# Diethyldithiocarbamate Doped Graphene Quantum Dots Based Metal Complex Nanoparticles by Resonance Light Scattering for Green Detection of Lead(II) (A Review)

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### ABSTRACT

Resonance light scattering (RLS) probe for sensitive determination of heavy metals such as lead (Pb) based on diethyldithiocarbamate (DDTC) doped graphene quantum dots (GQDs) is subject to approach on green procedure of metal complex nanoparticles. Regarding Pb(II), the RLS intensity of GQDs-DDTC increases linearly by an enhancement mechanism. Interactions between target analyte and the ligand doped nanoparticles lead to an aggregation of the complex particles to form bigger volume of the metal complex. Thus, it can be used as the RLS probe for such determination of lead ion at ultra-trace level, certainly under the optimum conditions; such experimental parameters including each of DDTC and GQDs concentration, pH of solution, ionic strength, masking agent, and interfering ions are investigated in details. The methods can then be implied for selective determination of some relevant toxic heavy metals in water, foods or environment samples.

Keywords: Diethyldithiocarbamate, Resonance light scattering, Graphene quantum dots, Heavy metals, Nanoparticles.

## INTRODUCTION

At present, concerning an environmental pollution via systematic monitoring of heavy metals in water and/or even if other foodstuffs involved is very important to protect human public health. Among the different environment from various industrial activities, lead ion is one of the main toxic metals that can be taken, and it can then be accumulated in the human body throughout the

lifetime[1-5]. Generally, water quality has become an issue of vital importance. There are several parameters and components to be determined in order to assess the water quality. Heavy metals thus fit in the group as being toxic for human health. Even small amounts of lead that enter the environment can accumulate in elevated concentrations resulting in adverse effects. Simply in drinking water, maximum allowable limit of total Pb of 50 $\mu$g L$^{-1}$ is considered safe by the World Health Organization (WHO), whereas less than 15 $\mu$g L$^{-1}$ is adopted by the United States Environmental Protection Agency (USEPA)[6]. Therefore, it is important to develop simple, fast and convenient method for lead detection, especially at ultra-trace level.

Several methods have commonly been applied for lead detection including flame atomic absorption[7-8], inductively coupled plasma - atomic emission spectrometry[9], spectrophotometry[10] and electrothermal atomic absorption spectrometry[11-12]. However, these methods have a greater cost, higher sample volume requirement and instrumentation complexity limiting. Therefore, this review focuses on determination of lead in such low matrix sample like drinking water detected by resonance light scattering technique. The resonance light scattering (RLS) is a special light scattering occurring when the wavelength of the scattered light is located at or close to the molecular absorption band. The spectral characteristics and scattering intensity are strongly influenced by molecular size, shape, conformation, and interfacial properties[13], which further provide favorable new information for the study of the interaction of biological macromolecules and the molecular recognition[14]. In recent years, RLS technique has been widely applied in quantitative analysis of drugs[15-16], surfactants[17], proteins[18-19], heavy metal ions[20-22] and nanoparticles[23]. It is characterized by high sensitivity, rapidness, simplicity and convenience which can be conducted by spectrofluorophotometer.

Regarding to this method, lead forms complex with diethyldithiocarbamate. This ligand is a chelating agent with strong tendency to form stable heavy metal complexes[24]. Furthermore, use of graphene quantum dots as an auxiliary ligand has attracted much attention in

Pb-DDTC interactions due to it has excellent properties, such as high photostability against photobleaching and blinking, biocompatibility, and low toxicity[25]. GQDs possess large surface area, large diameter, fine surface grafting using the $\Pi-\Pi$ conjugated network or surface groups and other special physical properties[26-27]. It has the carboxyl and hydroxyl groups at their edge enable them to display excellent water solubility and suitability for successive functionalization with various organic, inorganic, polymeric or biological species[28-29]. For these reasons, GQDs have attracted significant attention worldwide. Presently, it has been applied as sensor for detection of various target analytes in numerous kinds of samples such as ferric ion in water sample[30], free chlorine in drink water[31], aminothiols in blood serum[32], glucose in human serum[33] and lead in tap water and drinking water samples[34], indicating that GQDs can be used as a promising sensor for the detection of not only metal ions but also non-metal ions and/or organic compounds in various applications.

