

Solvent Extraction of Non Ferrous Metal using Calix(6)Arene

Shirin Imam

Department of Chemical Sciences, Christian Eminent College, Indore, India

Corresponding Author: drshirin14@gmail.com

Available online at: www.isroset.org

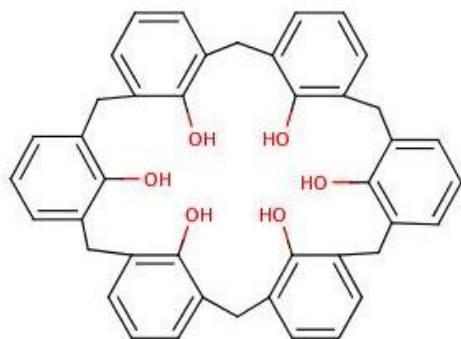
Received 20th Apr 2017, Revised 09th May 2017, Accepted 05th Jun 2017, Online 30th Jun 2017

Abstract— Calix(6) arenes are macrocyclic polyethers and supermolecular compounds having three dimensional structure of 4-8 bridged phenolic units with varying annular space in which metals can be trapped due to ionic interaction. The beauty of calix(6) arenes is that it is cone shaped with varying annular space ranging from 1.0-2.4. There is good possibility to change the annular space by varying *n* i.e. number of phenyl rings attached to the structure by having conformation as 1,3 or 1,2 alternate favoring the stereochemistry for the formation of complexes with metal with varying coordination number. Cobalt(II) was extracted at varying pH. At the pH range of 7-8 maximum extraction of cobalt is found. A stock solution of cobalt was prepared by dissolving cobalt chloride in distill water. A UV-visible spectrophotometer and digital pH meter was used in the experiment. It was seen that among various diluents toluene shows better separation. Cobalt(II) complex, was stripped by using 0.05 M nitric acid. The above method is simple, selective, fast and useful.

Keywords— Cobalt, spectrophotometer, calix(6)arene, pH, supermolecule

I. INTRODUCTION

Calix arenes are basically cyclic oligomers of phenol and formaldehyde having many phenolic units. It is characterized by a three-dimensional silo, cup or pail shape. Calix(6)arene has 6 units in the ring. Calixarene nomenclature is simple. It involves counting of the number of recurring units in the ring and include them in the name. The word calix arene is obtained from calix or trophy because the molecule resembles a pot and from the word arene that refers to the aromatic building block.



“Fig. 1”

Calix arenes have hydrophobic cavities or space that can hold smaller molecules or ions and belong to the class of cavitands known in host-guest chemistry. Calixarene chemistry has become an important aspect of supramolecular chemistry. An precise and exhaustive history of the calix

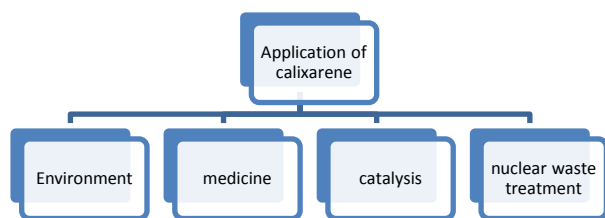
arenes along with extensive discussion of calix arene chemistry can be found in the 1989 publication [1],[4].

Conformational flexibility of calix[6]arenes and presence of functional groups facilitates the binding of metal centers, there are eight conformations of calix(6)arenes described in the literature³. They are called as distorted or fuzzy cone, packed in cone, pinched cone, double partial cone, winged, 1,2,3-alternate, 1,3,5-alternate, and distorted 1,2,3- alternate.

Table 1. Crystal data and structure

Empirical formula	C ₆₀ H ₆₆ C ₁₆ O ₁₂
Crystal system	Triclinic
Space group	P-1
Unit cell dimensions	a = 10.2536(13) = 102.215(3)°Å b = 13.0158(16) 92.621(3)°Å c = 23.641(3) 104.494(3)°Å

Calixarenes are very attractive to researchers from numerous science and technology fields. A method was studied for the solvent extraction of cobalt(II). Method adapted involved solvent extraction of cobalt(II) using hexaacetato calix(6)arene in toluene A superamolecular compound, hexaacetato calix(6)arene in low concentration 0.0001M in toluene quantitatively extracts microgram concentration of cobalt at pH 7.5. It can be eluted from the organic phase with 2 M nitric acid as and determined spectrophotometrically at 500 nm.



"Fig. 2"

II. MATERIAL AND METHODS

Two-phase solvent extraction of metals with calixarene derivatives has been reported by several groups. This paper deals with the metalophilic properties of calix(6)arene derivatives specifically for cobalt. Shinkai⁴ et al. noticed that calix(5)arene and calix(6)arene had an ideal architecture because of the introduction of ligand groups into each benzene unit of these calixarenes exactly provided the required pseudoplanar penta- and hexacoordinate structures.

