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Synthesis of Zinc Layered Hydroxide with 2-Napthoxyacetic Acid (BNOA) with Chloride as Counter Ions via Ion Exchange Method

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Abstract The development of environmentally friendly herbicides had raised a formulation of controlled release by using nanocarrier as one of the alternatives as it provide slow-release characteristics with precise concentration and time setting. The synthesis of host using zinc chloride ($ZnCl_2$) as a precursor to form zinc layered hydroxide-chloride (ZLC) and intercalated into herbicide which is 2-napthoxyacetic acid (BNOA) to produce nanocomposites via ion exchange called Zinc layered hydroxide-2-napthoxyacetic acid (ZLCB). The formation of nanocomposites was analysed by using Powder X-Ray Diffraction (PXRD). The result showed an expanding of basal spacing from 7.9Å to 27.3Å, indicated the intercalation of an anion into the interlayer of the host. Then, Fourier Transform Infrared Spectroscopy (FTIR) proved a new peak formed around 1607 cm^{-1}indicated the COO^- (carboxylate anions) confirmed the present of BNOA anions has successfully incorporated into the interlayers of ZLC. The surface area and pore volume were increased from 6.6 m^2g^{-1} to 27.5 m^2g^{-1} and 0.02 $cm^3g^{-1}$to 0.04 cm^3g^{-1} respectively, showing that the BNOA had successfully inserted into the interlayer of ZLC. Moreover, the loading percentage of BNOA estimated by CHNS analyser was 83.9%. The controlled release property shown that the release of BNOA in the various aqueous solution is in order of Na_2PO_3> Na_2SO_4> NaCl and fitted into pseudo-second order of kinetic models.

Keywords: *zinc layered hydroxide, 2-naphtoxyacetic acid, release kinetic, ion exchange, herbicides*

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1. Introduction

The major challenges in the agriculture sector are optimized production of agriculture products from a limited area without any adverse effect on environment and sustainability. However, the unknowledgeable and over usage of fertilizers and pesticides has resultant residues and toxins in the soil, ground/surface waters, and air, confronting the cost of increased fertilization, irrigation, and energy to retain productivity, health costs, and ultimate desertion of the health of agriculture lands all over the world. Nonetheless, nanotechnology technology, has provided an innovative and resourceful platform to improve the competence of agricultural materials while offering solutions to agricultural and environmental problems. The nano fertilizers substitute conventional fertilizers, accumulation of nutrients in the soil and thus eutrophication and pollution of drinking water may be diminished and allow the release of nutrients in a controlled manner in required amounts. Hence, it is essential to develop a method for the sustainable and efficient release of fertilizer. Layered Double Hydroxide (LDH) is a better option.

A significant number of possible arrangements and metal as well as anion intercalation make LDH a versatile material with unique characteristics such as high chemical stability, basicity, pH-dependent solubility, and bio and environmental compatibility (Figure 1).

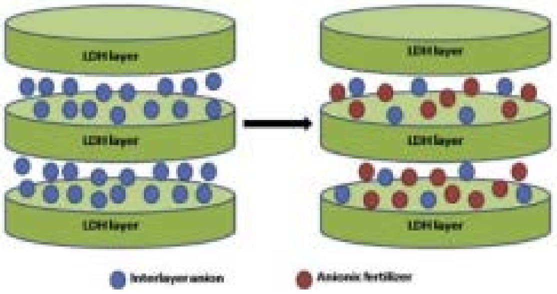

Figure 1. Anionic exchange in LDH

Layered double hydroxides (LDHs) are derived from the brucite-like layer with general formula of $[M^{II}_{1-x}\ M^{III}_{x}(OH)_{2}]^{x+}(A^{m-})_{x/m}]^{\cdot}nH_2O$, where M^{II} and M^{III} are the divalent and trivalent metal ions, A^{m} are organic and inorganic anions, n is the number of interlayer water, and $x = M^{III}/M^{II} + M^{III}$ is the layer charge density. In contrast, the zinc layered hydroxide (ZLHs) are modified from the brucite layer of LDHs with the general $M^{II}(OH)_{2-x}\ A_{x}{}^{\cdot}nH_2O$ where M^{II} is the only divalent metal ions, A_x is organic or inorganic anions and n is the number hydroxyl group of interlayer [1].

These LDHs are based on the stacking of layer consisting of anions that octahedrally coordinated to six OH hydroxyl group is which part of divalent cations are substituted by the trivalent ions. The replacement leads to the formation of positively charged layers, whose net charge is compensated, to preserve the neutrality the presence of exchangeable anions (nitrates, chlorides, and carbonates) in between the layers conjointly with water molecules [2].

Previous researchers have carried out intercalation of 2-naphtoxyacetic acid (BNOA) with LDHs of ZnAl or MgAl showing that BNOA is widely applied in agricultural planting and regulating plant growth. BNOA is being used as a regulator of cell division, cell elongation, and cell differentiation of plant development through the absorption of leaves and roots [3]. However, the intercalation of BNOA onto basal spacing zinc layered chloride (ZLC) has not been published elsewhere.

Interestingly, the zinc layered chloride (ZLC) that uses zinc chloride as precursor and subsequently chlorides will be the exchangeable anion that helped to neutralize the layer with the water molecules. The tetrahedral zinc atom with three vertices is occupied by hydroxyl groups. The fourth position is directly bonded to the chloride atom. These zinc cations layers leave an open octahedral site and create cationic centers that are tetrahedrally stacked up and down the octahedral sheet. The different in spatial orientation of LDHs and ZLC makes a huge different in the value of basal spacing after the intercalation of the guest anions (herbicides) in the interlayer. The presence of tetrahedral zinc between the layer will help to expand the basal spacing that makes ZLC more efficient as a release carrier compared to LDHs [4].

