Functionalization of C-4-methoxyphenylcalix[4]resorcinarene with several ammonium compounds

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Abstract. The synthesis of new quaternary ammonium modified calix[4]resorcinarenes were accomplished by the reaction of C-4-methoxyphenylcalix[4]resorcinarene (I) with various ammonium compounds i.e. (1) (3-chloro-2-hydroxypropyl)trimethylammonium chloride, (2) glycidyltrimethylammonium chloride, and (3) N-hexadecyl-N,N,N-trimethylammonium bromide. The modification processes were carried out through just over one-step reaction by using minimal solvent and suitable reaction conditions to yield resorcinarene II, III, and IV, respectively. All products were characterized by FT-IR, 1H-NMR, and 13C-NMR data. Solid morphology of some resorcinarenes was determined using SEM equipped with EDX detector. Based on the spectral properties of 1H-NMR, all calixresorcinarene compounds were isolated in the molecular shape of the chair (C2h) conformation.

1. Introduction
Modifications on calixarene compounds have been carried out primarily for phenol and substituted phenol derivatives calixarenes [1-6]. However, it is different with resorcinol derivatives calixarenes (resorcinarenes) due to their low solubility in various organic solvents [7,8]. C-arylcalix[4]resorcinarenes, for example, only dissolve in warm DMF and DMSO. In fact, there are a lot of resorcinarenes which can be synthesized from resorcinol and its derivatives with various of both aliphatic [9-11] and aromatic aldehydes [7, 10, 12].

In the field of supramolecular chemistry, it is very important to make the calixarenes have multifunction as hosts for cations [13], anions, and neutral organic molecules via further functionalization or modification. Calix[4]resorcinarenes provide a cavity, which in combination with several active sites, can be modified by suitable substitution at their upper rim of methylene bridges [7, 12] and extra annular –OH groups [14, 15]. Functionalization on these sites has been intensively studied through aminomethylation by refluxing the C-methylcalix[4]resorcarenare and C-phenylcalix[4]resorcinarene with a primary amine and formaldehyde in mixtures of benzene with the minimum amount of ethanol to achieve a homogeneous reaction mixture [15].

In our previous work, we then introduce this method in order to obtain quaternary ammonium modified resorcinarenes by reacting C-3,4-dimethoxyphenylcalix[4]resorcinarene with formaldehyde and dimethyamine in the presence of HCl according to the Mannich procedure followed by alkylation with MeI. This resorcinarene, tetrakis-N,N,N-trimethylammoniummethyl-C-3,4-dimethoxyphenylcalix[4]resorcinarene iodide, has been proven to chelate and reduce the toxic chromium (VI) content.
in serum, liver, and kidney of mice [12]. Unfortunately, this procedure synthesis needs long steps wise of reactions and consume more solvents and reagents. For this reason, this research study the synthesis of similar compounds were conducted through just over one-step reaction. The modification processes are expected to take place directly to the C-4-methoxyphenylcalix[4]resorcinarene using various quaternary ammonium compounds with minimal solvent and suitable reaction conditions.

The existence of two types of active groups located far apart in the targeted resorcinarenes (Figure 1 and 2), hydroxyl and the quaternary ammonium groups, make the compounds can act as host molecules for both of anionic and cationic heavy metals. Quaternary ammonium active sides will be able to bind anionic species while the hydroxyl groups will be able to bind cationic species.

2. Experimental

2.1. Materials
Resorcinol, 4-methoxybenzaldehyde, ethanol, fuming hydrochloric acid, dichloromethane, sodium hydroxide, ethyl acetic, acetone, dimethylformamide, dimethyl sulfoxide, glycidyltrimethylammonium chloride, hexadecyltrimethylammonium bromide (HDTMA-Br), and (3-Chloro-2-hydroxypropyl)trimethylammonium chloride solution. All reagents of analytical grade were obtained from E Merck Co Inc. (Germany), and Sigma-Aldrich used without further purification.

