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Tris-(8-hydroxyquinoline) Aluminum for Detecting Nitro-Group Containing Explosives by Photoluminescence

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ABSTRACT

In the article, the tris(8-hydroxyquinoline) aluminum (AlQ_3) were examined for applications to detect nitro-group containing explosives compounds such as 2,4,6-trinitrotoluene (TNT), 2,3-dinitrotoluene (2,3-DNT), 3,4-dinitrotoluene (3,4-DNT), 2,6-dinitrotoluene (2,6-DNT) and *p*-nitrotoluene (4-NT), can obviously cause the fluorescence quenching of AlQ_3. Under the optimum condition, a linear response was observed over the stock solution concentration range of 10^{-4} to 10^{-3}M for various nitro-group containing explosives in methanol. The fluorescence quenching follow the Stern-Volmer equations of F_0/F versus [Q] show a good linear relation in 10^{-3}M order of magnitudes and the detection limits approach 10^{-5} to 10^{-6} M.

Keywords: AlQ_3, nitro-group containing explosives, fluorescence, Stern-Volmer equation

三-（8-羥基喹啉）鋁經由光致激發光檢測含硝基火炸藥

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摘　　要

文章中,三-（8-羥基喹啉）鋁(AlQ_3)能應用在檢測含有硝基的火炸藥例如三硝基甲苯(TNT),二硝基甲苯(2,3-DNT, 3,4-DNT, 2,6-DNT)和對位-硝基甲苯(4-NT),能夠明顯的造成三-（8-羥基喹啉）鋁(AlQ_3)的螢光消光作用.於最佳的狀態下,溶於甲醇溶液中,含有硝基所有不同的火炸藥濃度範圍由10^{-4}到10^{-3}莫耳濃度(M),螢光消光過程經由Stern-Volmer方程式中F^0/F與[Q]之間所產生的最佳線性關係的濃度等級在10^{-3}莫耳濃度(M),檢測極限值在10^{-5}到10^{-6}莫耳濃度(M).

關鍵詞：三-(8-羥基喹啉)鋁，含硝基火炸藥，螢光，Stern-Volmer方程式

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I. INTRODUCTION

Explosive vapor detection is important in military bases or post-blast residue environmental pollution determinations [1-3] and the detection of hidden explosives as well as unrecovered land mines. All countries face the problem of cleaning up at least some of their contaminated sites. The development of new devices capable of rapidly and cost-efficiently detecting explosives has become an urgent worldwide necessity. Many techniques which have been studied for detecting explosive compounds include various physical methods such as gas chromatography coupled with a mass spectrometer, nuclear quadrupole resonance, X-ray diffraction imaging, laser-based as well as electron capture detection have been used for this purpose [4] and electrochemical sensing [4,5]. These techniques are highly selective but some of them are expensive.

Explosive detection techniques can be broadly classified into two mains categories: bulk detection and trace detection. Trace detection analysis as tiny amounts of explosive vapor or particles in chemiluminescence technology [11]. Over the past several years, chemical sensors based on absorption, fluorescence and conductivity transduction mechanisms have attracted much attention in the rapid detection of explosive [6–10], therefore, the fluorescence- based sensor schemes are very promising because they can be easily incorporated into inexpensive and portable microelectronic devices. Vapor detection examined the vapor emanating from a liquid or a solid explosive and some explosives have a low vapor pressure, there detection technology would be very sensitive [4,12]. Although ion mobility spectrometry (IMS) has evolved to be a fast and mature technology for the analysis by vapor sampling and swipes [13], the limit of detection (LOD) for trinitrotoluene (TNT) using toluene as the sensitizing solvent was only $187 \mathrm{mgL}^{-1}$ [14].

Tris-(8-hydroxyquinolate) aluminum (AlQ_3), a kind of organometallic complex with electro- optical properties, used to be a prototype molecular emitter has became known from 23 years ago [15] because AlQ_3 has good electron transport and light emission under relative stability and easy wide-area depositions. The recent interest about AlQ_3 clearly derives from its application as a constituent of Organic Light Emission Diodes (OLEDs) [16-20]. The characteristics concur to make it a desirable material for detecting nitro-group containing explosives by photoluminescence.