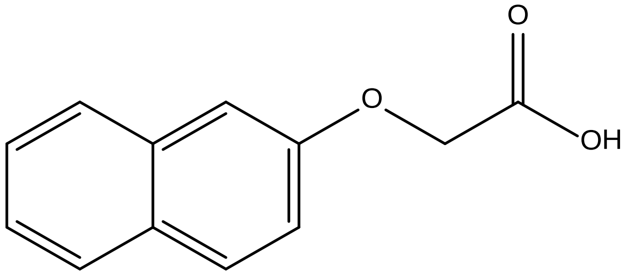

Figure 2. Chemical Structure of 2-napthoxyacetic acid (BNOA)

The synthesis to encapsulate the BNOA into the interlayer of ZLC is via ions exchange. The mechanism of ion-exchange reaction by ZLC is when a proton of acid and ethanol, could assist the release of Cl as HCl from the interlayer space of ZLC leaves the layer in the form of positively charge and the carboxylate ions (COO^{-}) of BNOA will be attached to the layer to remain the neutral brucite layer. The BNOA will be inserted into the interlayer of ZLC to form nanocomposite which will be used as herbicides to provide the nutrient to the plant [5]. The chemical structure of BNOA is shown in Figure 2.

The zinc contribute by the ZLC also plays very important role in plant metabolism which the enzyme activates by zinc involved in carbohydrate metabolism, maintenance of the integrity of cellular membranes, protein synthesis, regulation of auxin synthesis and pollen formation [6]. Although the BNOA and ZLC give a lot of benefits towards the development and growth of the plant, the effectiveness of the herbicides mostly depends on the specific concentration and time setting.

Thus, the control release formulation (CRF) was introduced to overcome the overused of herbicides concentration in the environment allowing the slow release of the herbicides by implement the kinetic study using the adsorption model of zeroth order, first order and pseudo second order [7]. The process mainly involves the adsorption as it is time dependent with considerable ecosystem impact influencing transport of herbicides in the soil environment during short term and consequently affecting the leaching potential [8].

The objectives of the study were to examine the characterization and physicochemical of the synthesized intercalated ZLC with BNOA (ZLCB) such as surface area, pore size distribution and organic-inorganic composition as well as the controlled release properties which were explored in this research [9].

2. Experimental

2.1. Synthesis of ZLC with 2-naphtoxyacetic Acid (BNOA) via Ion Exchange Method

All chemicals used in the synthesis were obtained from various chemical suppliers and used without further purification. The synthesis of $ZnCl_2$ (1.38M) solution was by slow addition of NaOH (0.75M) and pH was adjusted to pH 7 ± 0.2 under magnetic stirring with presence of N_2 gas. The product obtained was centrifuged, washed several times with deionized water, and dried in an oven at 70°C for 48 hours [10].

Then, ZLC (0.8g) was reacted with BNOA (20 ml) solution (75% ethanol) at concentrations ranging from 0.15M to 0.6M was adjusted to pH 7.5 ± 0.2 by gradually adding NaOH (0.75M) under magnetic stirring with presence of N_2 gas. The solution was aged for 18h in an oil bath shaker at 70°C. The resulting product was centrifuged, washed thoroughly with deionized water, and dried in an oven for 48h [11].

2.2. Controlled Release of BNOA

The release of BNOA was studied by using ZLCB (0.1M) into 0.005M aqueous solution of Na_3PO_4, Na_2SO_4 and NaCl. The amount of BNOA released into the solution was measured in a pre-set time at λ_{max} = 253.1 nm using a Perkin Elmer UV–Vis Spectrophotometer Lambda 365. Data was collected and fitted to zeroth order, first order and pseudo-second order kinetics model.

3. Characterization

The pattern of the samples was recorded using the Powder X-ray diffraction (PXRD) on a X'Pert MPD PRO diffractometer (PAnalytical International Corporation, Almelo, Netherland using Cu Kα radiation (λ = 1.5406 Å) at 45 kV and 30 mA. The data were gathered at a scanning rate of 2°/s over a range of 2θ = 2°- 60°. The KBr pellet method was used to obtain the Fourier transform infrared (FTIR) absorption spectra of each sample within the wavenumber range of 450-4000 cm^{-1}. The thermal analysis of the samples was determined by thermogravimetric analysis (TGA) on a Netzsch Thermogravimetric Analyzer (USA) with a heating rate of 10°C min^{-1} under nitrogen atmosphere. The loading percentage of ZLCB elements was calculated using a CHNS Analyzer 2400 PerkinElmer Series II (USA) with dynamic flash combustion technique. Thus, the sample surface morphology was characterized using the Field emission scanning electron microscopy (FESEM) (Carl Zeiss Supra 40, USA) at 15000 magnifications. Micromeritics ASAP 2060 (USA) was used to determine the surface area and porosity of the samples. The surface characterization of the materials was carried out by nitrogen gas adsorption–desorption at 77 K. The samples were degassed in an evacuated-heated chamber at 110°C overnight prior to the measurement.