In this review, we present a green approach to find a novel RLS probe for the determination of Pb based on DDTC doped GQDs. In the presence of Pb, the resonance light scattering intensity of GQDs-DDTC would increase linearly by enhancement mechanism. The interaction between the analyte and ligand leads to the aggregation of the particles to form bigger volume of the complexes. This is aimed to use the RLS probe for determination of lead. In addition, the optimum conditions are certainly investigated in details including each of DDTC and GQDs concentration, pH of the solution, ionic strength, masking agent, and interfering ions. Then the developed method could be applied for lead determination in such water samples, for instant.

**Lead determination**

Lead is one of the few metals that can be found in metallic form in nature. It is highly corrosion resistant, but tarnishes underexposure to air. Our environment contains countless sources of lead, which is a natural component of soil, water, vegetation, animal life and air[35,36]. Also, it is ubiquitous in food, water and air and is now known to be a poison absorbed into the bloodstream, from where, especially in the case of inorganic lead, it is

distributed to soft tissue, bones and teeth. Organic forms of lead are fat soluble and therefore have a particular tendency to concentrate in brain. Thus, it is necessary to develop method for determination of lead. Several methods for lead detection in water sample are widely based on optimum extraction prior to an instrumentation analysis.

Homogeneous liquid-liquid extraction in association with flame atomic absorption spectrometry for determination of Pb(II) was reported[37]. This method was optimized with lead-diethyldithiocarbamate (Pb-DDTC) complex which was extracted with a perfluorooctanoate anion under acidic condition. The method increases sensitivity and selectivity. Graphite furnace atomic absorption spectrometry was used for determination of Pb(II) in water samples by solidification of floating organic drop microextraction with 1-undecanol containing dithizone[38]. The dithizone complexes were formed in the sample solution under proper conditions, and cooled in an ice bath. The presented method enables a minimum use of toxic organic solvent, simplicity, low cost, rejection of matrix constituent, sensitivity and rapid analysis time.

Xylenol orange functionalized CdSe/CdS quantum dots as fluorescence probe for determination of Pb(II) was reported[39]. The CdSe/Cd SQDs were modified with mercaptoacetic acid and capped with chitosan. The mechanism of fluorescence quenching is possibly due to the disruption of the electron transfer from the coordination between Pb(II) and xylenol orange on the QD surface. This method is excellent sensitivity and selectivity for the analysis of Pb(II) in real samples. Jien *et al.,*[40] demonstrated the dual channels sensor for determination of Pb(II) using bromide-capped gold nanoparticles as probe. The results show that in the present of bromide the fluorescence emission and colorimetric change of the gold nanoparticles-based system can be used to detect Pb(II), which absorb violet right (300 nm) and give red emission (600 nm). Therefore, the method can be utilized to detect Pb(II) by naked eye. Woilloud *et al.,*[41] presented flow injection (FI) coupled with inductively coupled plasma atomic emission spectrometry (ICP-AES) prior to determination of lead. The FI-ICP-AES was

conducted using a microcolumn filled with Amberlite XAD-16 resin, which Pb(II) was retained as Pb-(5-Br-PADAP) complex. Table 1 compiles chemical materials used to detect Pb(II) prior to determination by various relevant instrumental techniques.

**Chemical sensors**

Chemical sensors are popular method for converting most an optical sensor. The chemical sensors are a device that transforms chemical information into an analytically useful signal. The chemical information, mentioned above, may originate from a chemical reaction of the analyte or from a physical property of the system investigated. They can have applications in different areas such as medicine, home safety, environmental pollution and many others. Chemical sensors usually contain two basic components connected in series: a chemical recognition system and a physicochemical transducer. In the majority of chemical sensors, the receptor interacts with analyte molecules. As a result, its physical properties are changed in such a way that the appending transducer can gain an electrical signal[51].