In this study, we report single-step solid-state synthesis that fluorescence material of AlQ_3 and detecting nitro-group containing explosives by photoluminescence. It is could be efficiently quenched by various nitro-group containing, which might provide a new pathway to detection of the nitro-group containing explosives and their relative compounds.

II. EXPERIMENTAL

2.1 Synthesis of AlQ_3

The complex AlQ_3 was prepared review the literature method is to combine 8-hydroxy -quinoline (8-HQ) anion with Al^{3+} in its aqueous solution [21]. AlQ_3 is precipitated under the optimum condition by adjusting pH value of solution. The aqueous solution of aluminum nitrate or aluminum sulfate was chosen as Al^{3+} ion. Sodium hydroxide (NaOH) was used to adjust the pH value of solution. The degree of chemical reaction depends on the acidity and alkalinity of reactive materials.

AlQ_3 was synthesized in laboratory. The preparation method is single-step solid-state synthesis. The concrete processes of synthesis are shown as follows: a well-ground mixture of $AlCl_3$ (0.52g, 3.75mmol) and 8-HQ (1.51g, 0.01mole) was put into a silica crucible and heated at 190℃ for 40min under a atmosphere. The crucible does not lid was heated in the electrical furnace to get the melt and continuous agitation in the mixing process. AlQ_3 was performed by heated these mixtures directly to 190℃ with the heating rate higher than 10℃/min. The melt was cooled slowly in an evacuated chamber. The mixed crystals were crushed to fine powder. After extraction the resultant black solid with $CHCl_3$ (50ml), the extract was filtrated to afford a yellow green filtrate. We get the solid bright yellow green amorphous AlQ_3 complex by rotary vacuum evaporator.

2.2 Reagents

Aluminum chloride anhydride ($AlCl_3$), 8-hydroxyquinoline (99%) and 2.4.6-trinitro-toluene (TNT), 2.3-dinitrotoluene (DNT), 2.6-dinitrotoluene (DNT), 3.4-dinitrotoluene (DNT) and *p*-nitrotoluene (4-NT) procured from Aldrich reagent. TNT, DNT and 4-NT stock solutions of nitro-group containing explosive were prepared in methanol at 4.41×10^{-4}M to 3.52×10^{-3}M, 5.5×10^{-4} to 4.4×10^{-3}M and 7.35×10^{-4}M to 6.62×10^{-3}M, respectively. Stock solutions of AlQ_3 were prepared in methanol at 4.35×10^{-6}M. AlQ_3 doped with various nitro-group containing explosive mass proportions was dissolved in methanol. AlQ_3 was dissolved in chloroform at a concentration of $7mgml^{-1}$ ($1.52x10^{-2}$M) and the solutions were spin-coated onto quartz glass substrates to a roughness of surface 200nm.

2.3 Instrumentation

Scanning electron micrographs (SEM) images, Roughness of surface were obtained on Hitachi 4800S (15.0kV) and Surface profiler measuring system Dektak 3030. UV-vis absorption spectrum, fluorescence and lifetime measured using a Cary 50, Cary Eclipse luminescence spectrometer (Varian, USA). The excitation and emission slit widths were set at 10 nm, respectively. The samples were excited at 375 nm and the fluorescence signal was monitored at 515 nm. All the experiments were carried out at room temperature.

III. RESULTS AND DISCUSSION

3.1 SEM and Optical Images of AlQ_3

Scanning electron micrographs (SEM) of the AlQ_3 shown in Fig. 1.(a). In these micrographs, AlQ_3 can be seen as an amorphous crystal region. The SEM images also reveal that the AlQ_3, with a diameter average ranging of 200nm, are well distributed on the surface single tubes. AlQ_3 has excellent photoluminescence property. It emits bright fluorescence under ultraviolet radiation. The fluorescence spectra of AlQ_3 with excited wave 375 nm are shown in Fig. 1.(b), the UV-vis absorption and photoluminescence (PL) spectrum of AlQ_3 in methanol solution. The characteristic fluorescence peak of AlQ_3 is observed at 515 nm (green range), which agrees well with previous reports [21-24].