4. Results and Discussion

4.1. Powder X-ray Diffraction Analysis

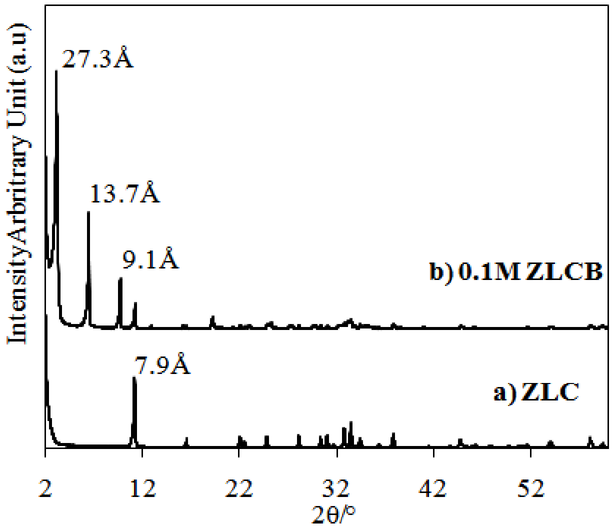

Figure 3. PXRD pattern for ZLC (a) and ZLCB (b)

Figure 3 indicates the PXRD patterns of ZLC host and the intercalated compound, labelled as ZLCB were synthesized using ion exchange method. The PXRD pattern shown the basal spacing for ZLH with chloride as interlamellar anions was 7.9 Å which in agreement with previously study [12]. The resulting nanocomposite given a well ordered nanolayered structure with expanded basal spacing from 7.9Å in ZLC to 27.3Å for 0.1M ZLCB [17.3Å = 27.3Å – (4.8+2.6+2.6)] (Figure 4). Considering that the layer thicknesses of ZLC are 4.8Å for the octahedral of brucite layer structure and 2.6Å for each zinc tetrahedron respectively (Figure 4) [13].

The basal spacing of BNOA intercalated into the ZLC in this study was 27.3Å, which significantly bigger than the basal spacings of BNOA intercalated with ZnAl (19.5Å) and MgAl (20.3Å) in prior studies [3,14]. This was due to spatial orientation of BNOA inserted into the interlayer of ZLC that includes the basal spacing of octahedral of brucite layer structure and the two unit of zinc tetrahedral layer which gradually expand the basal spacing of ZLC.

These have demonstrated that ZLC offers greater basal spacing compared to ZnAl-LDH and MgAl-LDH, which allowed the release of herbicides to the soil to be much easier. The patterns of PXRD also exhibit some features of layered materials such as narrow, symmetric with high intensity peaks at low 2θ values [15]. Thus, nanocomposite prepared using 0.1M BNOA was subsequently used for further characterization. The illustration of Figure 3 is drawn by using Chem 3D software.

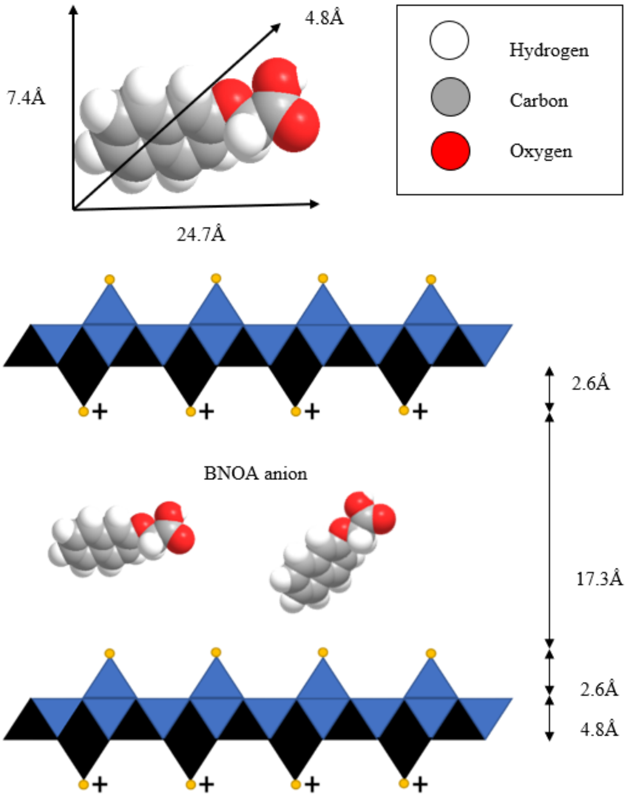

Figure 4. Proposed spatial orientation of BNOA in the interlayer of ZLC

4.2. Fourier Transform Infrared Analysis

The FTIR spectra for ZLC, pure BNOA and ZLCB are shown as Figure 5. A characteristic peak of ZLC (Figure 5(a)) at around 3455 cm^{-1} which attribute OH stretching due to presence of hydroxyl group of the host. The band 1632 cm^{-1} was wave number representing the bending vibration of the interlayer OH water molecule. The appearance of strong absorption at 732 cm^{-1} is attributed to the presence of Cl_2 in ZLC [16].

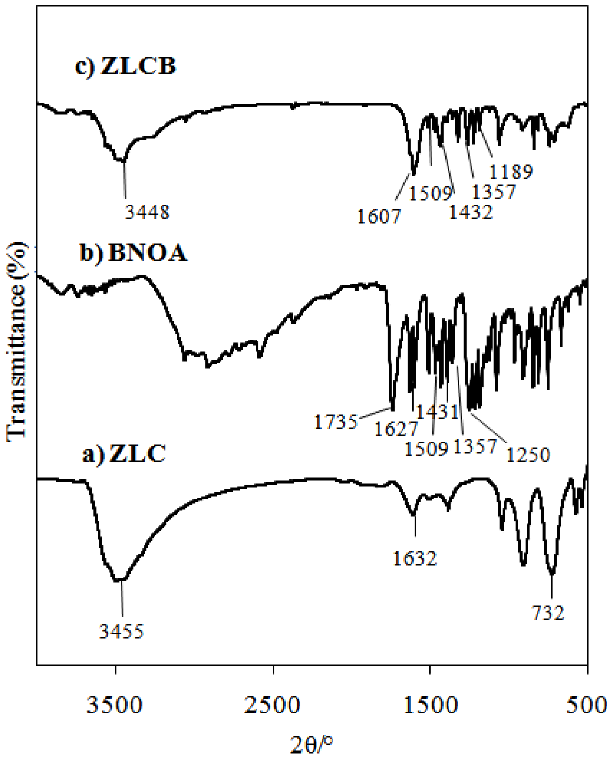

Figure 5. FTIR spectra of ZLC (a), pure BNOA (b) and 0.1M ZLCB (c)

The BNOA spectra shown (Figure 5(b)) strong band at 1735 cm^{-1} indicates the carbonyl, C=O stretching vibration. The band at 1627 cm^{-1} due to the vibration stretching of C=C aromatic rings. The other strong band at 1250 cm^{-1} shows the stretching of C-O-C and band at 1431 cm^{-1} indicates the presence of CH_2 scissoring vibration [3,17].

