SYNTHESIS AND CONFORMATIONS OF *p*-(ACETAMIDO)-BUTOXYCALIX[4]ARENE AND *p*-(BENZAMIDO)-BUTOXYCALIX[4]ARENE

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ABSTRACT

*The conversion of p-(amino)butoxycalix[4]arene, **1**, to p-(acetamido)butoxy-calix[4]arene, **2**, and p-(benzamido)butoxycalix[4]arene, **3**, via acetylation and benzoylation reactions, respectively have been conducted. The acetylation reactions was conducted by reflux method and room temperature method. The reflux method was conducted by refluxed a solution of **1** and acetic anhydride in acetic acid glacial solvent for 12 hours to result **2** in 53.8% yield; while the room temperature method was conducted by stirred a solution of **1**, acetyl chloride, and dry pyridine in dry toluene solvent and inert atmosphere at room temperature for 24 hours to result **2** in 97.8% yield. By the room temperature method and using benzoyl chloride, the compound **3** was obtained in 67.1% yield. Structures of the both compounds were confirmed using IR, ^{1}H NMR, and ^{13}C NMR spectroscopy methods. Pursuant to the ^{1}H-NMR spectral patterns of their bridge methylene and calix aryl protons, they were known that the compound **3** exist in partial cone conformation; whereas the compound **2** exist in partial cone, cone, and 1,2-alternate conformations where the partial cone was the main conformer.*

Since was introduced by Gutsche in 1978 [1], calixarenes continue to attract attention from chemists because of their remarkable ability to act as selective ion carries and host molecules for inclusion of organic compound [2]. The parent *p-tert*-butylcalix[4]arene of these compounds contains two interesting substructures. At the lower rim, four hydroxyl groups are present in very close proximity; this substructure can be used for cation binding [3] and transport [4]. The upper rim contains a hydrophobic cavity that is potentially able to complex neutral substrates [5]. Although the parent *p-tert*-butylcalix[4]arene are of limited utility, compounds derived from it have numerous application for building more complex structures, even some of them are selective to certain ion [1,4,6,7].

The extractability and selectivity of calixarenes have remarkable influenced by cavity size, position and kind of donor groups, and hydrophobicity ligand's [8,9]. Introducing other groups to the lower or/and upper, or convert them to other groups via certain reactions can increase their extractibility and selectivity. The introducing tetraketones or tetraesters groups to the lower rim of *p-tert*-butylcalix[4]arene giving sodium-selective ligands [1,10], the amide group giving heavy metals-selective ligands [11,12], and tetrakis(*N,N*-dietilaminocarbonyl-methoxy) derivative of *p*-(1,1,3,3-tetra-methylbutyl)calix[4]arene is selective for ion silver [13]. Most of amide-derivatives specially urea-derivatives of *p-tert*-butylcalix[4]arene exhibit selectivity for an anion [14-17].

Comparing to via lower rim, only limited research have been conducted to improved the selectivity of calix[4]arene compounds via modification of their upper rim groups, though at least there are two advantages from the functionalization of the upper rim of calix[4]arene. First, the cavity of upper rim is wider than lower rim, so that the functionalized upper rim compatible to trap larger ions or neutral molecules. The secondly is in application, if we need to immobilize calix[4]arene onto polymer surface, for example the immobilized calix[4]arene via lower rim hydroxyl groups onto silica that was conducted by Katz *et al.* [18], the functionalized upper rim still available to be host for ions or neutral molecules.

Beside of the size and functional groups of calix[4]arene, conformational is an important factor that governed selectivity of the calix[4]arene compounds. The calix[4]arene tetraalkyled derivatives can exist in
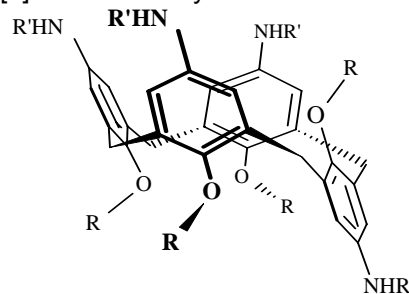
1 : R = B u, R' = H

