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# Nature Environment and Pollution Technology

**Vol. 21, No. (3), September 2022**

## CONTENTS

1. **Zoyem Gouafo Mathurin, Talla Pierre Kisito, Ngapgue Francois and Médard Fogue,** Risk Assessment of Chemical Pollution of Industrial Effluents from a Soap Production Plant 931-940
2. **S. Isworo, S. Febrianto, T. Aji, P. S. Oetari and E. Jasmiene,** The Study of Air Quality and Risk Assessment at the Location of the Planned Railroad Between Makassar-Parepare, South Sulawesi, Indonesia 941-950
3. **Rohan Jeffry Robert and C. R. Girish,** Pork Lard Derived Biodiesel Production: Characterization, Engine Performance and Emission Analysis 951-970
4. **G. C. Saha, M. A. Hasanath, M. N. Uddin and M. Hasan,** Sustainable Utilization of Textile Dyeing Sludge and Coal Fly Ash by Brick Production Through Traditional Kilns 971-980
5. **P. P. Nandusekar, U. S. Mukkannawar, R. G. Jaybhaye, U. D. Kulkarni and P. N. Kamble,** Evaluation of Source Emissions Dispersion Potential Near a Coastal Village of Maharashtra, India 981-992
6. **Yeshi Choden, Kinley Pelzang, Abeshik Dev Raj Basnet and Krishna Bdr Dahal**, Modeling of Leachate Generation from Landfill Sites 993-1002
7. **K. Harshala and N. D. Wagh,** Use of Agricultural Waste-Based Biosorbents for the Removal of Heavy Metals from Aqueous Solution: A Review 1003-1014
8. **Kai Su, Hongyun Chen and Cuili Gan,** Spatial Differentiation and Dynamic Evolution of Agricultural Carbon Emissions in Fujian Province of China 1015-1025
9. **Xiaoping Yan, Leixiang Wu, Jun Xie, Yongqian Wang, Chencheng Wang and Bing Ling,** Dynamic Changes and Precision Governance of Soil Erosion in Chengde City Using the GIS Techniques and RUSLE Model 1027-1037
10. **Abhinav Joseph, Pawan Gupta, Gahin De, Manohar Lal, Mukesh Kumar Meena, Laliteshwar Pratap Singh and Jyotsna Rattan,** Biodegradation of Natural Rubber by Fungi and Bacteria 1039-1048
11. **Fangbo Zhang, Zhiqin Li, Lei Tang, Jingting Wang, Chao Shen, Xiaojia He, Ran Li, Jingjie Feng and Naiwen Li,** Effects of Submerged Macrophyte Decomposition on Water Quality 1049-1058
12. **S. Saha, A. K. Bhagabati and R. Thakur,** Assessment of Drinking Water Accessibility and Quality in the Indo-Bhutan Himalayan Foothill Region of Assam, India 1059-1071
13. **Da Li, Zhonghua Yang, Junjie Zheng, Fangping Liu and Gang Ge,** The Influence of Floodplain Vegetation Patches on Hydrodynamic Characteristics: A Case Study in the Old Course of Fuhe River 1073-1085
14. **P. Devesh, B. Aishwarya and R. Gyana Prasuna,** Studies on the Isolation of Lipids from Mangrove Isolated Cyanobacterial Species 1087-1096
15. **Akhilesh Kumar Dewangan, Isham Panigrahi and R. K. Paramguru,** An Investigation of a Hybrid Plasma Gasification System for Various Waste Plastics Thermochemical Degradation in the Fuel Extraction Process 1097-1112
16. **Yang Hu and Zhongqiu Sun,** Assessing the Capacities of Different Remote Sensors in Estimating Forest Stock Volume Based on High Precision Sample Plot Positioning and Random Forest Method 1113-1123
17. **Wenjie Yu, Biao Qian, Beifeng Lv, Haibo Kang, Ping Jiang, Wei Wang and Na Li,** Efficient Adsorptive Performance of Graphene Oxide by Nano Clay 1125-1135
18. **A. Choudhary, P. Kumar, S. K. Sahu, C. Pradhan, S. K. Singh, M. Gašparovi, A. Shukla and A. K. Singh,** Time Series Simulation and Forecasting of Air Quality Using In-situ and Satellite-Based Observations Over an Urban Region 1137-1148
19. **Ramzi H. Amran, Mamdoh T. Jamal, Arulazhagan Pugazhendi, Mamdouh Al-Harbi, Mohammed Ghandourah, Ahmed Al-Otaibi and Md Fazlul Haque,** Biodegradation and Bioremediation of Petroleum Hydrocarbons in Marine Ecosystems by Microorganisms: A Review 1149-1157
20. **R. Megha, Divya Murali, S. Amblikuttan, Rajesh Reghunath and K. Anoop Krishnan,** Palaeoclimatic Studies of the Late Quaternary Sediments from Chirakkara, Kollam District, Kerala, India 1159-1165
21. **C. P. Del Río-Galván, R.C. Hernández-León, M.O. Franco-Hernández and J. Meléndez-Estrada,** Primary Sewage Sludge Treatment Using A Spiral Support System 1167-1174
22. **Deepali Rajwade, Sadhana Jaiswal, Vinit Singh Baghel and Hemant Kumar,** Isolation, Identification and Characterization of Novel Azo Dye Degrading Bacteria from the Industrial Effluents of Raipur City, Chhattisgarh 1175-1182
23. **M. M. Sari, I. W. K. Suryawan, B. S. Ramadan, I. Y. Septiariva and S. Notodarmojo,** Marine Debris Management in the Parangtritis Beach Tourism Area, Yogyakarta During Covid-19 Pandemic 1183-1190
24. **Quratulain Maqsood, Esha Ameen, Muhammada Mahnoor, Aleena Sumrin, Muhammad Waseem Akhtar, Riya Bhattacharya and Debajyoti Bose,** Applications of Microbial Fuel Cell Technology and Strategies to Boost Bioreactor Performance 1191-1199
25. **Sudhir Kumar Chaturvedi,** Landslide Assessment Using Sentinel-I SAR-C Interferometry Technique 1201-1207
26. **T. R. Shanthi, Mohammed Hatha and T. R. Satyakeerthy,** A Study on the Diversity of Pesticide-Resistant Bacterial Population from Different Agricultural Fields of Manjoor 1209-1216
27. **I. Benali, M. Bouderbala and N. Chèvre,** Study on the Establishment of the Gastropod *Lymnaea stagnalis* (Linné, 1758) as a Bio-sentinel to Monitor the Water Quality of North Algerian Rivers: Case of the El-Malah River 1217-1225