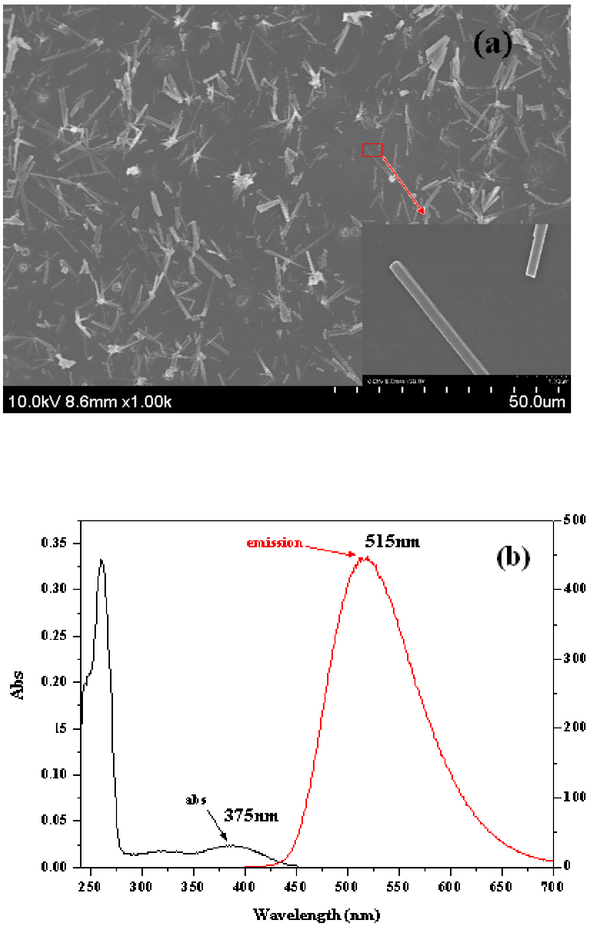

Fig. 1. (a) SEM of AlQ_3, (b) UV-vis absorption and fluorescence spectrum.

3.2. The Role of Molecular Structure

AlQ_3 is a chelate-complex and exhibits strong fluorescence. The photoluminescence and absorption spectra of AlQ_3 are influenced by the 8-hydroxyquinoline. It has been suggested that the electronic $\pi\rightarrow\pi^*$ transitions in AlQ_3 are localized on the quinolate ligands. The phenoxide π-system in AlQ_3 is the highest energy filled state, and hence should be the most readily oxidized, making it the likely site of the trapped hold. Conversely, the pyridine system is the lowest energy vacant state, making it the likely site of reduction and thus the trapped electron [25].

3.3 Measurements of Fluorescence Spectra

The fluorescence quenching technique was a helpful method for the study of the mechanism of molecular interaction, energy transfer or charge transfer. AlQ_3 was a typical electron donor and nitro-group containing explosive was a typical electron acceptor. To study the interaction of various nitro-group containing explosive with AlQ_3 in methanol solution, the stock AlQ_3 solution was added to different concentration of various nitro-group containing explosive solution. Their relative compounds inclusive of TNT, 2.3-DNT, 2.6-DNT, 3.4-DNT and 4-NT were found to quench the AlQ_3 fluorescence. Fluorescence quenching is often achieved through an electron-transfer donor-acceptor mechanism [26], as depicted in Fig. 2. Electron-transfer fluorescence quenching.

