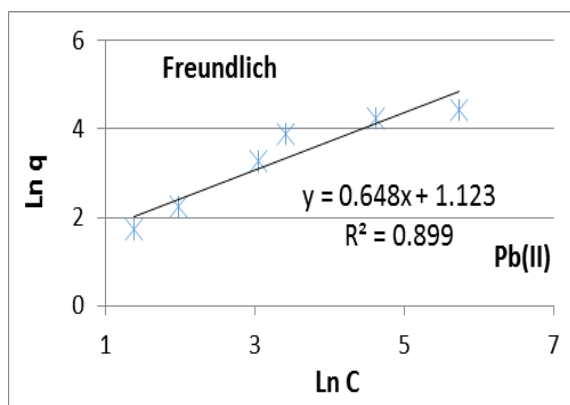


Figure 3.47. Graph of Freundlich isothermal adsorption model of PANi-C5

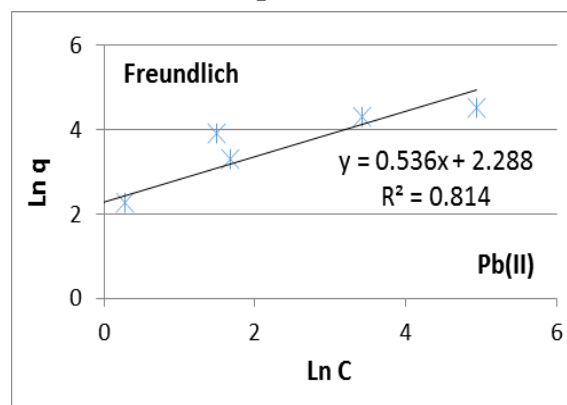


Figure 3.48. Graph of Freundlich isothermal adsorption model of PANi-C6

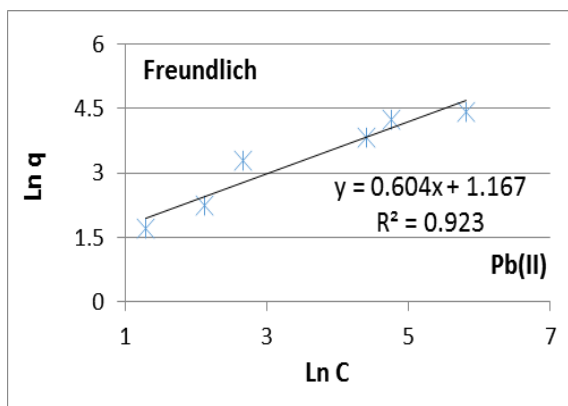


Figure 3. 49. Graph of Freundlich isothermal adsorption model of PANi-S7

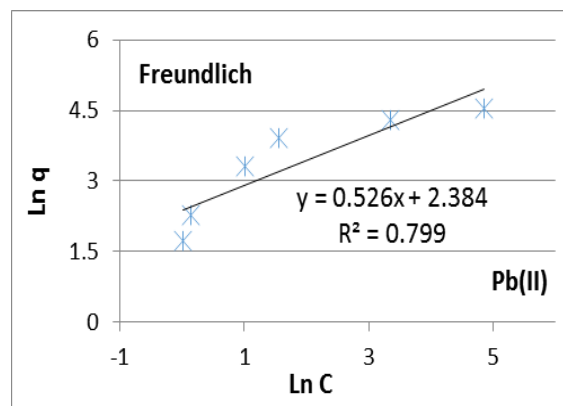


Figure 3. 50. Graph of Freundlich isothermal adsorption model of PANi-S8

Freundlich model equation:

$$\ln q = \ln K_F + 1/n \cdot \ln C$$

Table 3.3. Parameters of Freundlich isothermal adsorption model

Name	Linear equation	R ²	n	K _F
Cr(VI)				
PANi-C5	y = 0,454x + 2,212	0,898	2,2026	9,1339
PANi-C6	y = 0,745x + 1,807	0,893	1,3422	6,0921
PANi-S7	y = 0,658x + 0,961	0,880	1,5197	2,6143
PANi-S8	y = 0,657x + 2,096	0,945	1,5221	8,1335
Pb(II)				
PANi-C5	y = 0,648x + 1,123	0,899	1,5432	3,0741
PANi-C6	y = 0,536x + 2,288	0,814	1,8657	9,8552
PANi-S7	y = 0,604x + 1,167	0,923	1,6556	3,2123
PANi-S8	y = 0,526x + 2,384	0,799	1,9011	10,8482

3.5. Study on application of treatment of Pb (II), Cr (VI) in wastewater

3.5.1. Researching and applying PANi original materials to choose wastewater treatment

3.5.2. Proposing solutions to apply materials for Cr (VI) and Pb (II) treatment

CONCLUSION

1. The sample of tea residue and rose myrtle leaves are collected and applied. The rose myrtle samples are extracted in water and ethanol 96° to

obtained the S1 and S2 samples.