28. **Rajiv V. Shah,** Financial Incentives for Promotion of Electric Vehicles in India- An Analysis Using the Environmental Policy Framework 1227-1234
29. **Pradeep Khyalia, Jyoti Dangi, Sheetal Barapatre, Geeta Dhania, J.S.Laura and Meenakshi Nandal,** Comparative Analysis of Compost Quality Produced from Fungal Consortia and Rice Straw by Varying C/N Ratio and its Effect on Germination of *Vigna radiata* 1235-1242
30. **Zheng Ennan, Yinhao Zhu, Jianyu Hu and Tianyu Xu,** Effects of Humic Acid Organic Fertilizer on Soil Environment in Black Soil for Paddy Field Under Water Saving Irrigation 1243-1249
31. **Loay Alzriqat, Sana'a Odat and Ismaiel Abuamoud,** Main Characteristics of Trails on Yarmouk Forest Reserve, A Quantitative Approach to Trails Assessment 1251-1257
32. **K. Kalaivanan and J. Vellingiri,** Survival Study on Different Water Quality Prediction Methods Using Machine Learning 1259-1267
33. **G. Dhanya, T. S. Pranesha, Kamsali Nagaraja, D. M. Chate and G. Beig,** Comprehensive Modeling of Seasonal Variation of Surface Ozone Over Southern Tropical City, Bengaluru, India 1269-1277
34. **Kaizar Hossain, Mohd Talha Anees, Ahmad Farid Bin Abu Baker, Mohammad Muqtada Ali Khan, Amin E. Khalil, K. S. Ishola, Abdullah K., Mohd Nawawi M. N. and Mohd. Omar A. K.,** Application of Geo-electrical Methods for Estimating Water Infiltration in Soils 1279-1288
35. **E. Morales, S. Chávez, L. García, A. C. Caetano, J. Veneros, M. Á. Barrena and M. Oliva,** Variation in the Alcohol Components of *Coffea arabica* L. Wastewater Distillate Fermented Under Different Conditions 1289-1295
36. **Asad Ashraf and Izharul Haq Farooqi,** Investigation on the Treatment of Slaughterhouse Wastewater in a Sequential Batch Reactor 1297-1305
37. **Amarnath Bose and Devender Kumar Saini,** Biomass Fired Thermal Power Generation Technology- A Route to Meet Growing Energy Demand and Sustainable Development 1307-1315
38. **A. S. Nur Chairat, L. Abdullah, M. N. Maslan and H. Batih,** Applications of Marginal Abatement Cost Curve (MACC) for Reducing Greenhouse Gas Emissions: A Review of Methodologies 1317-1323
39. **H. P. Suseno and A. D. Warisaura,** Immobilization of Humic Acid on Bentonite and Its Application for Adsorption of $Cs^{137}$ and $Am^{241}$ 1325-1331
40. **P. Jayakumar, S. Padmanabhan, R. Lilly and K. Suthendran,** Bio-medical Waste Remediation by Environmental Safe Gelatin Coated Blood Sample Paper and its Effective Utilization 1333-1340
41. **B. K. Das, P. K. Das and P. Dash,** Evaluation of Phenotypic Responses of Selected Rice (*Oryza sativa* L.) Cultivars to Hexavalent Chromium Stress in Soil 1341-1347
42. **J. M Nyika and M. O Dinka,** A Mini-Review on the Use of Constructed Wetland Systems for Water Treatment in Developing Countries 1349-1356
43. **Xingjiang Song, Lin Zhou, Haibo Kang, Na Li, Wei Wang and Ping Jiang,** Study on Adsorption Properties and Mechanism of Graphene Oxide (GO) by Kaolin 1357-1365
44. **Simranjeet Singh, Anubha Kaushik and Bhoopesh Kumar Sharma**, A Study of Nutrient Removal Efficiency from Simulated Agriculture Run-off (SAR) Using Constructed Wetland Technology 1367-1374
45. **Arun Kalia and M. S. Dhanya,** Evaluation of Biodegradation Efficiency of Xylene Pretreated Polyethylene Wastes by Isolated *Lysinibacillus fusiformis* 1375-1380
46. **Priyanka Mary Sebastian and K. V. Bhaskara Rao,** Approaches in Bioremediation of Dioxins and Dioxin-Like Compounds – A Review on Current and Future Prospects 1381-1387
47. **Haifu Li, Chengjiu Guo, Fangli Su and Lifeng Li,** Effects of Cadmium on Superoxide Dismutase Activity in Reed Leaves 1389-1393
48. **Akshata Bandi H. and Nagraj S. Patil,** Estimation of Water Balance Components for the Watershed of Ghataprabha Sub-Basin 1395-1400
49. **Jie Li, Da Yao, Zhongyu Shi and Kaiyue Song,** Changes in Carbon, Nitrogen and Phosphorus Stoichiometry of Leaf-Litter-Soil in Differently Stands Under 'Plain Afforestation Program' in China 1401-1409
50. **N. A. Hemali and A. A. P. De Alwis,** Application of Material Flow Analysis to Municipal Solid Waste in Urban Areas in Developing Countries and Possible Solutions Under Circular Economic Framework 1411-1419
51. **Mahmuda Akter Khuky, Law Siong Hook, Lee Chin and Mohd Yusof Bin Saari,** The Impact of Textile and Clothing Export on Environmental Quality in Bangladesh: An ARDL Bound Test Approach 1421-1430
52. **Khan Md. Rabbani Rasha,** Salinity Prediction at the Bhairab River in the South-Western Part of Bangladesh Using Artificial Neural Network 1431-1438
53. **Vyacheslav M. Krayev, Alexey I. Tikhonov and Irina Kuzmina-Merlino,** Perspectives for the Use of Hydrogen Energy in European Countries 1439-1444
54. **N. P. A. Widjanarko and A. P. Siregar,** How Livestock and Industrial Energy Affect Indonesia's Surface Temperature 1445-1454
55. **Guanhua Zhang, Baoyang Sun, Feipeng Ren and Hao Li,** Response and Environmental Adaptation of Plant Community to Periodic Flooding in the Riparian Zone of Three Gorges Reservoir, China 1455-1462
56. **Ping Jiang, Xuhui Zhou, Jiandong Yang and Lin Zhou,** Strength Characteristics of Cement-Modified Iron Tailings and Their Adsorption on Graphene Oxide 1463-1470