2 : R = B u, R' = A c

3 : R = B u, R' = B z

Fig 1. Structures of precursor and target compounds

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four conformation, i.e. cone, partial cone, 1,2-alternate, and 1,3-alternate [19]; the cone is the preferable conformation for hosting ions or neutral molecules. In our previous research, we have been functionalized the upper rim of p-t-butylcalix[4]arene with amino group and blocked its lower rim with butyl group via etherification, ipso nitration, reduction reactions to resulted p-(amino) butoxycalix [4]arene, **1**, (the systematic name : 5,11,17,23-tetra-amino-25,26,27,28-tetrabutoxy-calix[4]arene) compound as a partial cone conformer [20]. In this research, the compound has been converted to p-(acetamido)butoxycalix[4]arene, **2**, (systematic name : 5,11,17,23-tetra-acetamido-25,26,27,28-tetra-butoxycalix[4]arene) via acetylation reaction by two methods, i.e. reflux method using acetic anhydride reagent, and room temperature method using acetyl chloride reagent; and to p-(benzamido)butoxy-calix[4]arene, **3**, (the systematic name : 5,11,17,23-tetra-(benzamido)-25,26,27,28-tetrabutoxycalix[4]arene) via benzoylation reactions using benzoyl chloride reagent for a purpose to find out calix[4]arene derivatives compounds that having high extractability and selectivity to a heavy metals ion or an anion. The structures and conformations of the both products were confirmed using IR, ^{13}C-NMR, and ^{1}H-NMR spectroscopy methods.

As long as our literature investigation, we never find a report about the synthesis method of the compounds, or that the compounds have been synthesized.

EXPERIMENTAL SECTION

Material

The p-(amino)butoxycalix[4]arene was synthe-sized according to literature [20], anhydride acetic p.a. (Merck), acetyl chloride p.a. (Merck), acetic acid glacial p.a. (Merck), benzoyl chloride p.a. (Merck), chloroform p.a. (Merck), toluene p.a. (Merck), pyridine p.a. (Merck), ethyl acetate p.a. (Merck), n-hexane p.a. (Merck), sodium sulfate anhydrous p.a. (Merck), and nitrogen gas.

Instrumentation

Reflux glass apparatus, rotary evaporator (Buchii R 124), heating mantle and stirrer, magnetic stirrer, melting point apparatus (electrothermal 9100), IR spectrophotometer (Shimadzu FTIR-8201 PC), NMR spectrometer (JEOL 500 MHz), and other standard glass apparatus in organic chemistry laboratory.

Procedures

Synthesis of 5,11,17,23-tetra-acetamido-25,26,27,28-tetrabutoxycalix[4]arene

Reflux method,- The solution of 0.50 g (0.70 mmol) p-aminobutoxycalix[4]arene in 5.0 mL acetic acid glacial was added 1.5 mL (1.62 g = 15.87 mmol) of acetic acid anhydrate and stirred for 5 min. The yellow solution was heated at refluxed at 120 °C for 12 h, cooled in ice bath, added 25 mL of water, stirred for 5 min, and filtered. The solid was washed with 25 mL of water, dried in desiccator, boiled in chloroform for 5 min, cooled, and filtered to obtained 0.40 g of yellow amorphous (65,2%), mp. 325-327 °C. The pale-pink amorphous of compound **2** was obtained in 53.8% yield (0.33 g) after recrystallized from chloroform-hexane solvents, mp. 329 °C; IR (KBr) 3301.9 cm^{-1} (single and sharp, N-H stretching), 1666.4 cm^{-1} (amide I band), 1604.7 cm^{-1} (C=C stretching of aromatic), 1550.7 cm^{-1} (amide II band), 1465.8 cm^{-1} (CH_2 bending), 1411.8 cm^{-1} (C-N stretching amide), 1373.2 cm^{-1} (CH_3 bending), 1218.9 cm^{-1} (C-O-C asymmetric stretching), 1033.8 cm^{-1} (C-O-C symmetric stretching).