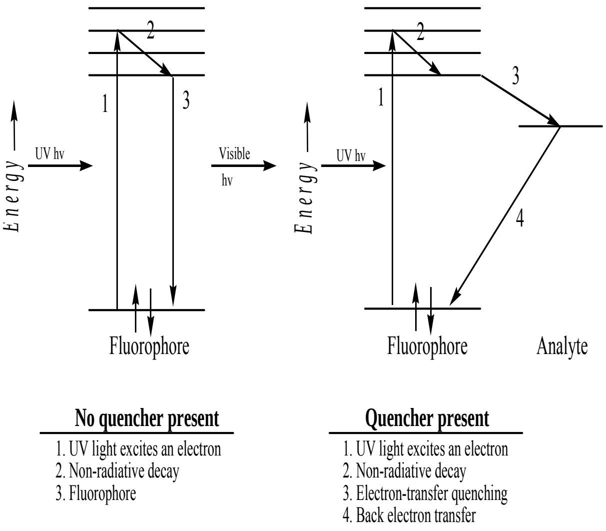

Fig. 2. Electron-transfer fluorescence quenching.

Fig. 3 shows fluorescence quenched percent of AlQ_3 with increasing concentration after of various nitro-group containing explosive in methanol. Overall, the AlQ_3 show relatively strong fluorescent quenching upon exposure to all the analysis.

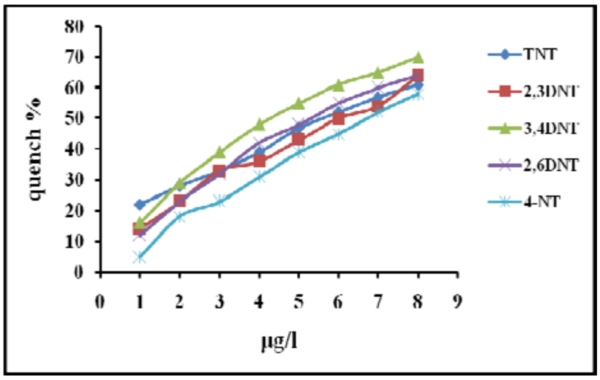

Fig. 3. Fluorescence quenched percent of AlQ_3.

As seen in Fig. 4-8, nitro-group containing explosive analysis quenches AlQ_3 emission intensity. Fluorescence quenching can occur by dynamic quenching (collision quenching), static quenching, or by combined dynamic and static quenching mechanisms. Fluorescence quenching data can be described by the Stern-Volmer equation and the data are interpreted by plotting F_0/F versus [Q] graph, where F_0 and F are the fluorescence intensities in the absence and presence of the quencher, respectively, and [Q] is the quencher concentration. A Stern-Volmer plot can be linear or non-linear (upward or downward curving) depending on the quenching mechanism [27,28].

In static process, the classical Stern–Volmer equation is $F_0/F = 1 + K_{SV}[Q]$, where F_0 and F are the fluorescence intensities in the absence and presence of the quencher concentration [Q] and K_{SV} is a linear Stern–Volmer quenching constant [28]. A plot of F_0/F versus [Q] also yields a linear graph as in dynamic quenching process. $K_{SV} = [FQ]/[F][Q]$, where [FQ] is the concentration of the complex, [F] the concentration of free fluorophore and [Q] is the concentration of quencher.

Fig. 4-8, the fluorescence quenching spectra of AlQ_3 by nitro-group containing explosive analysis in various concentration investigated here in range from 10^{-4} to approaching 10^{-3}M and the plots of F_0/F (F_0 and F being the fluorescence intensity of AlQ_3 at 515nm in the absence and presence of nitro-group containing explosive compounds, respectively) versus quencher concentration of analysis [Q to approaching 10^{-3}M] yields a linear relationship between two parameters with the value of the y-intercept at unity as shown in depicted insets. So we focused the quenching concentration on 10^{-3}M order of magnitude. The quenching plots showed as linear property in this concentration scale for all analysis compounds. The slope for 3,4-DNT is higher than that for other compounds.