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# Immobilization of Humic Acid on Bentonite and Its Application for Adsorption of $Cs^{137}$ and $Am^{241}$

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$Cs^{137}$
Humic acid
Immobilization

## ABSTRACT

In the present work, immobilization of humic acid on bentonite and its application as an adsorbent of $Cs^{137}$ (Cesium) and $Am^{241}$ (Americium) is performed. The parameters studied are rate constant, adsorption capacity, and adsorption energy. This research started with the immobilization of humic acid on a bentonite surface followed by a stability test for immobilization of humic acid at various acidities. Adsorption of $Cs^{137}$ and AM $^{241}$ ions was conducted by first examining the optimum pH giving minimum complexed ions by the soluble fraction of humic acid and maximum adsorbed ions on the adsorbent. While adsorption energy and capacity were determined by the Langmuir isotherm adsorption model. The result shows that the amount of immobilization of humic acid on bentonite is 39.75 % (w/w) at optimum conditions. Immobilization of humic acid was stable up to pH 12.0. For both ions, optimum adsorption occurred at pH 5. At this optimum condition, the adsorption energies of 21.481 $kJ.mol^{-1}$ and 22.276 $kJ.mol^{-1}$ for $Cs^{173}$ and $Am^{241}$ are obtained, respectively. The result also shows that adsorption capacity and energy for $Am^{241}$ were higher than that for $Cs^{137}$ which indicates the affinity of $Am^{241}$ for both adsorbents was higher than that of $Cs^{137}$.

## INTRODUCTION

Cesium ($Cs^{137}$) and Americium ($Am^{241}$) are the radioactive waste generated from plutonium-fueled power reactors or the result of nuclear weapon explosions. These radioactive can spread to the atmosphere as well as to the soil (Glikson 2017, Meszaros et al. 2016, Snow et al. 2019) and can be very harmful to human health. In the future, these radioactive emissions may increase since more nuclear power plant has been built around the world. Thus it is important to find out a sustainable method to encounter the $Am^{241}$ and $Cs^{137}$ pollution to the atmosphere and or to the soil. One method that can be utilized in controlling water pollution due to heavy metal and radioactive waste pollution is the adsorption method. The adsorption method was first introduced by Cihan et al. (2019). Mojiri et al. (2019) stated that adsorption is one method that can be used to treat waste. Adsorption is a method that is generally based on interactions between metals and functional groups that exist on the surface of the adsorbent that occurs through complex formation interactions and usually occur on solid surfaces that are rich in functional groups such as: -COOH, -OH, and NH (Yang et al. 2019, Kumar & Jiang 2016). The adsorbent that can be used for the purpose is humic acid which can be obtained from peat soil. According to Uda et al. (2017), Indonesia has peatlands of approximately 26.5 hectares, mostly in Kalimantan and Sumatra Island.