2.2. Instruments
Melting points were determined with an electrothermal IA9100I and were uncorrected. The homogeneity of all the newly synthesized compounds was routinely checked by TLC on silica gel 7730 60GF
d plates and spots were observed by short and long wavelength ultraviolet light. IR spectra were recorded on a Fourier-transform infrared spectrometer (Shimadzu FTIR-8201 PC) in KBr pellets. H-NMR and C-NMR spectra were performed on a Bruker AC300F and an Aligent400 instruments using DMSO-d6 as solvent and Tetramethysilane (TMS) as an internal reference. Solids morphology were determined using SEM supplied with EDX detector (JSM-6360).

2.3. Procedures

2.3.1. Synthesis of C-4-methoxyphenylcalix[4]resorcinarene (I). Compound I was synthesized from 4-methoxybenzaldehyde according to the previous procedure [7] with slight modification. The mixture of 4-methoxybenzaldehyde (1.66 g; 10 mmol), resorcinol (1.1 g; 10 mmol), ethanol absolute (100 mL), and fuming hydrochloric acid (0.5 mL) was refluxed under inert condition for 18 hours until the spot of 4-methoxybenzaldehyde totally used up at the reaction (monitoring by TLC). The mixture was cooled, and the product was filtered followed by washing with ethanol-water (1:1) and acetone, respectively. Finally, the product was filtered followed by washing with ethanol-water (1:1) and acetone, respectively. Finally, the product was dried to give the desired compound as a light purple crystal in 94.26%, mp 339-341°C (dec); FTIR (KBr) v (cm⁻¹): 3397 (OH group), 3007 (Csp2-H), 1605 and 1505 (C=C aromatic), 2910-2834 (Csp3-H aliphatic), 1432 (-CH methine bridge), 1368 (-CH3); H-NMR (500 MHz, DMSO-d6) δ (ppm): 7.92 and 8.03 (8H,s,O-H), 6.16-6.56 (24H,m,ArH), 5.53 and 5.68 (4H,s,Ar2-CHAr), 3.66 (12H,s,OCH3).

2.3.2. Synthesis of 5,17-di(2-hydroxypropyl trimethyl ammonium chloride)-C-4-methoxyphenylcalix[4]resorcinarene (II). Resorcinarene I (0912 g, 1.0 mmol) was dissolved in 30 mL of warm DMF accompanied by stirring. Into a solution of resorcinarene I was then added (3-Chloro-2-hydroxypropyl)trimethylammonium chloride and 0.5 mL of fuming hydrochloric acid respectively. The mixture was stirred and heated at 100°C under inert condition over the night (18 hours) until the stain material on the basis of TLC spots has completely reacted. The solvent was then removed on the proper evaporator and the residue was triturated with distilled water. The mixture was cooled, and the precipitate was filtered off. The precipitate that formed was washed with ethanol: water (1: 1),
recrystallized from acetone-water and dried to yield 0.705 g (58.02%) of resorcinarene II; mp. 362 °C; FTIR (KBr) v (cm⁻¹): 3245 (OH), 3026 (C(sp²)-H), 1607 and 1511 (C=O aromatic), 3000-2756 (C(sp³)-H aliphatic), 1474 (-CH₂-), 1425 (-CH methine bridge), 1375 (-CH₃), 1249 and 1201 (C-N), 1077 (-O-CH₃); 1H-NMR (300 MHz, DMSO-d₆) δ (ppm): 8.47 (8H,s,OH resorcin), 8.00 (2H,s,OH hydroxypropyl), 6.13-6.54 (22H,m,ArH), 5.50 (4H,d of s,Ar-CHAr), 4.15 (2H,m,-CH(OH)-), 3.65 (12H,s,OH), 3.18 (18H,s,N-CH₃), 2.91 (4H,d,-CH₂-N), 2.75 (4H,s,Ar-CH₂-); 13C-NMR (125 MHz, DMSO-d₆) δ (ppm): 156.8 (C=O-Me), 152.9, 152.8 (Ar-C=O), 136.6, 130.1, 121.7, 121.3, 112.7 (Ar), 54.9 (-C-N), 41.6 (-C-OH), 40.7 (O-CH₃), 36.2 (N-CH₃), 31.0 (Ar-CHAr), 21.5 (Ar-CH₂-).