Chemical sensors are classified according to the operating principle of the transducer. This may be further subdivided according to the type of optical properties[51] including absorbance, reflectance, luminescence, fluorescence, refractive index, optothermal effect, and light scattering. Tayyebeh *et al.,*[14] presented a resonance light scattering (RLS) for determination of Pb(II) and Cd(II) in water using cysteamine functionalized CdS hollow nanospheres as a probe. The mechanism of resonance light scattering enhancement is due to the aggregation between the nanoparticle of complexes. The RLS intensity increases with the size of particle after adding the analytes. The results showed that under the optimized conditions, the metals ions with detection limits at ultra-trace level were obtained. Haiyan *et al.,*[52] reported that dithiocarbamate (DTC) capped silver nanoparticles (AgNPs) was used as sensor probe for determination of Pb(II) and cysteine based on the RLS. They gave strong affinity to Pb(II) with N-C-$S_2$ group in DTC molecule. The RLS enhancement with the aggregation of DTC-AgNPs was obtained. The proposed method shows

excellent selectivity, wide linear range and high sensitivity for Pb(II). Moreover, the strong binding of cysteine towards Pb(II) by formation of Pb(II)-S bonds, Pb(II) was removed from the surface leading to re-dispersion of DTC-AgNPs and decreased the RLS signal.

Fang *et al.,*[53] reported a resonance light scattering method for determination of perfluorooctanoic acid (PFOA) based on crystal violet (CV) interaction. The PFOA-CV complexes interact with acid medium. The results showed enhancement of resonance light scattering intensity. They consisted of the concentration of PFOA in 0.1 to 25.0 $\mu$ mol $L^{-1}$, with the detector limit of 11.0 nmol $L^{-1}$. The proposed method is new possibility for sensitivity detecting PFOA in water. Ruiling *et al.,*[54] presented RLS technique for determination of phenol in water. The method implied graphene quantum dot (GQDs) in the presence of horseradish peroxidase (HRP) and $H_2O_2$. Strong RLS spectral band at 310 nm was obtained. It gives widely linear range and very low detection limit of $2.2 \times 10^{-8}$ M. Da-Qian *et al.,*[55] reported the determination of glucose by RLS technique based on the conversion of gold nanoclusters into gold nanoparticle. The proposed method has good performance with easy operation,

low cost, high selectivity, sensitivity and stability. Table 2 shows the determination of some target analytes by using RLS technique.

**Functional ligands**

Ligands are referred to atom, ion, or molecule that binds to a central metal to produce a coordination complex. They are viewed as Lewis bases, although rare cases are known involving Lewis acidic ligands. There are also classified in many ways: charge, size, coordinating atom, and number of electrons donated to the metal. For example, DDTC has sulfur electron donor electron with several metal[66-67].

New fluorescence sensor for determination of Cu(II) based on the quenching effect of the DDTC - functionalized quantum dots (QDs) was reported[68]. The DDTC was bonded the quantum dots via the surface ligand exchange to from DDTC-QDs conjugates following the capping of 2-mercaptoacetic acid on core shall CdSe/Cds QDs. The mechanism of fluorescence is quenching after coordination with Cu(II). The proposed method shows excellent selectivity and successful for determination Cu(II) in real sample. Zhiliang *et al.,*[69] presented luminescence properties of metal(II)-DDTC chelate complex nanoparticles. The