2. The raw plant materials are processed and modified by pulverisation and activation with KOH, H₃PO₄. After modification, the adsorption capability of the plant materials reach much higher value on As(V), Cr(VI), Cu(II), Pb(II) ions. This result is consistent with the outlined assumptions while study the characterized structure by SEM and BET.

3. The 14 PANi-based materials with plant products are synthesized including: 06 samples of PANi – tea residue (PANi-C1÷PANi-C6) and 08 samples of PANi – rose myrtle (PAN-S1÷PANi-S8). The characterized structure study are studied by IR, SEM, TEM, BET technique, result showed that the plant products take the role of carrier in the PANi-based materials. After processing, the PANi is synthesized and covered the surface of the tea residue / rose myrtle powder, the obtained products has relatively homogeneous surface, fiber-shaped, containing uniform and multi-layered pores and overlapping on each other with a size of about 50 ÷ 200 nm. The capillary size of the modified materials ranges from 27.68 ÷ 39.93 nm, belonging to the medium capillary material (mesopore) group.

4. The adsorption capability of the raw materials (S3÷S8, C1÷C6) and 14 samples of PANi-based materials with plant products on As(V), Cr(VI), Cu(II), Pb(II) over time are evaluated. Based on the results, the 04 samples PANi-C5, PANi-C6, PANi-S7, PANi-S8 with the highest adsorption capability on Cr(VI), Pb(II) have been selected to applied in further study. The effect of some factors such as initial concentration, quantity of adsorbent, pH environment on adsorption efficiency of the 04 selected materials were studied. The adsorption efficiency of these 04 materials on Cr(VI), Pb(II) ranges from 90÷99%. With the solution of Cr(VI), Pb(II) 1000mg/l, in order to efficiency adsorption, the optimal quantity of adsorbent used for treatment the volumn of 100ml is about $m = 1 \div 2$ g, and the favor pH of the environment is neutral and weak acid from 5,5 ÷ 7,5.

5. The adsorption isotherm of 04 selected marterials on Cr(VI), Pb(II) was studied, results shown that the Langmuir adsorption model was more consistent than the Freundlich model. It could be assumed that Cr(VI), Pb(II)

ions are adsorbed on the active center on the surface of the PANi-based material and this process is monolayer adsorption.

6. Following the results, 02 materials of PANi-C6, PANi-S8 have been practically applied to the treatment of Cr(VI) contaminated plated sewage with good efficiency. Several recommendations have been proposed in applying these materials on wastewater contaminated with Cr(VI), Pb(II) ions, as well as directions for further studies.

PUBLICATION LIST

1. **Tran Thi Ha**, Vo An Quan, Nguyen Quang Hop, Tran Minh Thuy, Nguyen Thi Ngoc, Le Xuan Que (2018). *Studying on the adsorption of chromium(VI) on polyaniline modified with activated tea residue*, Journal of Chemistry, Vol 56 (5), p. 559-563, DOI: 10.1002/vjch.201800047
2. **Tran Thi Ha**, Vo An Quan, Nguyen Quang Hop, Le Xuan Que (2017). Studying of adsorption model of chromium (VI) on polyaniline modified with activated rose myrtle powder and tea residue, *Proceedings The 6th Asian Symposium on Advanced Materials*, p.321-325.
3. **Tran Thi Ha**, Vu Minh Tan, Do Thi Viet Huong, Le Xuan Que (2017). *Studying kinetics and mechanism of Cr(VI) absorption by PANi materials synthesized with Rhodomyrtus tomentosa water extracts*, Journal of Analytic Sciences, No-22 (2), p.53-58.
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5. **Tran Thi Ha**, Nguyen Quang Hop, Hoang Van Hoan, Le Xuan Que (2016). *Evaluating the adsorption capability of Cu(II), Pb(II), Cr(VI) in aqueous solution of modified Polyaniline with rose myrtle*, Journal of Science Hanoi National University: Natural Science & Technololy, No. 32, Vol. 4, p.1-6.