2.3.3. Synthesis of 6,18-di(2-hydroxypropyl trimethyl ammonium chloride)-C-4-methoxyphenylcalix[4]resorcinarene (III) (solvent free). Into 30 mL of NaOH solution (0.01 M) was added resorcinarene I (0.912 g, 1.0 mmol) then stirred and heated at temperatures of 35 °C until the sample dissolved completely. To this homogeneous mixture, glycidyltrimethylammonium chloride (0.671 mL; 2.5 mmol) was inserted carefully and refluxed under the mild condition for 15 hours (by TLC observation) with constant stirring. The reaction mixture was cooled to room temperature, and the resulting pink precipitates were filtered instantly over a Buchner filtration with the help of vacuum and washed thoroughly with acetone. The product was dried under reduced pressure to give 0.764 g (54.9% yield). 

The result obtained was 6,18-di(2-hydroxypropyl trimethyl ammonium chloride)-C-4-methoxyphenylcalix[4]resorcinarene (III) (solvent free).

2.3.4. Functionalization of C-4-methoxyphenylcalix[4]resorcinarene with HDTMA-Br (IV) (solvent free). Resorcinarene I (0.912 g; 1.0 mmol) was added to a solution of 35 mL HDTMA-Br (1 mol/L) in a round bottom flask. The critical micelle concentration (CMC) of HDTMA-Br at 30 °C was 0.94 mmol/L as estimated by conductivity measurements of aqueous HDTMA-Br solutions at different concentrations [17]. The mixture was stirred at room temperature and then added 0.1 M NaOH dropwise until completely dissolved. The mixture was further stirred at a temperature of 70 °C for 18 h, and the reaction was monitored by TLC. Trituration with 0.01M HCl until neutral and the precipitate was filtered, recrystallized using ethanol: water (1:1) and dried to obtain a fixed weight of the desired product (0.814 g; 60.85%); mp. 358 °C; FTIR (KBr) v (cm⁻¹): 3373 (OH), 3231 (C(sp²)-H), 1608 dan 1511 (C=O aromatic), 2993-2838 (C(sp³)-H aliphatic), 1473 (-CH methine bridge), 1383 (-CH₃), 1257-1183 (C-N), 1077 (-O-CH₃); 1H-NMR (400 MHz, DMSO-d₆) δ (ppm): 8.60 and 8.55 (6H,s,OH resorcin), 8.00 (2H,s,OH hydroxypropyl), 6.05-6.44 (24H,m,ArH), 5.45 (4H,d,-CH₂-N), 3.99 (18H,s,N-CH₃); 13C-NMR (125 MHz, DMSO-d₆) δ (ppm): 162.7 (-C=O-Me), 157.6 (Ar-C=O), 152.6 (-C=O-), 133.9, 133.4, 130.3, 129.8, 124.4, 123.5, 113.3, 107.7, 107.2 (Ar), 55.1 (Ar-O-C), 51.3 (-C-N), 43.7 (-C-OH), 42.6 (-O-CH₃), 36.2 (N-CH₃), 31.1 (Ar-CHAr).