Room temperature method,- A solution of 0.5 g (0.7 mmol) p-(amino)butoxy-calix[4]arene in 25 mL dry toluene was placed in a 100-mL three-necked round-bottomed flask fitted with a mechanical stirrer, ball condenser, and a gas nitrogen inlet tube to gave inert atmosphere. The solution was added 0.25 g (3.16 mmol) of dry pyridine, 1.0 mL (1.10 g = 14.00 mmol) of acetyl chloride, and stirred for 24 h at room temperature. The acetyl chloride excess was denaturated with 30 mL of ice-water and stirred for 15 minutes. The insoluble materials was filtered, dried in desiccator, and recrystallized from ethyl acetate-hexane solvent to yield 0.60 g (97.9%) of **2** as pale-pink amorphous; mp. 327-329 °C; IR (KBr) 3263.3 cm^{-1} (single and sharp, N-H stretching), 1665.2 cm^{-1} (amide I band), 1546.8 cm^{-1} (amide II band); ^{1}H-NMR (DMSO-d6) δ 9.8449, 9.6249, and 9.3001 ppm (s, 1:1:2, **H**-N of partial cone), δ 9.5600 ppm (s, **H**-N of cone), δ 8.7350, 8.7265 ppm (s, 1:1, **H**-N of 1,2-alternate), δ 7.7218 and 7.7059 ppm (d, J = 6.10 Hz , 1:1, **H**-Ar of 1,2-alternate), δ 7.5409 and 7.3429 ppm (s, 1:1, **H**-Ar of partial cone), 7,3069 and 6,4568 pppm (d, 1:1, **H**-Ar of partial cone), δ 7.2830 ppm (s, **H**-Ar of cone), δ 2.0620, 1.9991, and 1.8683 ppm (s, 1:1:2, C**H**$_3$-CO of partial cone), δ 1.9026 ppm (s, C**H**$_3$-CO of 1,2-alternate), δ 1.0470, 0.8612, and 0.8025 ppm (t, J = 6,75-7,35 Hz, 2:1:1, C**H**$_3$(CH$_2$)$_3$O of partial cone), δ 0.9260 ppm (t, J = 7,30-7,95 Hz, C**H**$_3$(CH$_2$)$_3$O of 1,2-alternate), δ 0,7377 ppm (t, J = 7,35 Hz, C**H**$_3$(CH$_2$)$_3$O of cone); ^{13}C-NMR (DMSO-d6) δ 167.4385 ppm (**C**=O), δ 56.1740 ppm (**C**H$_3$-C=O), δ 432.4901, 125.6416, and 119.6229 ppm (Ar of calix), δ 73.9630, 31.8704, 18.8219, and 14,0242 ppm (**C** of butoxy), δ 23.7246 ppm (Ar**C**H$_2$Ar).

Synthesis of 5,11,17,23-tetrabenzamido-25,26,27,28-tetrabutoxycalix[4]arene

A solution of 0.5 g (0.7 mmol) p-(amino)butoxycalix[4]arene in 25 mL dry toluene was placed in a 100-mL three-necked round-bottomed flask

fitted with a mechanical stirrer, ball condenser, and a gas nitrogen inlet tube to gave inert atmosphere. The solution was added 0.25 g (3.16 mmol) of dry pyridine, 1.0 mL (1.21 g = 8.60 mmol) of benzoyl chloride, and stirred for 24 h at room temperature. The mixture was filtered, and the filtrate was washed 2 times with 20 mL of ice-water. After was dried with sodium sulfate, the organic layer was evaporated to leave pink-solid, and than the pink-solid was recrystallized from ethyl acetate-hexane solvents to yield 0.53 g (67.1%) of **3** as pale-pink amorphous; mp. 274 ^{o}C; IR (KBr) 3367.5 cm^{-1} (single and sharp, N-H stretching), 1670.2 cm^{-1} (amide I band), 1527.5 cm^{-1} (amide II band); ^{1}H-NMR (CDCl$_3$) δ 7.9938, 7.9619, and 7.5639 ppm (s, 2:1:1, *H*-N), δ 7.9253, 7.8794, and 7.6319 ppm (d, 1:1:2, J = 7.30-7,35 Hz, *ortho H*-Ar of benzoyl), δ 7.5867 and 7.3761 ppm (s, 1:1, *H*-Ar of calix), δ 7.5561, δ 7.4974, and δ 7.1515 ppm (t, 1:1:2, J = 7.90-7.95 Hz, *meta H*-Ar of benzoyl), δ 7.5219 and 7.3410 ppm (t,1:1, J= 7,35 Hz, *para H*-Ar of benzoyl), δ 7,3056 and 6,5527 ppm (d, 1:1, J = 2,45 Hz, *H*-Ar of calix), δ 6.5527 and 5.3057 ppm (d, 1:1, J = 2.45 Hz, *H*-Ar of calix), δ 4.1167 and 3.0985 ppm (d, 1:1, J = 13.40-13.45 Hz, ArC*H₂*Ar), δ 3.7109 ppm (s, 1, ArC*H₂*Ar), δ 1.0096, 0.8899, and 0.8006 ppm (t, 2:1:1, J = 7.30-7.35 Hz, C*H₃*(CH$_2$)$_3$O); ^{13}C-NMR (CDCl$_3$) δ 164.8335 ppm (*C*=O), δ 152.7103, 127.5482, 127.4624, 121.7394 ppm (Ar of calix), δ 135.000, 132.0000, 133.0000, 127.3861 ppm (Ar of benzoyl), δ 73.9620, 31.8884, 19.2692, and 14.1185 ppm (*C* of butoxy), δ 30.7152 ppm (Ar*C*H$_2$Ar).