Humic acid is a very abundant and essential component in the natural environment and can bind to pollutants, both organic and inorganic pollutants (Ceci et al. 2019). Humic acid is a heterogeneous compound, consisting of many oxygen-containing groups (Boguta et al. 2019, Shi et al. 2018, Zhang et al. 2018). The various functional groups found in humic acid are the –COON, OH enolic, quinone, lactone, ether, and alcohol-OH groups (Zhang et al. 2018, Wu et al. 2019). The main functional groups found in humic acid are carboxylic groups, -OH phenolics and –OH alcoholic, in addition to other groups in minor quantities, such as carboxyl, phosphate, sulfate, amides, and sulfides which all can react with metal ions in solution. Spectroscopic studies show that- the –COOH group is a group that plays a vital role in the formation of complexes between metal ions with humic and fulvic acid. Moreover, the interaction also involves OH groups, -C = 0 and –NH.

Humic acid immobilization has also been reported by Cruz-Zavala et al. (2018), which is by immobilizing humic acid in chitin, by combining humic acid molecules with chitin through electrostatic interactions due to different loads, so we get a combined adsorbent of humic and chitin acids and produce a decrease in humic acid solubility to highly alkaline acidity. To enrich the surface of an inorganic solid through immobilization with functional groups of organic compounds, the solid should have active sides such as

siloxane (-Si-O-Si-), silanol (-SiOH), and aluminol groups (-AIOH) which can be chemically bonded with immobilized organic compounds, besides the solids must also have a large surface area. Fig. 1 shows the reaction mechanism of humic acid-binding in the adsorption process according to Tunega et al. (2019) and Smilek et al. (2017). The reaction mechanism of humic acid-binding in the adsorption process of humic compounds is supporting solids can occur through the reaction between hydrophilic functional groups of humic acid (carboxylic groups) with –OH groups on the surface of the supporting solid

The humic acid molecular structure consists of a set of active groups/micelles that form polymers naturally with the basic structure of the aromatic ring. Because of the very complex structure of humic acid, its structure cannot be stated with certainty and can only be expressed in a hypothetical structure (El-Sayed et al. 2019). Humic and fulvic acids contain groups consisting of oxygen atoms (such as -OH, -COOH, and -C = 0) with large concentrations per unit weight, so these compounds are hydrophilic (Ai et al. 2020). Some hypothetical structures of humic acid have been proposed by several researchers, such as Fuch, the German scientist, who proposed the structure of humic acid as shown in Fig. 2 (Klucakova & Veznikova 2017).

Another material that can be used as an adsorbent is bentonite. Based on the chemical nature of bentonite as ion exchange, bentonite can be used as an absorber in the treatment of liquid radioactive waste. Bentonite is a designation for commercial quality montmorillonite, with various compositions, but the formula is often stated as A12O3.4SiO2.H2O + XH2O. The montmorillonite clay is a 2: 1 clay type, a clay structure built by two tetrahedra sheets and one octahedral sheet. Two types of structures have been proposed for montmorilloniten namely structures according to (1) Hofmann & Endell, and (2) Edelman & Favajee, as shown in Fig. 3 (Tournassat et al. 2018).

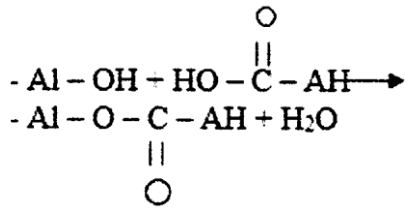



Fig. 1: Reaction mechanism of humic acid-binding in the adsorption process.

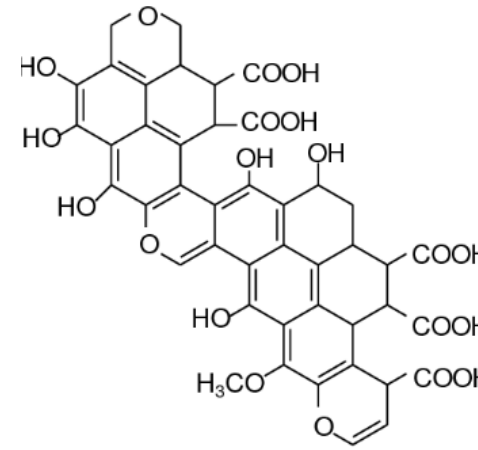
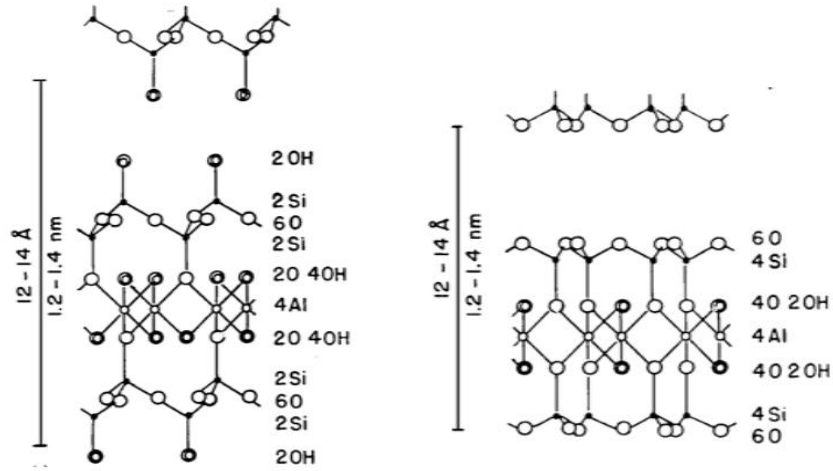