As expected, the FTIR spectrum of ZLCB (Figure 5(c)) nanocomposites resembles a mixture of both the spectra of BNOA and ZLC, indicating that both functional groups of BNOA and ZLC are simultaneously present in ZLCB and confirmed the intercalation of BNOA in the interlamellar of ZLCB. The absorption band at 732 cm^{-1} which was due to chloride ions was absence indicates that the anion completely replaced by BNOA anion. This shows that BNOA has higher affinity than chloride ion towards the inorganic interlamellar, thus occupies the interlamellar region between the inorganic layers. The most important feature in the FTIR spectra was the disappearance of band 1735 cm^{-1} (due to carboxylic group) and the present of new band at around 1607 cm^{-1} which shifted from 1627cm^{-1} attributed to COO^- (carboxylate anions) confirmed that the present of BNOA in the anionic form in the interlayer of zinc layered [14].

4.3. Elemental Analysis

Table 1. Elemental and Surface properties of ZLC and intercalated compound, ZLCB

Material	d(Å)	%C	%N	%H	% Loading	BET surface area (m^2g^{-1})	BJH total pore volume (cm^3g^{-1})
ZLC	7.9	0.1	-	1.8	-	6.6	0.02
ZLCB	27.3	59.7	-	4.1	83.9	27.5	0.04

The intercalation of BNOA anions into the ZLC inorganic interlayer was also confirmed by direct evidence of elemental analysis (CHNS). Table 1 shows that the C percentage in ZLCB was 59.7% due to the carbon contain for BNOA. Then, the percentage loading of BNOA anions into the nanocomposite is 83.9% for ZLNB which in agreement of estimated from the carbon content in the nanocomposite (Appendix 1).

4.4. Surface Morphology

The surface morphology of layered ZLC and ZLCB nanocomposites at a magnification of 15k are displayed as in Figure 5. The ZLC (Figure 6(a)) possessed a flake-like structure typically for the morphology of ZLH was in the agreement from previous study [18]. The nanocomposites ZLCB (Figure 6(b)), shows an agglomeration, porous and granular structure compared to ZLC with non-porous surface [19]. After the intercalation, the resulting layered ZLH materials take on new properties that are a function of the intercalant and the way it associates with the host. Therefore, both physical and chemical properties including the surface morphology may be affected by intercalant.

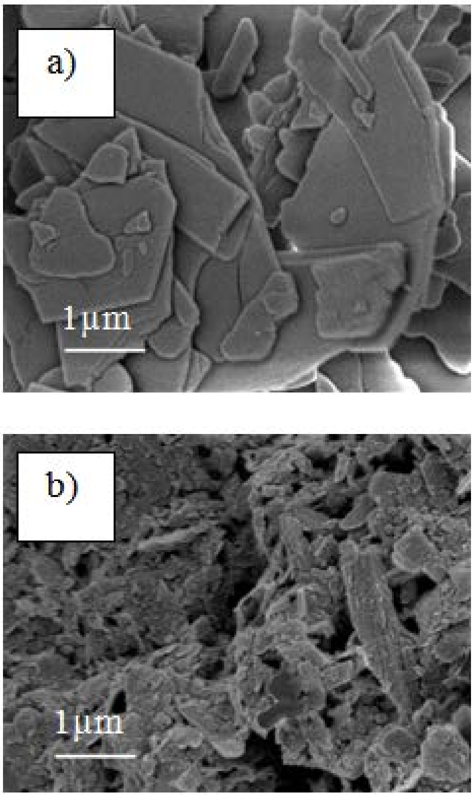

Figure 6. Surface morphology of ZLC (a) and ZLCB (b) with 15k magnification

4.5. Surface Properties

The nitrogen adsorption-desorption isotherm and pore size distributions for ZLC and its nanocomposites, 0.1M ZLCB were shown in Figure 7. According to the IUPAC classification, the type of isotherm can be classified as Type IV (mesopore), and the desorption hysteresis is a type H3 loop that aggregates flake-like particles that give rise to slit-shaped pores that complement the surface morphology shown in the Figure 6. The absence of any limiting adsorption at high $P/P_{\circ}$ suggesting there is no presence of macropores in the nanocomposites. The adsorption isotherm suggests that a monolayer adsorption was initiated on the pore surface at low partial pressures followed by multilayer development up to high partial pressures. The slow adsorbate uptake of nitrogen at pore volumes 0.0–0.8 cm^3/g with a maximum adsorbate uptake of 10 cm^3/g. There was no limiting desorption of nitrogen gas observed at high partial pressures, the hysteresis loop, indicative of capillary condensation in the mesopores, occurred on the desorption isotherm down to partial pressure of around 0.75 (P/P∘) for ZLCB [20].