**Table 1: Determination of lead ion using various chemical materials with their relevant instrumental techniques**

| Technique | Chemical material | Linear range | Detection limit | Ref. |
|---|---|---|---|---|
| GFAAS | Diethyldithiocarbamate | 0.1 - 30 ng $mL^{-1}$ | 27 ng $mL^{-1}$ | [42] |
| Paper-based analytical device | *n.d.* | 10 - 100 $\mu$g $L^{-1}$ | 10 $\mu$g $L^{-1}$ | [43] |
| FAAS | Diethyldithiocarbamate | 0.041 - 0.026 mg $L^{-1}$ | 5.7 $\mu$g $L^{-1}$ | [24] |
| UV-visible spectrometry | Gold nanoparticles/ dithiocarbamate derivative 4'-aminobenzo-18-crown-6 | 0.1 - 75 $\mu$M | 50 nM | [44] |
| Microfluidic device/ Photoluminescent | Graphene oxide quantum dots | 1 - 1000 nM | 0.64 nM | [45] |
| Screen-printed/ Electrochemical | Gold electrode/ Gold nanoparticles | 20 - 200 ppb | 2.2 ppb | [46] |
| Fluorescence | CdSe/ZnS quantum dot/graphene oxide | 0.1 - 10 nM | 90 pM | [47] |
| ETAAS | $MnO_2$ coated carbon nanotubes | *n.d.* | 4.4 ng $L^{-1}$ | [48] |
| ICP-OES | 8-Hydroxyquinoline | 0.04 - 0.28 $\mu$g $mL^{-1}$ | 0.34 $\mu$g $L^{-1}$ | [49] |
| HPLC | Fe@Ag@dimercaptobenzene | 0.05 - 200 $\mu$g $L^{-1}$ | 0.034 - 0.016 $\mu$g $L^{-1}$ | [50] |

results show the strongest luminescence of those metal complexes.

Determination of Pb(II) via gold nanoparticles (AuNPs) coated with a dithiocarbamate-modified 4'-aminobenzo-18-crown-6 (DTC-CE) based on colorimetric sensor was also reported[44]. The mechanism is Pb(II) ion-induced DTC-CE-AuNPs aggregation was achieved by the sandwich complex formation between multiple DTC of 4'-aminobenzo-18-crown-6-capped Au NPs and Pb (II). The results showed that a red-shift of DTC-CE-AuNPs, and good linear range of 1.0 to 75 µM, with a detection limit of 50 nm. The proposed method is a simple determination of Pb(II) with good selectivity and sensitivity. Saksit *et al.,*[24] presented solid-phase extraction with DDTC as chelating agent for preconcentration and determination of Cu(II), Fe and Pb(II) by flame atomic absorption spectrometry. The PF of 15-fold was obtained with the appropriate ratio of sample volume. The method is simple and inexpensive for routine analysis of

**Table 2: Determination of some target analytes by using resonance light scattering technique**

| Chemical material | Target analyte | Linear range | Detection limit | Ref. |
|---|---|---|---|---|
| 4-[(5-chloro-2-pyridy)azo] -1,3-diaminobenzene complex | Nucleic acid | 0 - 400 ng mL$^{-1}$ | 1.4 ng mL$^{-1}$ | [56] |
| Ammonium salts | Bismuth | 0.022 - 5.01 µg mL$^{-1}$ | 0.0036 µg mL$^{-1}$ | [57] |
| Silver nanoparticles | Sudan dyes I | 0.2 - 2.4 µM | 3.2 nM | [58] |
| Mercaptoacetic acid-capped CdSe quantumdots | Papain | $1.0\times10^{-8}$ - $6.0\times10^{-7}$ M | $5.1\times10^{-9}$ M | [59] |
| ssDNA templated silver nanoclusters | Cu(II) | $5.0\times10^{-9}$ - $7.5\times10^{-7}$ M | 2 nM | [60] |
| Magnetic nanoparticles/ thymine-containing aptamers | Hg(II) | 1 - 80 pM | 500 fM | [61] |
| Tetraphenylboronate anion/ sodium dodecyl benzene sulfonate | K(I) | 0.2 - 2.0 µg mL$^{-1}$ | 20 ng mL$^{-1}$ | [62] |
| Molecularly imprinted polymer coated magnetite nanoparticles | Mefenamic acid | 100 - 2000 ngL$^{-1}$ | 50 ng L$^{-1}$ | [63] |
| Resorcinol yellow and OP | Proteins | 0.02 - 4.0 µg mL$^{-1}$ | 10.4 ng mL$^{-1}$ | [64] |
| Silica nanoparticles | Methylene blue | 0.01 - 3.0 µg mL$^{-1}$ | 4.36 ng mL$^{-1}$ | [65] |