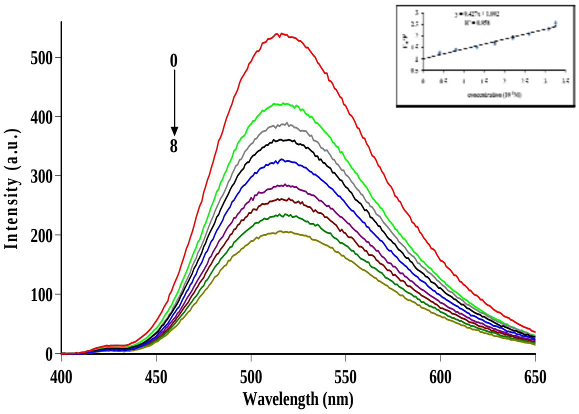

Fig.4. Fluorescence quenching spectra of AlQ_3 with increasing concentration of TNT.(From no. 0 to 8: 0.0, 4.41×10^{-4}, 8.81×10^{-4}, 1.32×10^{-3}, 1.76×10^{-3}, 2.2×10^{-3}, 2.6×10^{-3}, 3.08×10^{-3}, 3.52×10^{-3}M, respectively.)

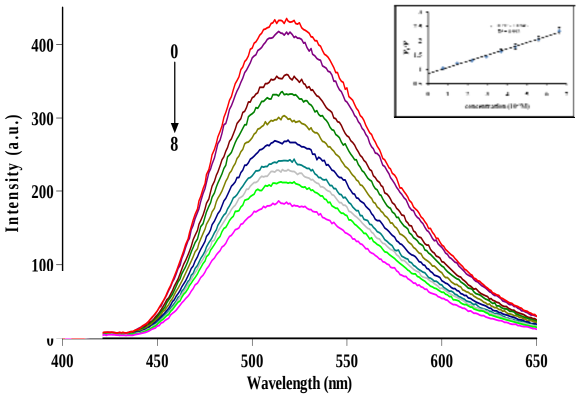

Fig.5. Fluorescence quenching spectra of AlQ_3 with increasing concentration of 4-NT. (From no. 0 to 8: 0.0, 7.35×10^{-4}, 1.47×10^{-3}, 2.21×10^{-3}, 2.94×10^{-3}, 3.68×10^{-3}, 4.41×10^{-3}, 5.58×10^{-3}, 6.62×10^{-3}M, respectively.)

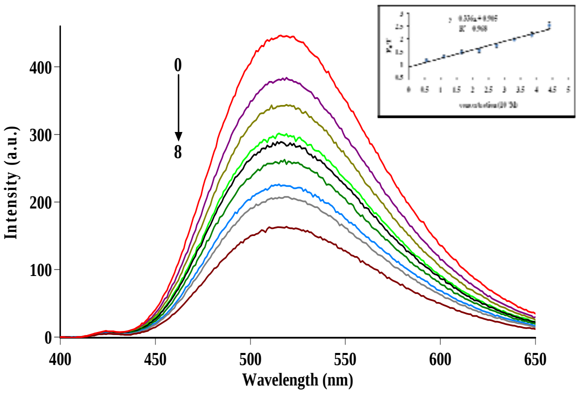

Fig.6. Fluorescence quenching spectra of AlQ_3 with increasing concentration of 2,3-DNT. (From no. 0 to 8: 0.0, 5.5×10^{-4}, 1.1×10^{-3}, 1.65×10^{-3}, 2.2×10^{-3}, 2.75×10^{-3}, 3.3×10^{-3}, 3.85×10^{-3}, 4.4×10^{-3}M, respectively.)

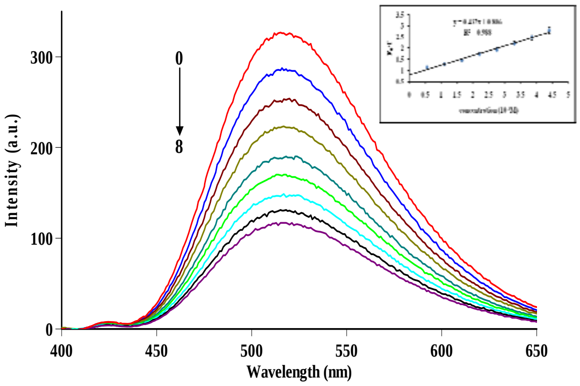

Fig.7. Fluorescence quenching spectra of AlQ_3 with increasing concentration of 2,6-DNT. (From no. 0 to 8: 0.0, 5.5×10^{-4}, 1.1×10^{-3}, 1.65×10^{-3}, 2.2×10^{-3}, 2.75×10^{-3}, 3.3×10^{-3}, 3.85×10^{-3}, 4.4×10^{-3}M, respectively.)