2.3.5. Functionalization of C-4-methoxyphenylcalix[4]resorcinarene with HDTMA-Br (IV) (solvent free). Resorcinarene I (0.912 g; 1.0 mmol) then stirred and heated at temperatures of 35 °C until the sample dissolved completely. To this homogeneous mixture, glycidyltrimethylammonium chloride (0.671 mL; 2.5 mmol) was inserted carefully and refluxed under the mild condition for 15 hours (by TLC observation) with constant stirring. The reaction mixture was cooled to room temperature, and the resulting pink precipitates were filtered instantly over a Buchner filtration with the help of vacuum and washed thoroughly with acetone. The product was dried under reduced pressure to give 0.764 g (62.88%) of ammonium modified resorcinarene II; mp. 369 °C; FTIR (KBr) v (cm⁻¹): 3373 (OH), 2993-2838 (C(sp³)-H aliphatic), 1473 (-CH methine bridge), 1383 (-CH₃), 1257-1183 (C-N), 1077 (-O-CH₃); 1H-NMR (400 MHz, DMSO-d₆) δ (ppm): 8.60 and 8.55 (6H,s,OH resorcin), 8.00 (2H,s,OH hydroxypropyl), 6.05-6.44 (24H,m,ArH), 5.45 (4H,d,-CH₂-N), 3.99 (18H,s,N-CH₃); 13C-NMR (125 MHz, DMSO-d₆) δ (ppm): 162.7 (-C=O-Me), 157.6 (Ar-C=O), 152.6 (-C=O-), 133.9, 133.4, 130.3, 129.8, 124.4, 123.5, 113.3, 107.7, 107.2 (Ar), 55.1 (Ar-O-C), 51.3 (-C-N), 43.7 (-C-OH), 42.6 (-O-CH₃), 36.2 (N-CH₃), 31.1 (Ar-CHAr).

3. Results and discussion

Synthesis of resorcinarene I in this study has a slightly different technique with the method that we have previously reported [7,16]. At this time, the synthesis reaction is made in an inert atmosphere so that the catalyst consumption can be minimized. The effectiveness of HCl as the catalyst was strongly influenced by the presence of water vapor and oxygen. In inert conditions, the two inference components do not exist so that the reaction can be carried out effectively, minimum catalyst consumption, faster time, and more yield. The product of resorcinarene I was obtained as a light purple crystal in 94.26% yield with a melting point of 339-341 °C. Based on the proton NMR
investigation, resorcinarene I was observed in chair conformation proven by the exhibit of two singlet signals on four methine bridges.

Based on the structure and functional groups that are owned by resorcinarene I, the compound allows to be modified through some reaction on the accordingly active sides. Not only at the ortho position of the hydroxyl group alone (aromatic ring), as has been reported previously [18], but the eight hydroxyl groups can also be further functionalized through the base catalyzed reaction. However, in contrast, to the methoxy groups on the resorcinarene compound are difficult to be modified due to the low reactivity. In this publication, we report several possible functionalization on compound I.

3.1. Modification on C atom number 5 and 17 (aromatic ring)

3.1.1. Synthesis of 5,17-di(2-hydroxypropyl trimethyl ammonium chloride)-C-4-methoxyphenylcalix[4]resorcinarene (II). Due to the existence of two reactive sites on resorcinarene I, the synthesis of ammonium modified resorcinarene II can only accomplish under certain conditions, i.e. under acid catalyzed reaction. In acidic condition, the eight phenolic hydroxyl groups, that are also acidic, are inactive sides. Therefore, the central of reaction occurs only on the aromatic rings. In this case, the most reactive position held by carbon atoms of 5, 11, 17, and 23 in resource residues where the atoms are located at the ortho position of the hydroxyl groups.

At this stage of the research, modification process was done by using 3-Chloro-2-hydroxypropyl trimethylammonium chloride solution in the presence of fuming hydrochloric acid. Reactions involved in this process is the electrophilic aromatic substitution reaction as shown in Figure 1. Functionalization was held only on two position i.e. carbon atoms of number 5 and 17 by using a molar ratio of 1:2.5 of resorcinarene I: 3-Chloro-2-hydroxypropyl) trimethylammonium chloride.

From the research that has been conducted, the product compound was obtained as brown crystalline solids in 58.02% yield with the melting point of 362 °C. Based on the solubility assessment, resorcinarene II was not soluble in water, chloroform, and dichloromethane, slightly soluble in dimethyl sulfoxide and acetonitrile, but soluble in NaOH solution and hot dimethylformamide.
3.2. Modification of hydroxyl groups

3.2.1. Synthesis of 6,18-di(2-hydroxypropyl trimethyl ammonium chloride)-C-4-methoxyphenylcalix[4]resorcinarene (III) (solvent free). In alkaline conditions, from three active sites on resorcinarene I only hydroxyl groups that became the center of the reaction. Eighth phenolic OH groups were acidic, with the presence of a base causes the protons (H⁺) will be released so that the resulting of phenoxide ion capable of acting as a nucleophile towards reagent, in this case, was glycidyl trimethyl ammonium chloride.