Fig. 2: Hypothetical structure of humic acid according to Fuch.




(a) (b)

Fig. 3: The model of montmorillonite structure according to a) Edelman & Favajee, b) Hoffman & Endell.

The use of humic acid and bentonite as adsorbents for $Cs^{137}$ and $Am^{241}$ filtration is limited. Thus, the present work aims to investigate the potential use of humic acid from peat land and bentonite as Adsorption material of $Cs^{137}$ and $Am^{241}$. The work investigates the immobilization of humic acid on bentonite and its characteristic in terms of rate constant, adsorption capacity, and adsorption.

## MATERIALS AND METHODS

**Immobilization of Humic Acid on Bentonite:** 60 mg of humic acid fills to three tubes in which the humic acid had been dissolved with 10 mL of 0.01 M NaOH and 100 mg of bentonite, with 0.001 M ion strength variation; 0.010 M and 0.100 M of NaNO3. Furthermore, a magnetic stirrer was performed to mix the solution for 1 h and allowed to stand overnight. It was then filtered and washed to produce neutral pH. The solid was dried at 66 °C in an oven until a constant weight was obtained. An ultraviolet spectrophotometer analyzed the filtrate at a wavelength of 466 nm.

**Identification and Clarification of Adsorbent Functional Groups:** To identify and clarify the changes in groups found on bentonite and humic acid, which are immobilized on bentonite with optimal ion strength, 1 mg of each sample was sampled to be dissolved and analyzed using an infrared spectrophotometer.

**Investigation of Adsorption of $Cs^{137}$ and $Am^{241}$ as the Function of Acidity:** Bentonite or AH-bentonite as much as 0.01 g and 10 mL of 100 ppm $CsI^{37}$ feed solution with pH of 1 were poured into vial continued with 60 min shaking and allowed to stand overnight. It would produce segregation of deposits and filtrates. The filtrates were analyzed by -θ spectrometer. The following steps were conducted in terms of pH 3, 5, 7, and 9. The adsorption test procedure of $Am^{241}$ is similar to that of $Cs^{137}$.

## RESULTS AND DISCUSSION

**Immobilization of Humic Acid on Bentonite:** Fig. 4 shows the immobilization reaction of humic acid on bentonite. The humic acid is a fraction of humic compounds that are insoluble in water solvents under acidic conditions (pH around 2), but it starts to dissolve if the pH of the solution

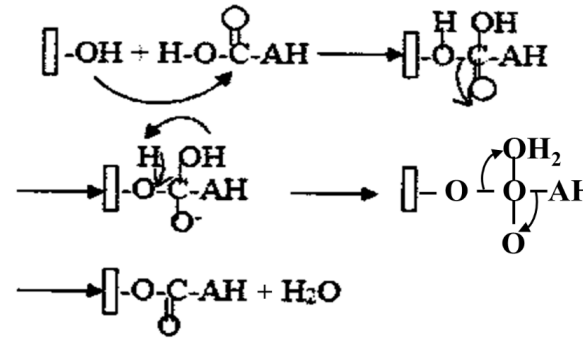



Fig. 4: Immobilization reaction of humic acid on bentonite.

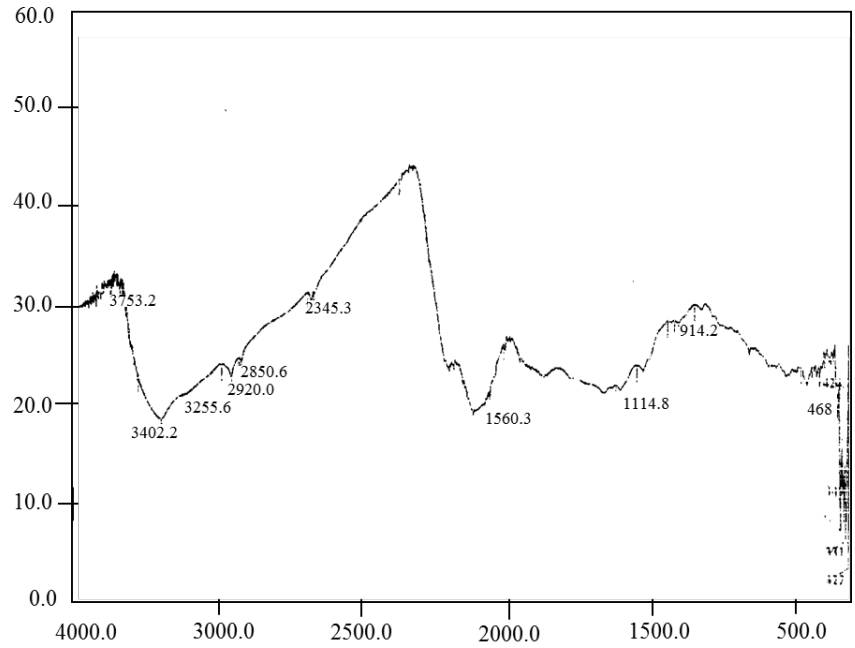



Fig. 5: Humic acid infrared spectra.