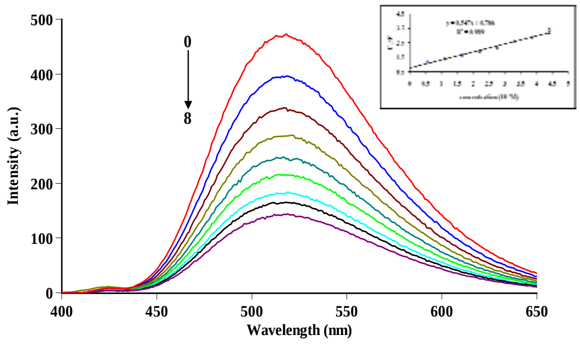

Fig.8. Fluorescence quenching spectra of AlQ_3 with increasing concentration of 3,4-DNT. (From no. 0 to 8: 0.0, 5.5×10^{-4}, 1.1×10^{-3}, 1.65×10^{-3}, 2.2×10^{-3}, 2.75×10^{-3}, 3.3×10^{-3}, 3.85×10^{-3}, 4.4×10^{-3}M, respectively.)

As both dynamic and static quenching

processes give linear Stern–Volmer plot, the AlQ_3 emission intensity quenched by nitro-group containing explosive is either by dynamic or static quenching process. Dynamic quenching process affects the excited states of the fluorophore and the ground state. For this reason absorbance spectra are not expected to change during dynamic quenching. The static quenching where a complex is formed in the ground state and change in the absorbance spectrum of the fluorophore are expected [28] and observed (as seen in Fig. 9). Fig. 9 illustrates one of the difference spectra showing a peak (absorbance increase) at 254nm when the AlQ_3 doping in the 3.4-DNT.

As a result, the linear Stern–Volmer plot indicates static quenching; the quenching coefficient K_{SV} is the association constant to be TNT: 498.88 M^{-1}, 2.3-DNT: 393.18 M^{-1}, 2.6-DNT: 406.82 M^{-1}, 3.4-DNT: 524.48 M^{-1} and 4-NT: 220.41 M^{-1}. 3.4-DNT showed the strongest quenching efficiency. The detection limits are listed in Table 1.

Table 1, it can be seen that the fluorescence of AlQ_3 were quenched by the nitro-group containing explosives compounds, linear dynamic range from 10^{-4} to approaching 10^{-3}M. However, in the concentration range we could achieve a good linear correlation between F_0/F and Q, as shown as in depicted Fig. 4-8 insets. The Stern-Volmer plots, F_0/F versus Q, 4-NT showed good linear relationship (R^2>0.9919). From the experimental result shows：stock solution of AlQ_3 (4.35×10^{-6}M) for detected of nitro-group containing explosives compounds. The detection limits approach 10^{-5} to 10^{-6}M (ppm for TNT).

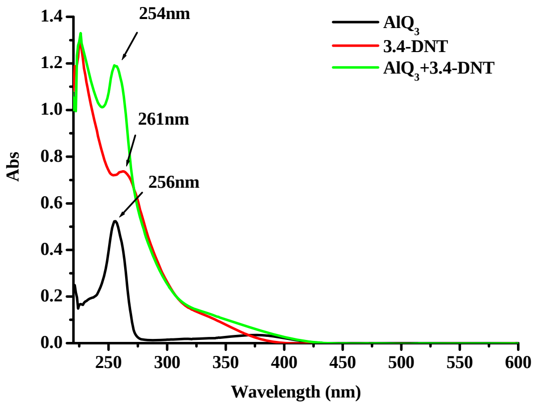

Fig. 9. The absorbance spectra of AlQ_3 and AlQ_3+ 3.4-DNT.

Table 1.The potential analytical characteristic of nitro-group containing explosives compounds.