Reactions involved in the synthesis of compound III from I was the epoxide ring opening reaction of glycidyl trimethyl ammonium chloride (Figure 2). The carbons in an epoxide group are very reactive electrophiles, due to in large part to the fact that substantial ring strain is relieved when the ring opens upon nucleophilic attack (phenoxide ion of the resorcinarene I). Ring-opening reactions can proceed by either SN1 or SN2 mechanisms, depending on the properties of the epoxide compound as well as on the reaction conditions. When an asymmetric epoxide (in this case: glycidyl trimethyl ammonium chloride) undergoes solvolysis in basic solution, the ring-opening occurs by an SN2 mechanism, and the less substituted carbon is the site of nucleophilic attack, leading to what we will refer to as the product of resorcinarene III.

![Figure 2. Based-catalyzed reaction in the synthesis of resorcinarene III.](image)

3.2.2. Functionalization of C-4-methoxyphenylcalix[4]resorcinarene with HDTMA-Br Surfactant (IV) (solvent free). As Mentioned before, resorcinarene I have multiple active sites, namely hydroxyl (-OH), methoxy (-OMe), and an aromatic ring. All of these groups have a negative character, its mean that the surface of resorcinarene I have a negative surface as can be described in figure 3. Meanwhile, the surfactant of HDTMA-Br has two distinct parts of characters. The tail of the alkyl chain is nonpolar (uncharged) while the head is polar side (+). Thus, when the HDTMA-Br were added into a solution of resorcinarene I, the negative sides on the resorcinarene will interact each other and form bonds with the polar end (+) of HDTMA-Br.
Attachment of HDTMA\(^+\) on the resorcinarene surface is strongly influenced by the degree of surfactant adsorption on a solid surface [17]. At very low concentrations, the HDTMA\(^+\) molecules move freely and can be lined up parallel to the surface. By increasing the concentration of HDTMA\(^+\), then the number of surfactant molecules on the surface also increases, so that there is no longer space for the surfactant for parallel lining, so it starts moving in one direction, in which direction depends on the nature of the hydrophilic group and the surface. At higher concentrations, in this study, a bilayer was formed due to the amount of HDTMA\(^+\) was higher (1 mol/L) than its Critical Micelle Concentration (CMC, \(9.4 \times 10^{-4}\) mol/L). In this condition, the hydrophobic tails of the surfactant molecules will associate to form a bilayer. Surfactant molecules form a monolayer at the solid-aqueous interface via strong Columbic interaction below its CMC. The formation of monolayer and bilayer is represented in figure 4.

![Figure 3. Mechanism of interaction between HDTMA\(^+\) with the surface of resorcinarene I](image)

**Figure 3.** Mechanism of interaction between HDTMA\(^+\) with the surface of resorcinarene I

**Figure 4.** Attachment of HDTMA\(^+\) on the resorcinarene surface. (a), (b) HDTMA\(^+\) < CMC. (c), (d) HDTMA\(^+\) > CMC.