Table 1 summarizes the surface area and pore volume of ZLC and ZLCB as determined by the Brunauer, Emmett, and Teller (BET) method and the Barrett, Joyner, and Halenda (BJH) method. The BET surface area of ZLC (Figure 7(a)) was 6.6 m^{2}g^{-1} and while for ZLCB (Figure 7(b)) the surface area increased to 27.5 m^{2}g^{-1} indicated the enlargement of pore happened correlated to the basal spacing expansion in the PXRD pattern analysis in Figure 3. The BJH pore volume slightly increased from 0.02 cm^3 g^{-1} in ZLC to 0.03 cm^3 g^{-1} in ZLCB (Figure 7(c)) due to the intercalation of BNOA into the LDH interlayer, replacing the counter anion, chlorides. The intercalation process has altered the pore texture of the host, ZLC, as shown by the difference in peak value and pore diameter between the pore size distribution of LDH and the intercalated nanocomposite.

4.6. Thermal Analysis

Figure 8 shows the thermograms for BNOA, ZLC and ZLCB respectively. One step degradation behavior was observed in the thermal analysis of BNOA (Figure 8(a)). The decomposition temperature for BNOA was 243°C with the total weight loss of 76.3% due to the evaporation of adsorbed moisture and decarboxylation of pure BNOA [21].

The TGA-DTG profiles of ZLC (Figure 8(b)) reveal two weight losses occurring at 158.8°C and 439.2°C that amounted 14.4% and 13.0% respectively. The first weight loss was due to the removal of surface-physiosorbed water molecules, and the second weight loss is attributed to the decomposition of gas to formed zinc oxide from the counter ions, chlorides [22].

Meanwhile, for ZLCB (Figure 8(c)) there are three main thermal decomposition events that happened. Firstly, at temperature 167.0°C with weight loss 3.63% due to the removal of surface physiosorbed water molecules. Then, the largest stage of weight loss occurring at 286.1°C with 63.3% due to decomposition of the intercalated organic moiety, the BNOA anion between the inorganic interlayer of ZLC [23]. At temperature 352.2°C with weight loss of 8.87% because of collapsing of the layered structure of ZLC [24].

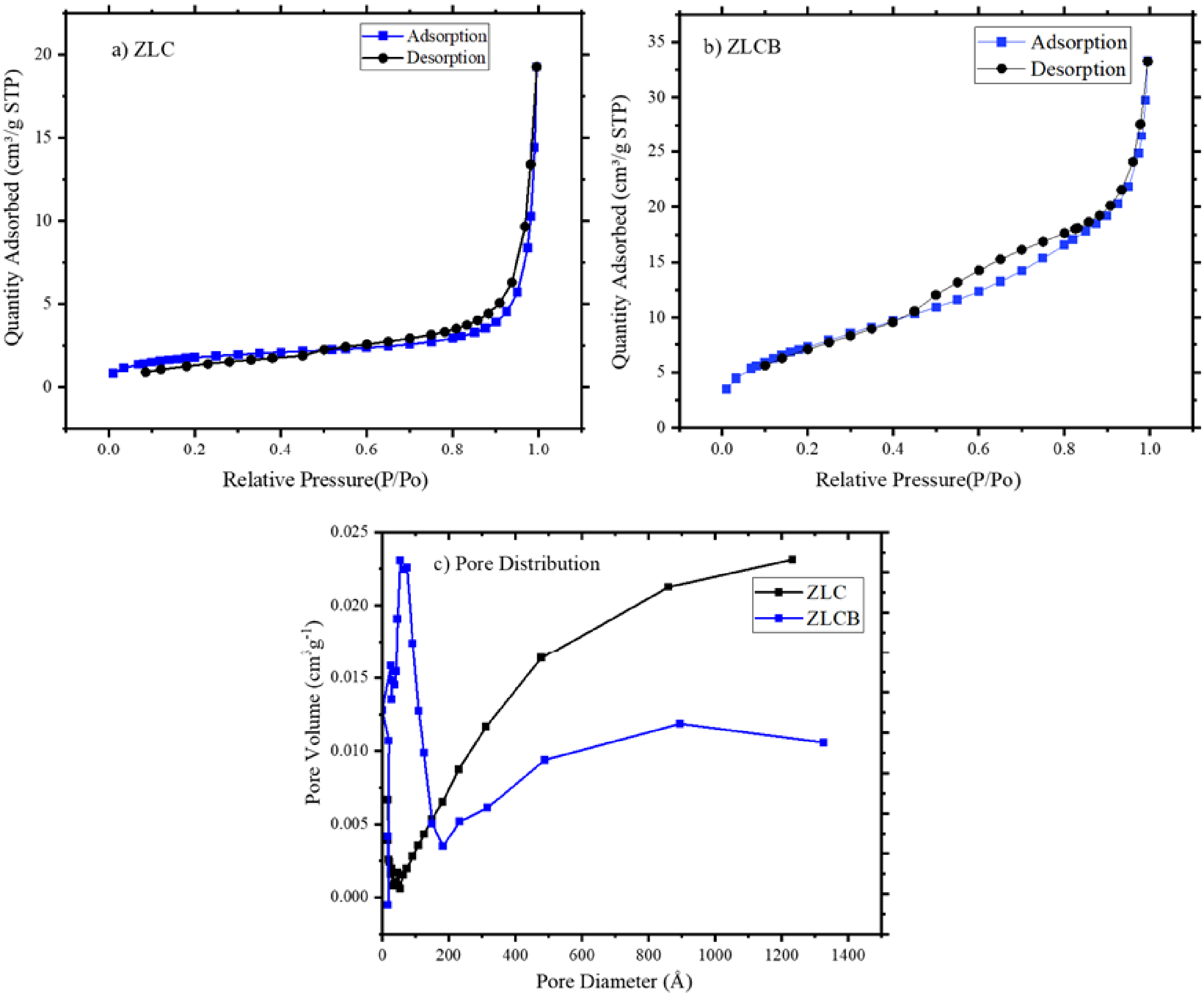

Figure 7. Nitrogen adsorption-desorption isotherm of ZLC (a) ZLCB (b) and pore size distribution (c)