RESULT AND DISCUSSION

Synthesis of 5,11,17,23-tetra-acetamido-25,26,27,28-tetrabutoxycalix[4]arene, 2

The compound **2** was synthesized via acetylation reaction by reflux and room temperature methods. In the reflux method, the acetylation reaction was performed by

refluxed the mixture of compound **1** and acetic anhydride (4.2 equivalent to amino group) in acetic glacial solvent for 12 hours to gave pale-pink amorphous (mp. 329 ^{o}C) in 53.8% yield. Absorption bands at 1666.4 cm^{-1} and 1550.7 cm^{-1} as amide I and amide II band, respectively and absorption band of N-H stretching at 3301.9 cm^{-1} corroborate the existence of the compound **2** as product of this reaction. In the room temperature method, the mixture of compound **1**, and acetyl chloride reagent (3.9 equivalent to amino group), dry pyridine in dry toluene solvent was stirred in inert atmosphere for 24 hours to gave pale-pink amorphous (mp. 327-329 ^{o}C) in 97.8% yield. IR spectrum of this product display bands that similar with the correlating bands in IR spectrum of product of the first method, although both methods gave different yields. Absorption band of N-H stretching appear as singlet-sharp band at 3263.3 cm^{-1}, amide I appear at 1662.5 cm^{-1}, and amide II band appear at 1546.8 cm^{-1}. The both methods gave different yields due to the difference of reactivity of their acetylation agents. Acetyl chloride is more susceptible to nucleophile attack than acetic anhydride.

The compound **2** as product of the acetylation reaction was indicated in ^{13}C-NMR spectrum too (Fig 2). Signals at δ 167.4385 and 56.1740 ppm are corresponding to the carbonyl and methyl of acetyl group, respectively. These signals corroborate the existence of acetyl group in the compound. The other signals in the ^{13}C-NMR spectrum are similar with the correlating signal in the ^{13}C-NMR spectrum of the precursor compound [20].