3.3. The Molecular Properties of The Resorcinarenes

The success of the modification processes above was confirmed by the results of the data analysis by FTIR and NMR apparatus. The IR spectrum of resorcinarene II and III were displayed in Figure 5. The most notable difference between resorcinarene II and III is located in the absorption area on 3300 cm\(^{-1}\) correspond to vibrational of hydroxyl groups (-OH), in which its intensity on resorcinarene II higher than that of resorcinarene III. This fact strengthens the evidence that the acid-catalyzed functionalization only occurs on an aromatic ring such that the abundance of the hydroxyl groups was not reduced even increased with the inclusion of (3-Chloro-2-hydroxypropyl) trimethylammonium chloride. In contrast, base-catalyzed modification only activates the hydroxyl groups on resorcinarene I. This resulted in the replacement of the hydroxyl groups by quaternary ammonium groups so that the absorption intensity of hydroxyl groups was reduced (Figure 5).
Clear evidence of the formation of resorcinarene II and III were further confirmed by their NMR data (figure 6). The existence of several new absorption at chemical shifts on 4.15 (2H, m, -CH(OH)-), 3.18 (18H, s, N-CH$_3$), and 2.91 (4H, d, -CH$_2$-N) indicates that the functionalization with quaternary ammonium compound has been reached. Similar to the IR spectrum, resorcinarene II, and III also has differences in their H-NMR spectrum, mainly on the hydroxyl proton signals and the appearance of the aryl protons. In resorcinarene II, the protons of hydroxyl group are identified by absorption in two different areas (i.e., 8.47 for OH resorcin and 8.00 for OH hydroxypropyl). While on resorcinarene III appears only in one area that is at δ 8.51-8.60 ppm with the smaller integration of protons than that of resorcinarene II. Furthermore, aryl proton signal at resorcinarene II more simple than resorcinarene III. This means that the type of proton on resorcinarene II less than that on resorcinarene III. This indicates that the functionalization on resorcinarene II occurs in aromatic rings, while the resorcinarene III does not.

Based on the signal at δ 5.5 attributed to methine bridge protons at both resorcinarene I, II, and III have the same appearance i.e., two singlets. This is consistent with a chair conformation, adopted by the calix[4]resorcinarene as reported previous [11], where two protons at methine bridges are placed at opposite sides in relation to the others.
The IR spectra of resorcinarene $I$ and its modified forms (resorcinarene-HDTMA$^+$, $IV$) are presented in figure 7. It can be noticed that there is net difference between the original sample and the resorcinarene $IV$ characterized by the appearance of new bands at 2841-2974 cm$^{-1}$ related to the symmetric and asymmetric CH$_2$ and CH$_3$ stretching vibration. Their two bending vibrations located at 1467 and 1379 cm$^{-1}$, due to the methylene and methyl scissoring modes, could be observed in IR spectra of resorcinarene $IV$. These new bands indicating that HDTMA surfactant has been substituted on the surface of resorcinarene $I$ to yield resorcinarene $IV$.

Figure 6. H-NMR spectrum of resorcinarene $II$ and $III$. 

Figure 7. IR spectra of resorcinarene $I$ and its modified forms (resorcinarene-HDTMA$^+$, $IV$).
Other evidence for the modification of resorcinarene I by HDTMA$^+$ was obtained by Scanning Electron Microscopy- Energy Dispersive X-ray Spectroscopy (SEM/EDX). Based on SEM images (Figure 8), there are notable morphological differences between resorcinarene I and the prepared resorcinarene IV. For resorcinarene I (figure 8a) considerable pores are clearly observed. These characteristics are not visible in the SEM micrographs of the resorcinarene IV due to surfactant coverage on the external crystal surface (Figure 8b) confirming, thus, a good homogeneity within the resorcinarene-HDTMA$^+$. 

![Figure 8. SEM images of resorcinarene I (a) and IV (b).](image-url)
4. Conclusion
Functionalization of C-4-methoxyphenylcalix[4]resorcinarene (I) has been conducted with several ammonium compounds with differences reaction conditions. Modification of resorcinarene I with (3-Chloro-2-hydroxypropyl)trimethylammonium chloride has been done in the presence of fuming hydrochloric acid via electrophilic aromatic substitution to yield resorcinarene II. Meanwhile, base-catalyzed functionalization of resorcinarene I by using glycidiltrimethylammonium chloride results resorcinarene III. The modification process involves the epoxide ring opening reaction of the ammonium compound. Attachment of HDTMA\(^+\) on the resorcinarene I surface has been accomplished in the alkaline condition in the presence of an excess HDTMA-Br surfactant. Based on the spectral properties of \(^1\)H-NMR, all calixresorcinarene compounds were isolated in the molecular shape of the chair (C\(_{2h}\)) or flattened partial cone conformation. FT-IR, NMR, and SEM analyzes indicated that the N-hexadecyl-N,N,N-trimethylammonium bromide molecules were substituted on the surface of initially C-4-methoxyphenylcalix[4]resorcinarene.

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