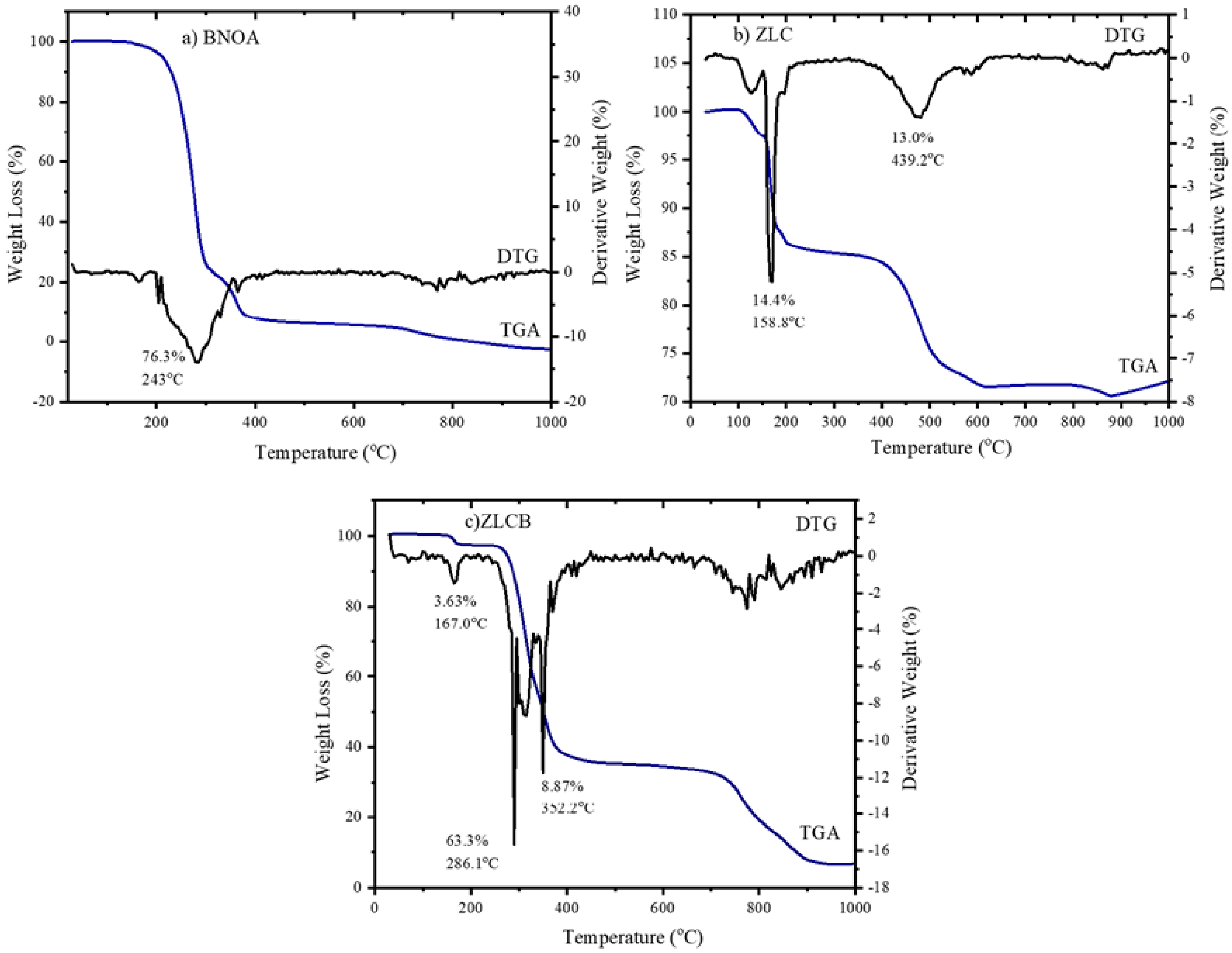

Figure 8. TGA-DTG thermograms of pure BNOA (a) ZLC (b) and ZLCB (c)

4.7. Release of BNOA into Aqueous Solution

Figure 9 shows the BNOA release profile into 0.005M sodium phosphate (Na_3PO_4), sodium sulfate (Na_2SO_4), and sodium chloride (NaCl) solutions from the ZLCB interlamellar. The release rate was found to be faster in the first 250 min, then slower until the equilibrium reached at around 1000 min. The amount of BNOA released from ZLCB into aqueous solution containing Na_3PO_4 was found to be the highest followed by Na_2SO_4 and NaCl. It was observed that at the end of rapid release rate, the amount of BNOA released from Na_3PO_4 was 86% followed by 79% of Na_2SO_4 and the least amount of BNOA released from ZLCB is 57% of NaCl solution. Therefore, the order of BNOA released from ZLCB at the end 1440 min can be summarized as $Na_3PO_4 > Na_2SO_4 > NaCl$ [14].

In general, several factor such as the affinity of the anion towards the ZLCB inorganic interlamellar is important and will contribute to the amount of BNOA released into the aqueous solution. The selectivity for the ZLCB increase with the increasing electric charge of the anion and decreasing of anion size which trivalent anion such as PO_4^{3-} and divalent anions SO_4^{2-} have higher selectivity compared to monovalent Cl anions [25].