the trace metals in these samples. Qingxiang *et al.,*[70] reported a simultaneous determination of Ni(II), Co(II) and Hg(II)by solid phase extraction using multiwalled carbon nanotubes (MWNTs) as adsorbent after chelating with DDTC prior to separation by HPLC. The results showed that MWNTs had high adsorption potential for the simultaneous enrichment of Ni(II), Co(II) and Hg(II) with DDTC as the chelating reagent. The method gives high enrichment factors, low detection limits and lower cost.

**Graphene quantum dots (GQDs)**

Graphene quantum dots as a new kind of quantum dots have emerged and ignited tremendous research interest. Due to the pronounced quantum confinement and edge effects, they have numerous novel chemical/physical properties[71-72]. They also show low cytotoxicity, excellent solubility, chemical inertia, stable photoluminescence, better surface grafting, making them promising in optoelectronic devices, sensors, bio-imaging[73-76]. Many preparing methods, both top-down and bottom-up, have been developed. However, most top-down methods, including re-oxidation[28], electrochemistry[77], hydrothermal graphene oxide[78], and chemical oxidation fiber[79] usually suffer from some disadvantages. Those bottom-up strategies, namely carbonizing some special organic precursors by thermal treatment[80-81], usually allow precise control over their morphology and size distribution of the products. This will focus on a simple bottom-up method to selectively prepare GQDs by citric acid pyrolysis.

Yongqiang *et al.,*[82] reported easy bottom-up method for preparation of photoluminescent GQDs and graphene oxide by tuning the carbonization degree of citric acid. The results showed that after moderate pyrolysis, citric acid can be partially carbonized to from nanosheet. The GQDs show a strong and excitation-independent photoluminescent activity. Xu *et al.,*[83] reported a facile bottom-up method for the synthesis of highly fluorescent GQDs by a one-step pyrolysis of a natural amino acid, L-glutamic acid. The results showed that GQDs showed strong blue, green and red luminescence under irradiation with ultra-violet, blue and green light, respectively. Li *et al.,*[84] reported an easy synthesis of GQDs based on carbonization of polycyclic aromatic hydrocarbon with strong acid and followed by hydrothermal reduction. The GQDs are in the range of 5-10 nm and 0.5-2.5 nm by TEM and AFM respectively.

Fabricated humidity sensor using GQDs was introduced[85]. Synthesis of GQDs by carbonization of citric acid was obtained. The results showed sensitivity to the variation of environment humidity of the synthesized GQDs, the response time was about 10 s and the recovery time was found to be somewhat longer. Synthesis and characterization of blue-color-emitting GQDs by pyrolyzing L-glutamic acid was presented[86]. BGQDs exhibits high quantum yield. Their good biocompatibility is promising for biological and medical applications. Table 3 shows some applications of GQDs for the determination of heavy metals and other analytes.

**Table 3: Some applications of GQDs for the determination of heavy metals and other analytes**

| Technique | Type of GQDs | Precursor | Target analyte | Detection limit | Ref. |
|---|---|---|---|---|---|
| Photoluminescence | GQDs | Citric acid | Hg(II) | 3.36 μM | [87] |
| | GQDs | Glucose | ClO$^-$ | 30 μM | [88] |
| | GQDs | Glycine | Fe(III) | 0.1 μM | [89] |
| | N-GQDs | Citric acid/ammonia | Cr(VI) | 40 nM | [90] |
| Fluorescence | GQDs | Citric acid | $H_2O_2$ | 0.1 μM | [91] |
| | GQDs | Citric acid | Hydroquinone | 0.084 μM | [92] |
| Electrochemical | GQDs | Citric acid | $S_2O_8^{2-}$ | 0.1 μM | [93] |
| Immunosensor | GQDs | Citric acid | CA199 tumor | 0.96 mU mL$^{-1}$ | [94] |

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