Condition	LDR (M)	LOD (M)	R^2
TNT	4.41×10^{-4} to 3.52×10^{-3}	4.41×10^{-6}	0.9580
3.4-DNT	5.50×10^{-4} to 4.40×10^{-3}	5.50×10^{-5}	0.9890
2.3-DNT	5.50×10^{-4} to 4.40×10^{-3}	5.50×10^{-5}	0.9680
2.6-DNT	5.50×10^{-4} to 4.40×10^{-3}	5.50×10^{-5}	0.9880
4-NT	7.35×10^{-4} to 6.62×10^{-3}	7.35×10^{-5}	0.9920

LDR, Linear dynamic range; LOD, limit of detection; R^2, correlation coefficient.

3.4 The Thin Film Measurements of Fluorescence

Fig. 10 shows the fluorescent quenching of AlQ_3 film (200nm) by TNT saturated vapor pressure at temperature 40℃. The result indicates the quenched efficiency is close to 5% after 10sec.

The vapor pressure increase with enhance temperature, the highest quenching efficiency of AlQ_3 film close to 35% of exposure at temperature 80℃ after 10sec, seen in Fig. 11.

The possible reasons inferred from the experimental result are follows: nitro-group containing explosive competitive complex processes with metal Al^{3+} and ligand atoms or molecules from nitro-group containing explosives and 8-HQ. Nitro group was a strongly deactivated of various nitro-group containing explosive and can reduce the fluorescent intensities of AlQ_3 from metal Al^{3+} ions. But the exact thin film quenched mechanism remains to be discussed in the future study.

Although the method proposed here is not a specific one, generally, the detection of explosives and their relative compounds can provide very useful information for probing landmines or other explosive devices. Therefore, as a screening method it should be acceptable.

IV. CONCLUSIONS

In conclusion, the various nitro-group containing explosive compounds caused the fluorescence quenching of AlQ_3. The synthesized AlQ_3 show large fluorescence quenched effects with explosives, making this system an interesting candidate for explosives measurement. A good linear relationship was observed up to concentrations of at different concentration of various explosives. The detection limits are from 10^{-5} to 10^{-6}M level in present state. The fact that AlQ_3 showed high fluorescence quenching sensitivity towards nitro-group containing explosive compounds investigated indicated that they are potential candidate material for detection of landmines or other explosive devices.

Acknowledgements

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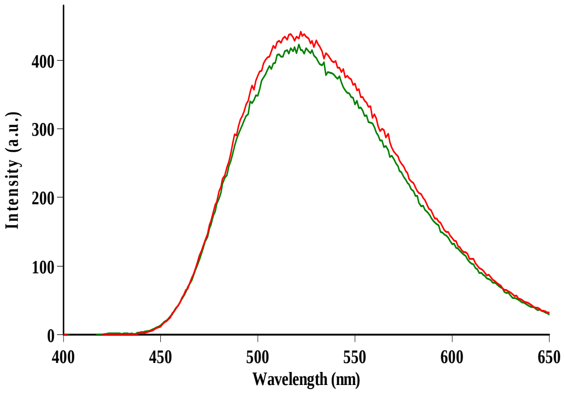

Fig. 10. The fluorescent quenched of AlQ_3 (red line) film by TNT at temperature 40℃ after 10sec (green line).

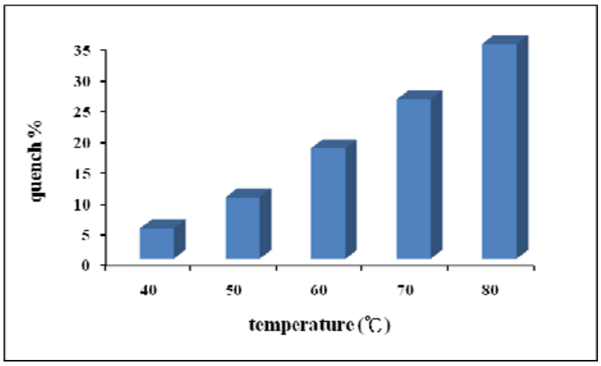

Fig. 11. The fluorescent quenched of AlQ_3 film by TNT saturated vapor pressure at different temperature.

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