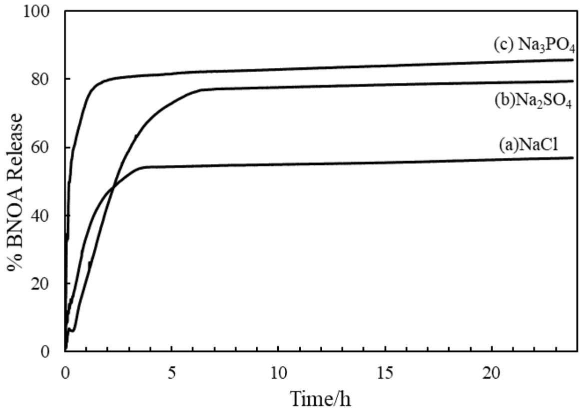

Figure 9. Release profile of BNOA from ZLCB into 0.005M (a) NaCl solution, (b) Na_2SO_4 solution and (c) Na_3PO_4 solution

4.7.1. Kinetic Release

Table 2. Fitting the release data of BNOA anion from ZLCB into various media using zeroth, first and pseudo second order kinetic models for release time 0-1400 min

Aqueous solution 0.005M	Release Time, min	Zeroth Order, r^2	First Order, r^2	Pseudo-Second Order, r^2
NaCl	1440	0.2726	0.8858	0.9998
Na2SO4	1440	0.3124	0.8259	0.0997
Na3PO4	1440	0.4185	0.8128	0.9907

The data from the release study of the quantitative analysis shown in Figure 10 were fitted to zeroth order, first order, and pseudo-second order functions. It was proposed that ZLCB dissolution may be used to control the release of anions. In the previous research, where the release kinetics of intercalated clay minerals was studied, a single straight line was used to fit the experimental data. The following zeroth-order (Equation (1)), first-order (Equation (2)) and pseudo-second order (Equation (3)), equations are given below in which C_{eq} and C_t is the percentage release of the herbicides at equilibrium and time, t, respectively, and c is a constant [25].

$$C_t = kt + c \tag{1}$$

$$-log\left(1 - C_t\right) = kt + c \tag{2}$$

$$t / C_t = 1 / k_2 {C^2}_{eq} + \left(1 / q_e\right).\, t \tag{3}$$

As shown in Figure 10 and in Table 2, the release of BNOA into 0.005 M NaCl, Na_2SO_4 and Na_3PO_4 of aqueous solutions were fitted using zeroth order, first order and pseudo-second order kinetic models for release time 0-1440 min. Poor fitting for data on the zeroth order kinetic models with 0.2726 to 0.4185 regression values. Then, for the first order the regression values for were fitted around 0.8000 for each salt solution. However, the release profiles follow nicely to the pseudo-second order kinetics for the long-time range of the ion-exchange process which is 1440 min with regression values around 1.000.

The dissolution of the nanocomposite and the ion-exchange between the intercalated anions in the interlamellar with chloride, sulphate, and phosphate anions in the aqueous solution were both involved for the release of BNOA from the inorganic ZLC interlamellar [8]. This entire process was controlled by a pseudo-second order kinetic model, as shown by the regression, r^2 values. The nanocomposite, ZLCB fitted with the pseudo second order as the process of release herbicides depend on the concentration of the solution and time as low concentration of solution will take lesser time until reaches the equilibrium. Apart from that, the ions exchange of chloride, sulphate and phosphate from the solution signifies that the ZLCB has a good ions exchange capacity. Based on the release profile of BNOA (Figure 9) the anions present in the soil water will force other cations off and take the place to exchange via the interlayer of ZLCB. Hence, able to take up the nutrient provided by the soil to be used by the plant through cation exchange capacity (CEC) by way of symplastic path towards the root xylem [26].