In the spectrum ^{1}H-NMR (Fig 3), the protons of acetyl groups of compound **2** provide six singlet signals. These facts indicate that the compound is not in cone conformation. In the further investigation to the spectrum of methylene protons (Fig 4), we can see that there are a couple doublet signals at δ 3.9811 and
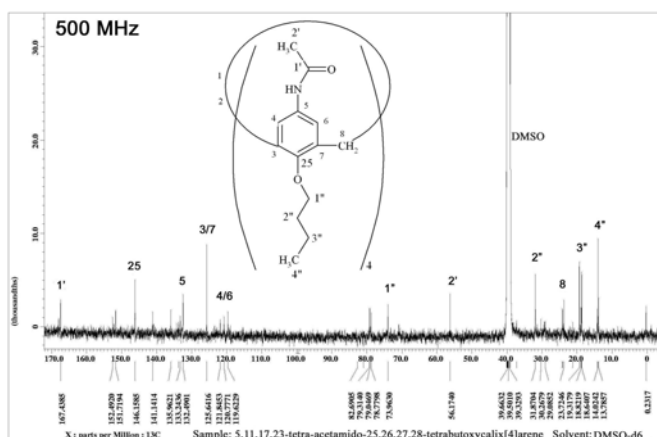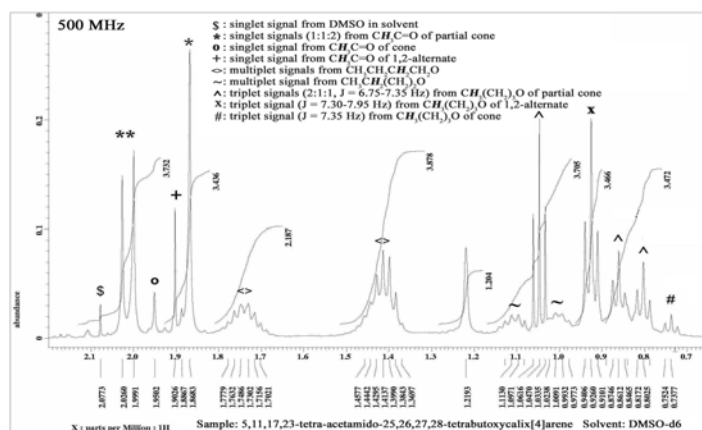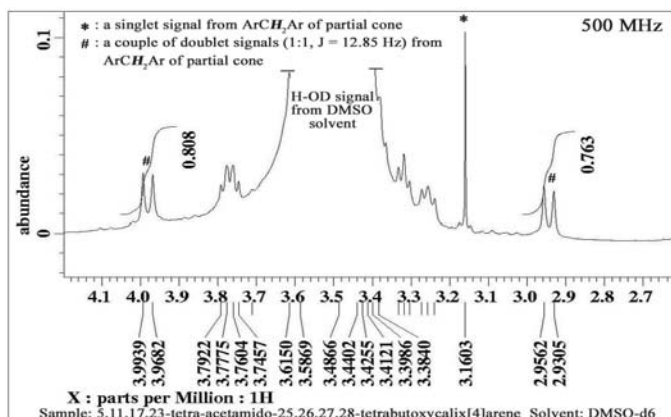

Fig 2. ^{13}C-NMR spectrum of compound **2**

Fig 3. ^{1}H-NMR spectrum of compound **2** in chemical shift area of butoxy groups protons

Fig 4. ^{1}H-NMR spectrum of compound **2** in chemical shift area of methylene groups protons
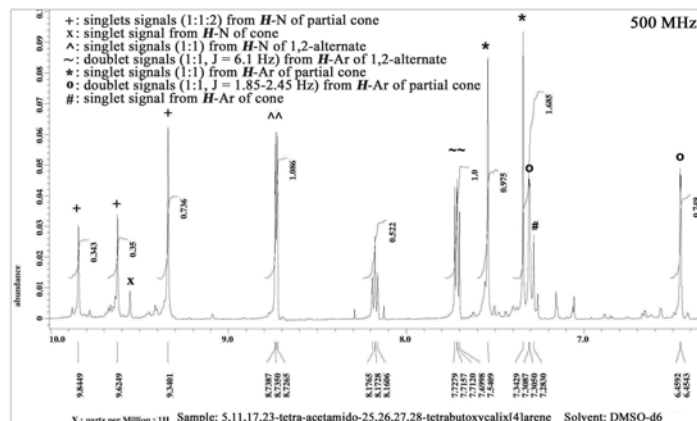

Fig 5. ^{1}H-NMR spectrum of compound **2** in chemical shift area of aryl groups protons

2.9434 ppm, and one singlet signal at 3.1603 ppm. The spectral pattern of methylene proton like that indicate that the calix[4]arene compound exist in partial cone conformation [19]. Beside the signals, there are gain signals that appear as a quartet and a triplet. These signals were probably arising from protons of butoxy groups.