is above 2, and dissolves completely at pH above 6. At high pH, functional groups such as -COON and -OH phenolics from humic acid begin to be ionized, thus, weakening the intramolecular hydrogen bonds of humic acid, repelling inter-ionized groups and also as a result of hydration by water molecules. As a result of the dissolved fraction of humic acid, the interaction of humic acid with metal ions is not solely in the form of adsorption by insoluble fractions of humic acid, but also the interaction of complex formation between metal and dissolved humic acid fraction. Therefore, to study the adsorption of metals by humic acid at high pH, the treatment was applied to reduce the solubility of humic acid at high pH. To reduce the solubility of humic acid, the immobilization of humic acid on the surface of bentonite is carried out. The humic acid-binding reaction mechanism in the process of humic acid immobilization on bentonite occurs between functional groups of humic acid (carboxylic groups) with OH groups on bentonite through nucleophilic substitution reactions as follows. Therefore, bentonite solids that have silanol groups bind with humic acid through OH groups establishing nucleophilic substitution reactions to C atoms in the humic acid COOH groups.

**Spectra Characterization:** Fig. 5 presents infrared spectra of the humic acid. The spectra indicate widely absorbed absorption at 3402.2 $cm^{-1}$ wave number shows the OH carboxylic stretching vibration, absorption at 3255.6 $cm^{-1}$ wave number is OH phenol vibration, absorption at 2920.0 $cm^{-1}$ wave number identifies the existence of aliphatic CH stretching vibrations, the absorption at 2850.6 $cm^{-1}$ wavenumber is the methylene group. The OH bending vibration of COOH and the C-O stretching vibration of COOH were identified by the absorption of 1114.8 $cm^{-1}$ wave number. The aromatic group of humic acid was identified by the absorption band at l 560.3 $cm^{-1}$ wave number indicates the presence of - C = C aromatic. The OH stretching vibration of -COOH is confirmed by the appearance of absorption bands at 2345 3 $cm^{-1}$ wave number. The absorption bands of the humic acid infrared spectrum following the infrared spectroscopic data of humic acid, which absorbs 3300 $cm^{-1}$ wavenumbers as a result of the -OH stretching vibration. The existence of C-H stretching vibrations at 2900 $cm^{-1}$ wavenumbers indicates the presence of C-H aliphatic, and weak absorption bands at wavenumbers around 1500 $cm^{-1}$ indicate the existence of aromatic C = C vibrations.

Meanwhile, Fig. 6 shows a spectrum of bentonite. The graph shows a wide absorption band of 3431.1 $cm^{-1}$ wave number; a sharp band at 1637.5 $cm^{-1}$: a wide band at 1028.0 $cm^{-1}$; and a sharp band at 916.1 $cm^{-1}$. It is following the in-

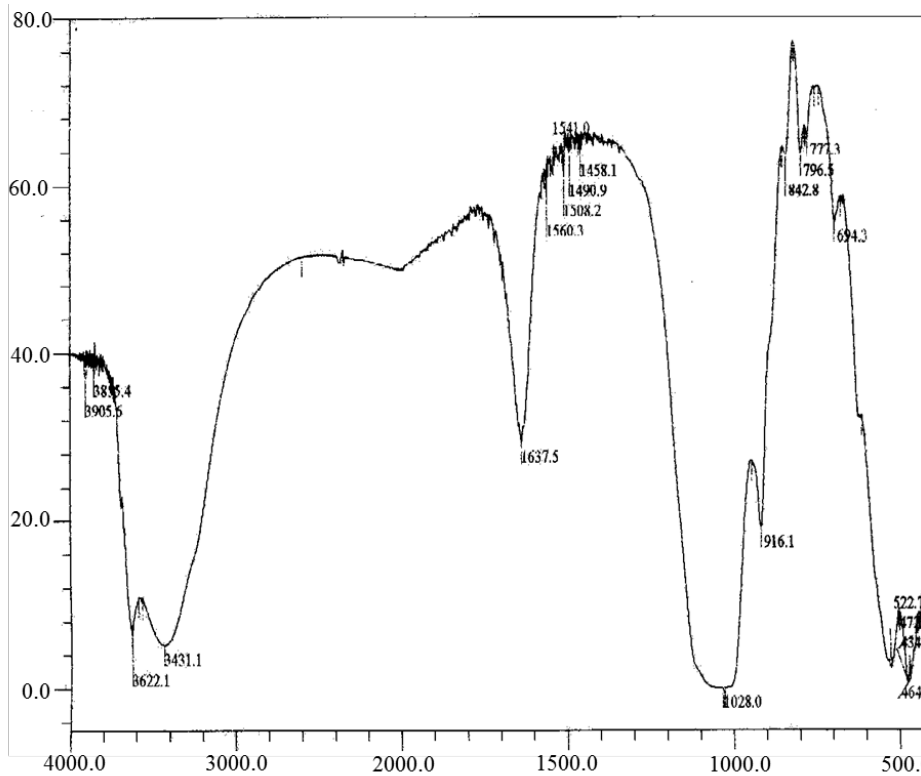



Fig. 6: Bentonite infrared spectra.