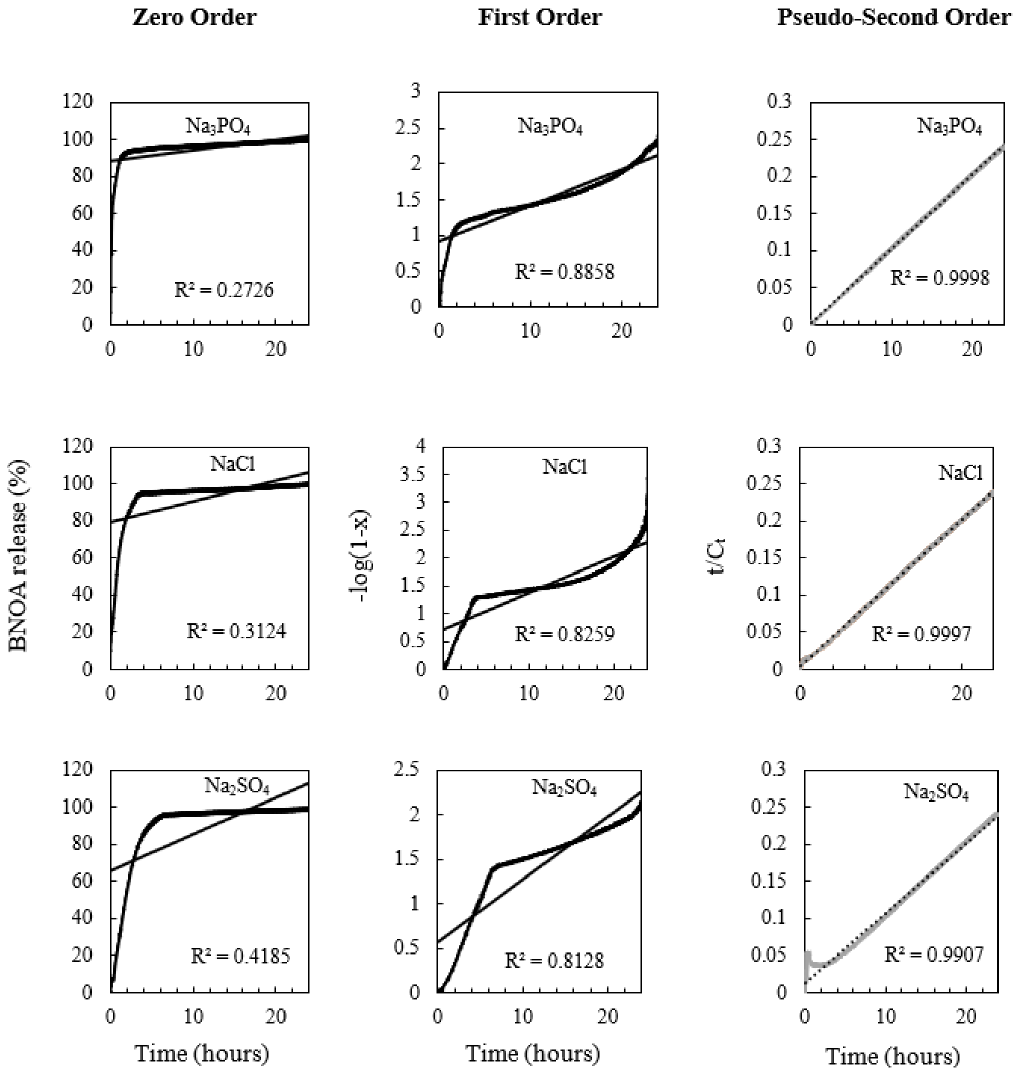

Figure 10. Fitting the release data of BNOA anion from ZLCB into various aqueous solution using zeroth order, first order and pseudo-second order kinetic models for release time 0-1440 min

5. Summary

The results showed that all the characterization was compensated with one to another. Firstly, the FTIR results has proved that the BNOA has successfully intercalated in the ZLC basal spacing. The chloride ions from the ZLC were absent and the functional group of BNOA such as the carboxylate ions was indicated into the ZLCB nanocomposite. Then, the expansion of basal spacing has been supported from the PXRD analysis. The ZLC basal spacing was increased with the BNOA molecules in the interlayer of ZLCB (Figure 3). The interlayer spacing of ZLC increased from 7.9Å to 27.3Å in the ZLCB due to the inclusion of BNOA into the ZLC interlayer space with a loading percentage of 83.9% (w/w). The FTIR and PXRD results have been supported with then, the elemental analysis (Table 1) proved that the presence of BNOA was successfully incorporated into the interlayer of ZLCB as loading percentage of estimated carbon was calculated in Appendix 1. In addition, the nitrogen adsorption– desorption isotherms of ZLC and ZLCB exhibited Type IV pattern with a Type H3 hysteresis loop, which is characteristic for mesoporous materials. The type IV isotherm supported the systematic ZLCB interlayer spacing. In addition, the surface properties study (Figure 8) revealed an increase in surface area from ZLC to ZLCB, which was further supported by the surface morphology from SEM analysis (Figure 7). The micrograph showed that the changed from a flake-like particle to an aggregated slit shaped pore of the ZLCB. The slow-release analysis revealed that the release pattern of anions from the aqueous solution (Figure 9) caused by the ion exchange phenomena govern the release of BNOA into the NaCl, Na_2SO_4 and Na_3PO_4 solution. The kinetic of slow release has been proved to be a pseudo second order model. The release of ZLCB follows pseudo-second order models with regression values from 0.9907 to 0.9998. The model indicated with the slow release was due to the increased basal spacing, thus the exchangeability of the BNOA with the anions (PO_4^{3-}, SO_4^{2-} and Cl^-) become more ease while followed the selectivity of electric charge of the anions.

6. Conclusion

A herbicides compound, BNOA was successfully intercalated into the layer of ZLC for the formation of a new organic-inorganic hybrid nanocomposite via ion exchange method, ZLCB which shows a potential to be used as a controlled-released formulation in agrochemicals. The study has successfully synthesized ZLCB nanocomposite material, with each characterization supported one to another. This study suggests the possibility of ZLC to be used as a carrier host for BNOA for the generation of environmentally friendly agrochemicals.

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Appendix

Weight of C in the sample = Percentage of C X Sample weight (mg)

$$=\left(\frac{59.7330}{100}\text{X}2.534\text{mg}\right)$$

$$=1.5136\text{mg}$$

Mole of C in the sample = Weight of C in the sample / Atomic weight of C

$$=\frac{1.5136\text{X}10^{-3}\text{g}}{12\text{g}/\text{mol}}$$

$$=1.2613\text{X}10^{-4}\text{mol}$$

12 moles of C + 10 moles of H + 3 moles of O = 1 mole of $C_{12}H_{10}O_3$

12 mol of C = 1 mol of BNOA

$$\frac{1.2613\text{X}10^{-4}\text{mol}}{12\text{mol}}\ \text{of C} = 1\ \text{mol BNOA X}\ \frac{1.2613\text{X}10^{-4}\text{mol of C}}{12\text{mol of C}}$$

$$= 1.0511\text{X}10^{-5}\text{moles of BNOA}$$

Weight of intercalated BNOA = moles BNOA in sample X molecular weight BNOA

$$=1.0511\text{X}10^{-5}\text{moles X}202.20\frac{\text{g}}{\text{mol}}$$

$$= 2.1253\text{X}10^{-3}\text{g of BNOA}$$

Estimated % loading.

= Weight of intercalated BNOA/ Sample weight X 100

$$=\frac{2.1253\text{X}10^{-3}\text{g}}{2.534\text{X}10^{-3}\text{g}}\text{X}100$$

= 83.87%

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