To investigate the existence of the other conformer in this product via spectrum of methylene groups could not be performed because a pronounced interference from signal of H-OD as impurity in solvent (Fig 4). However, the signals from H-Ar of calix clearly indicate the existence of the other conformer. In the chemical shift area of aryl proton (Fig 5), beside of a couple singlet signals (δ 7.5409 and 7.3429 ppm, 1:1) and a couple doublet signals (δ 7,3069 and 6,4568 ppm, 1:1, J = 1,85-2,45 Hz) from proton aryl of partial cone conformer, there are a couple doublet signals (δ 7,7218 and 7,7059 ppm, 1:1, J = 6,10 Hz) and one singlet at δ 7.2870 ppm. These signals arising from aryl proton of 1,2-alternate and cone conformer, respectively.

Spectral patters of H-N signals and butoxy protons also corroborate the existence of the three conformers in the product of acetylation reaction. All of the conformers provide H-N signals as singlet signals (Fig 5). The partial cone conformer provides three singlet signals at δ 9.8449, 9.2649, and 9.30401 ppm with ratio 1:1:2; 1,2-alternate conformer provide two singlet signal at δ 8.7350 and 8.7265 ppm with ratio 1:1, and cone conformer provide one singlet signal near δ 9.5600 ppm. In the Fig 3, there are five triplet signals from methyl protons of butoxy groups. Three of them arising from methyl protons of partial cone conformer, i.e. signals at δ 1.0470, 0.8612, and 0.8025 ppm with ratio 2:1:1 and J = 6.75-7.35 Hz. These signals are compatible with the proton signal of acetyl groups at δ 2.0260, 1.9991, and 1.8683 ppm with ratio 2:1:1. The triplet signal of butoxy protons at δ 0.9260 ppm is corresponding to the proton signal of acetyl groups at δ 1.9026 ppm, and corroborate the existence of 1,2-alternate conformer. The further
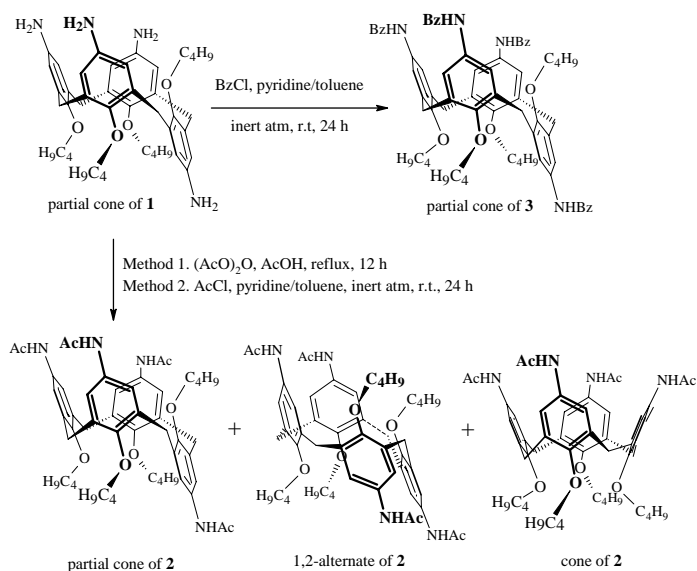
Fig 6. Acetilation and benzoylation reactions of **1** compound

signals, i.e. triplet signal of butoxy protons at δ 0.7377 ppm is corresponding the singlet signal of acetyl protons at δ 1.9502 ppm, and corroborate the existence of cone conformer. According to the height ratio of the correlating signals, the abundance order of **2** conformers in the product of acetylation reaction is partial cone > 1,2-alternate >> cone. The cone conformer can be regard as a trace. The overall reactions and conformations of their product in this research are shown in Fig 6.