frared results of the reference montmorillonite which shows a wide band in the area of about 3420 $cm^{-1}$ for OH stretching vibrations and a wide band at 1050 $cm^{-1}$ for Si-O vibrations, bands for OH stretching vibrations, and bandwidth at 1050 $cm^{-1}$ for Si-O vibrations, sharp bands at wavenumbers 3622.1 $cm^{-1}$ for free stretching vibrations and sharp bands at 1637.5 $cm^{-1}$ are OH Stretching vibrations of water molecules and sharp bands at 910-920 $cm^{-1}$ wavenumber for AI-O vibrations.

Whereas, immobilized humic acid on bentonite spectra is shown in Fig. 7. The infrared spectra of the humic acid adsorbent immobilized on bentonite provide absorption peaks that are somewhat different from the infrared spectra of bentonite, namely the appearance of absorption at 2923.9 $cm^{-1}$ wavenumbers as CO aliphatic stretching vibrations and 2852.4 $cm^{-1}$ wavenumbers as methyl group vibrations. It clarifies the presence of humic acid in the absorbent of AH-bentonite. The absorption peak at 1035.7 $cm^{-1}$ wave number shows a decrease in the crystallinity of bentonite due to the immobilization occurrence of humic acid.

**Adsorption rate of $Cs^{117}$ and $Am^{241}$ on a single ion condition:** The adsorption equilibrium time needs to be determined to achieve maximum adsorption of adsorbate on the surface of the adsorbent. The shorter the reaction time, the higher the reaction rate. Increasing the interaction time will not increase the amount of metal adsorbed if the adsorption equilibrium has been reached. The value of the adsorption equilibrium constant (K) and the first-order reaction rate (kl) constant values obtained from calculations with the application of the kinetic model proposed by Chuanjiang et al. (2016) are presented in Table 1.

It can be seen from Table 1, that the value of the first-order reaction rate $k_1$ for $CS^{137}$ and $Am^{241}$ adsorption using HA-ben-

Table 1: The kinetics constants for $Cs^{137}$ and $Am^{241}$ adsorption (Chuanjiang et al. 2016).

| Species | Adsorbent | Kinetics Parameter | |
|---|---|---|---|
| | | $K_1$ [$min^{-1}$] | K [$mol.L^{-1}$] |
| $CS^{137}$ | AH-bentonite | 1.2 x $10^{-3}$ | 2881.8 |
| | Pure bentonite | 5.3 x $10^{-4}$ | 1045.8 |
| $Am^{241}$ | AH-bentonite | 1.6 x $10^{-3}$ | 5624.7 |
| | Pure bentonite | 9.8 x $10^{-4}$ | 2724.4 |

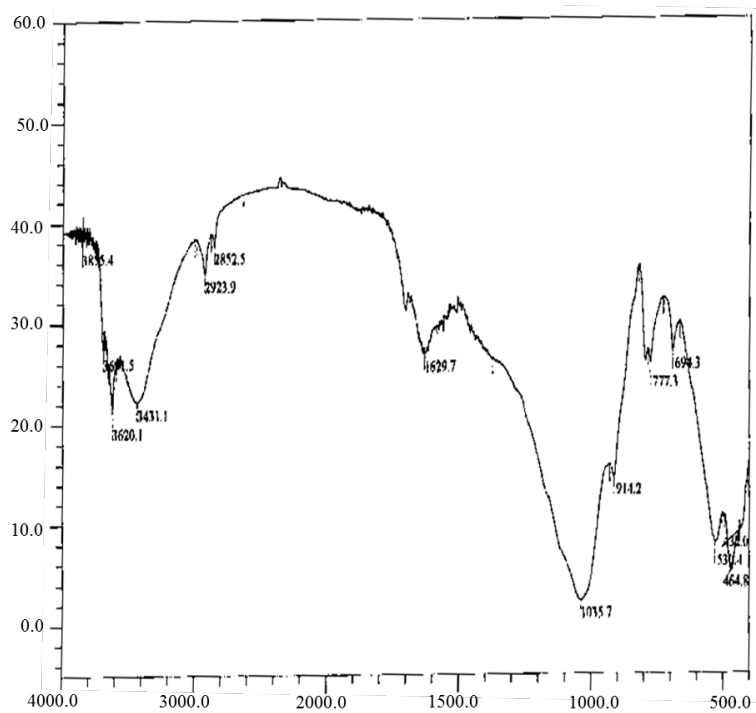



Fig 7: Infrared spectra of immobilized humic acid on bentonite.