Hexaaceto calix(6)arene was prepared by dissolving para tertiary butylphenyl in potassium hydroxide and 36 % formaldehyde solution and heating at 110-120°C for 1hour 45 min. yellow coloured precursor thus formed was mixed with xylene and again heated at 210-220°C for 2 hours 55 min in nitrogen using dean Stark Collector[2]. The mixture was cooled and filtered and mixed with chloroform to obtain suspension after this it was shaken well with 1M hydrochloric acid in separatory funnel. After shaking for 15 min the organic layer was separated from aqueous layer and dried over anhydrous sodium sulphate. Precipitation was carried out by the addition of methanol.

The cobalt(II) complex was formed by treating a neutral cobalt(II) salt solution with a neutral solution of the sodium salt of 1-nitroso-2-naphthol. The red-brown, water insoluble solid contained two organic molecules per cobalt atom[3]. During the present study Cobalt(II) was extracted at varying pH. At the pH range of 5-8 extraction of cobalt was studied and it was found that at pH range 7-8 the extraction was 92 %. Stock cobalt solution was prepared by dissolving cobalt chloride in distill water. A UV-visible spectrophotometer and digital pH meter was used in the experiment. It was seen that among various diluents toluene shows better separation[5]. Cobalt(II) complex, was stripped by using 0.05 M nitric acid. The above method is simple, selective, fast and useful.

III. RESULTS AND DISCUSSION

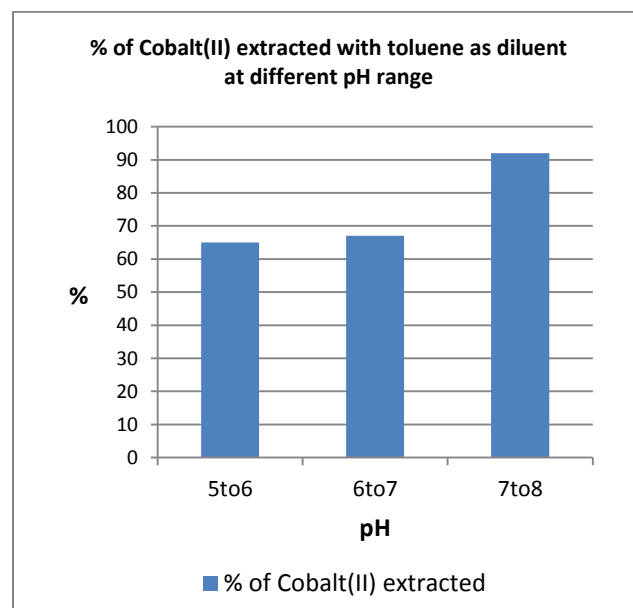
Calix(6)arenes represent a family of macrocyclic molecules with a wide range of possible applications in chemical, analytical, and engineering materials fields. Low toxicity of

these compounds makes them useful in applications of green chemistry and eco-friendly industrial processes[6]. Present paper demonstrated that calix(6)arenes is very good in different fields including separation processes e.g.,

solvent extraction may be useful for extracting cobalt from solutions. Application of such extracting agents enables the use of 10 to 100 times lower ligand concentrations necessary to reach an assumed extraction yield than with the other existing extractants. Extensive literature studies showed that extraction ability and selectivity of calixarene derivatives is closely related to their structural arrangement.

pH	Diluents	% of Cobalt(II) extracted
5-6	toluene	65
6-7	toluene	67
7-8	toluene	92

Table 2. Percentage extraction (%) of Cobalt(II)



"Fig. 3"

REFERENCES

- [1]. Burusco Goñi KK, Cardiel J. "Methodological approach to conformational search". Universitat Autònoma de Barcelona,; 2009.
- [2]. Mathew VJ, Khopkar SM. "Hexaacetato calix (6) arene as the novel extractant for palladium". Talanta, Vol.44, Issue.10, pp.1699-703, 1997.
- [3]. A. J. Petrella and C. L. Raston, "Calixarenes as platforms for the construction of multimetallic complexes," Journal of Organometallic Chemistry, vol. 689, no. 24, pp. 4125-4136, 2004.
- [4]. C. D. Gutsche, "Calixarenes: An Introduction," Royal Society of Chemistry Cambridge UK 2008.

- [5]. S. Shinkai, H. Koreishi, K. Ueda, T. Arimura, and O. Manabe, "*Molecular design of calixarene-based uranophiles which exhibit remarkably high stability and selectivity.*" Journal of the American Chemical Society, Vol. 109, No. 21, pp. 6371–6376, Oct 2012.
- [6]. Kanamathareddy, S.; Gutsche, C. D. "*Approaches to poly metallated calixarene derivatives,*" J. Am. Chem. Soc. 1993, 115,6572