Synthesis of 5,11,17,23-tetrabenzamido-25,26,27, 28-tetrabutoxycalix[4]arene, 3

The synthesis data of compound **2** via reaction acetylation reaction provide an information that the high yield was obtained when the reaction was performed
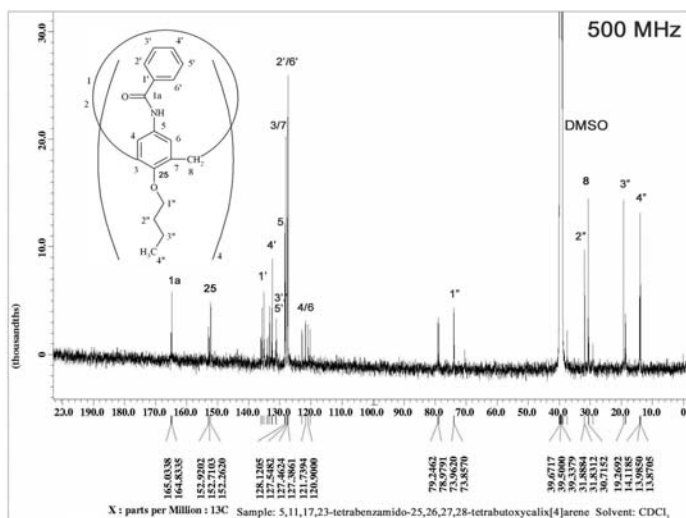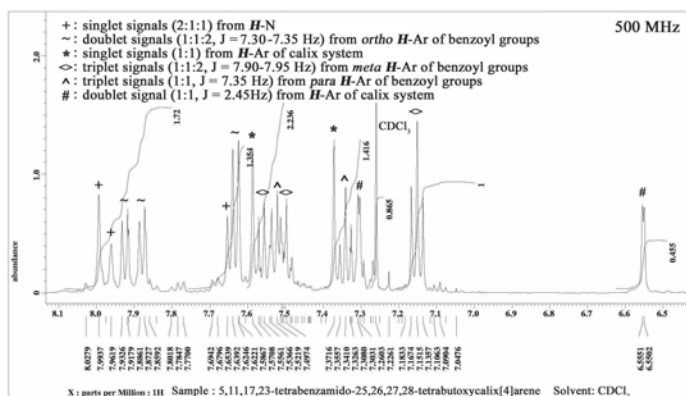
Fig 7. ^{13}C-NMR spectrum of **3** compound

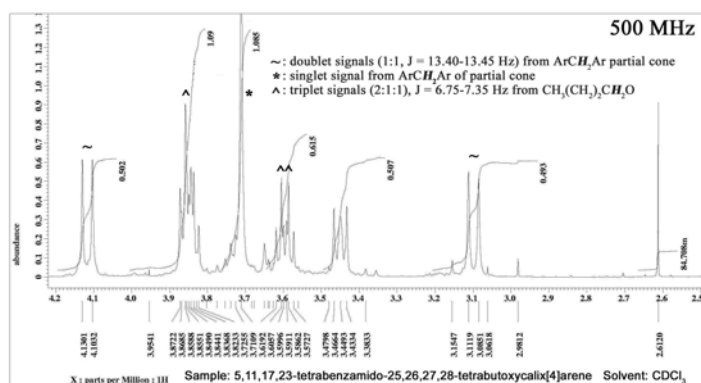**Fig 8.** ^{1}H-NMR spectrum of **3** compound in chemical shift area of aryl groups protons

Fig 9. ^{1}H-NMR spectrum of **3** compound in chemical shift area of methylene groups protons

using acetyl chloride as acetylation agent comparing when using acetic anhydride. Pursuant to the facts, the synthesis of compound **3** was performed using benzoyl chloride as benzoylation agent. In this synthesis, the mixture of compound **1** and benzoyl chloride (3.1 equivalent to amino group) in dry pyridine and dry toluene solvent was stirred at room temperature and in
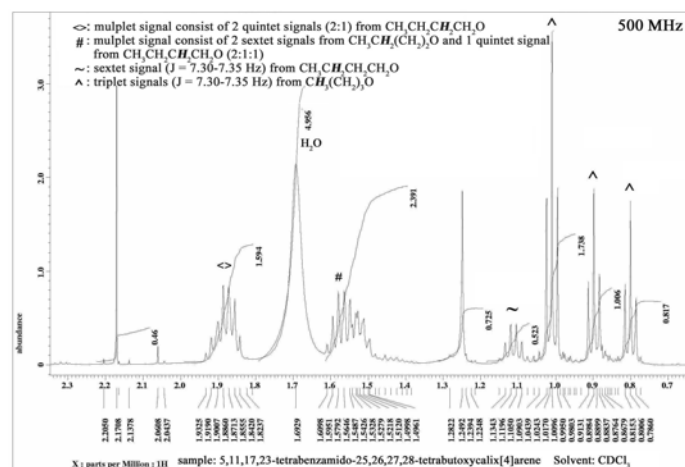