tonite adsorbent is higher than pure bentonite adsorbents. It is due to the rich active site of HA-bentonite resulting from immobilized of humic acid on the surface, so HA-bentonite binds more efficiently to metal ions. The -COOH and -OH alcoholic and phenolic groups of humic acid that are immobilized on bentonite give new characteristics to the surface of the adsorbent. Those active groups have a significant role in binding metal ions, whereas, on bentonite, the binding of metal ions is dominantly determined by the presence of silanol (SiOH) groups on the surface of bentonite. The adsorption rate of $Am^{241}$ by the adsorbent of humic acid immobilized on bentonite increased higher compared with $Cs^{137}$. It describes that $Am^{241}$ has a greater affinity compared with $Cs^{137}$ on HA-bentonite. It also can be seen that the adsorption rate of the $Am^{241}$ metal ion is higher than the adsorption rate of $Cs^{137}$. in both adsorbents. The electronegativity (the size of atoms in a molecule to attract electrons to itself) values of Am and Cs are 1.3 A and 0.86 A, respectively (Cotton 2014). The electronegativity value of Am is higher than Cs, so the bond between Am and the adsorbent surface-active site has a relatively higher covalent bond. The increased covalent bonds between Am and adsorbents make Am less dissolved and more Am adsorbed on the surface of the adsorbent. Cs with smaller electronegativity form relatively more polar bonds with active sites on the surface of the adsorbent. The effect caused by polarization by cation is the solubility in polar solvents such as water. Therefore, the bond between Cs and immobilized humic acid on bentonite is more polar. It makes Cs easier to dissolve in water, which resulted in more Cs in the solution.

Regarding K values, the adsorption capability of HA-bentonite towards $Cs^{173}$ and $Am^{241}$ is higher compared to bentonite. It is caused by the more prosperous active sites of HA-Bentonite as the result of humic acid immobilization on the bentonite surface. Humic acid, which is rich in the -COOH and -OH groups, both phenolic and alcoholic, will give new characteristics to surfaces that are bound to bentonite. These groups have a high affinity for binding metal ions, so it creates more active sites in binding metals, whereas, in bentonite groups, the silanol groups are responsible for the active sites. The adsorption capability of HA-bentonite adsorbent is higher than $Am^{241}$ compared with $Cs^{137}$ indicated by a higher K value. It occurs because $Am^{241}$ has a greater affinity than $Cs^{137}$ in interacting with the adsorbent. According to Vogel (1961), ions that have a higher charge will have more excellent adsorption capability than the smaller charges. $Am^{3+}$ has a total charge of +3, so the ability to adsorb is higher than Cs+ ions whose total charge is only +1.

## CONCLUSION

The work on immobilization of Humic Acid on Bentonite and Its Application for Adsorption of $Cs^{137}$ and $Am^{241}$ has been performed. It can be concluded that the more strength the ion concentration, the larger the immobilization of humic acid on bentonite was, and vice versa. The amount of immobilization of humic acid on bentonite is 39.75 % (w/w) at optimum conditions. Immobilization of humic acid was stable up to pH 12.0. For both ions, optimum adsorption occurred at pH 5. At this optimum condition, the adsorption energies of 21.481 $kJ.mol^{-1}$ and 22.276 $kJ.mol^{-1}$ for $Cs^{173}$ and $Am^{241}$ are obtained, respectively. The result also shows that adsorption capacity and energy for $Am^{241}$ were higher than that for $Cs^{137}$ which indicates the affinity of $Am^{241}$ for both adsorbents was higher than that of $Cs^{137}$.

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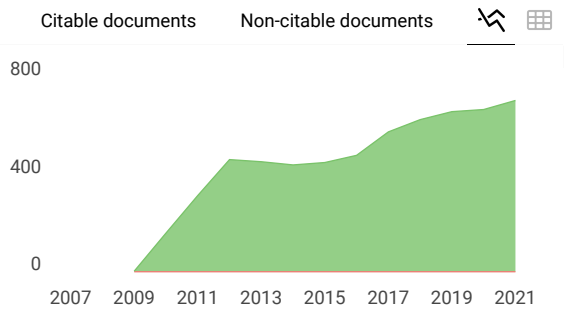


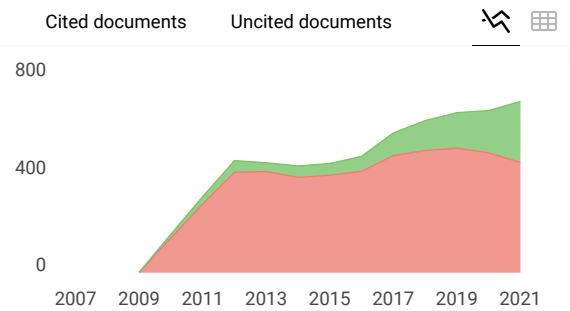

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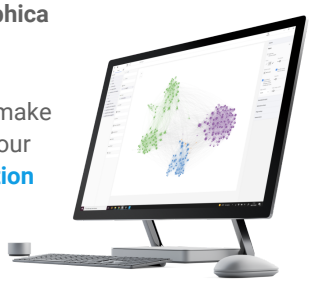

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