Fig 10. ^{1}H-NMR spectrum of **3** compound in chemical shift area of butoxy groups protons

inert atmosphere for 24 hours to gave pale-pink amorphous in 67.1% yield, mp. 274°C. Absorption bands at 3367.5, 1670.2, and 1527.5 cm^{-1} in the IR spectrum of the compound indicates that the benzoylation reaction have been successful. The IR data are corresponding to the signals in the ^{13}C-NMR spectrum (Fig 7) that contains carbonyl signal at δ 164.8335 ppm and Ar of benzoyl group signals in δ 135.0000-127.0000 ppm.

The introducing of benzoyl groups to the compound **1** provide complicated spectrum in the chemical shift area of aryl protons (Fig 8). The groups provide at least five triplets and three doublets in the spectrum. The doublet signals at δ 7.9253, 7.8794, and 7.6319 ppm (1:1:2), J = 7.30-7.35 Hz) arising from *ortho* protons, the triplet signals at δ 7.5561, 7.4974, and 7.1515 ppm (1:1:2, J = 7.90-7.95 Hz) arising from *meta* proton, and the triplet signals at δ 7.5219 and 7.3410 ppm (1:1, J = 7.35 Hz) arising from *para* protons. Signals of aryl protons of calix system arising as a couple of singlet signals at δ 7.5867 and 7.3716 ppm, and a couple of doublet signals at δ 5.3057 and 6.5527 ppm (1:1, J = 2.45 Hz). This spectral pattern of aryl protons of calix sytem is corresponding to the spectral pattern of bridge methylene protons (Fig 9). In the Figure 9, signals of bridge methylene protons arising as a couple doublet signals at δ 4.1167 and 3.0985 ppm (1:1, J = 13.40-13.45 Hz), and one singlet at δ 3.7109 ppm. The spectral pattern of aryl protons of calix system as a couple singlet signals and a couple doublet signals, and of bridge methylene protons as a couple doublet signals and a singlet signal indicate that the compound **3** exist in partial conformation [19]. This case is supported by the spectral pattern of N-H groups that appear as three singlet signals at δ 7.9937, 7.9619, and 7.5639 ppm with ratio 2:1:1 (Fig 9); and spectral pattern of butoxy protons as three triplet signals at δ 1.0096, 0.8899, and 0.8006 ppm (J = 7.30-

7.35 Hz) with ratio 2:1:1 (Fig 10). The other multiplet signals in protons butoxy spectrum are arising from methylene protons of butoxy groups.

The overall spectral patterns from calix aryl, bridge methylene, N-H, and butoxy protons support that the compound that was obtained from benzoylation reaction of compound **1** is the partial cone conformer of **3** as single product, while the product of acetylation reaction contains three conformers, i.e. partial cone, 1,2-alternate, and cone conformer. These facts provide information that the modification the upper rim groups of calix[4]arene sometime result conformation changes too, depend on the size of the additional groups. Bulky groups tend to suppress the product of reaction frozen in one conformation.

In addition, the interesting cases come from the positions of N-H signals and their spectral pattern. Each of the compounds provides three singlet signals of N-H groups with different ratio pattern and position. The partial cone of **2** provide three singlet signal with ratio 1:1:2 (Fig 5), while the partial cone of **3** provide three singlet signal with ratio 2:1:1 (Fig 8). The singlet signals from **2** arising at more deshielding area than that from **3**. Apparently, in these cases, the size group is pronounced factor than induction efect. Due to the benzoyl group is more bulky than the acetyl group, the shielding to the H-N groups from benzoyl groups in **3** is higher than from acetyl groups in **2**.

CONCLUSION

The obtained results showed that the Freundlich-type model is suitable to describe the transfer of chromium from water to root plant for both   water hyacinth (*Eichhornia crassipes (Mart.) Solms*) and water lettuce (*Pistia statiotes L.*). Beside the easy handling, the Freundlich model offers interpretation potential. The results suggest that the coefficients K_F and n of the Freundlich-type function reflect the plant's capability to regulate heavy metal concentrations in their tissue.

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