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SYNTHESIS OF 8-METHOXYQUINOLINE-5-AMINO ACETIC ACID AND ITS HERBICIDAL POTENTIAL

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Abstract: -

The quinoline skeleton is often used for the design of many synthetic compounds with diverse pharmacological properties. 8-Methoxyquinoline 5-amino acetic acid was synthesized from coupling of monochloroacetic acid with 5-amino-8-methoxyquinoline. The IR showed – OH stretch absorption and amino group (-NH) which is not too prominent at 3450cm^{-1} and 3300cm^{-1}, carbonyl (C=O) absorption depicts at 1614cm^{-1}. The herbicidal activity of the synthesized compound was tested on the weeds and after 11 days of application, the weeds have dried completely indicating the efficacy of the compound.

Keywords: *synthesis, 8-Methoxyquinoline 5-acetic acid, IR and herbicidal activity.*

INTRODUCTION

Herbicides are chemicals used with the intention of killing plants (Andrew et al 2011), chemical herbicides have replaced large number of farm labourers and boosted crop production. (Cindy, 1987), the chemicals are called weed killers; some of these weed killers act by interfering with the growth of the weed (Quastel 1950). Many compounds have been synthesized and have been found effective as weed control (Owolabi and Olarinoye (2008). The major aim of this research work was to synthesize 8-methoxyquinoline-5-amino acetic acid and test the compound for its herbicidal potential.

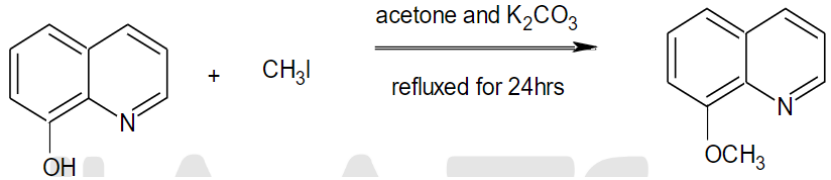
Methodology

Materials and methods:

8-Hydroxyquinoline was obtained from the chemical store of Chemistry Department, Federal University of technology, Akure, Nigeria. The sample appears as a pale light yellow crystalline compound.

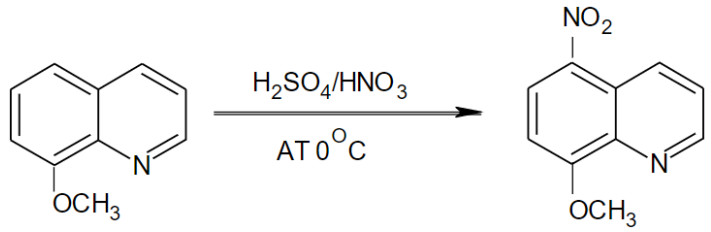
Preparation of 8-methoxyquinoline

The principle of Norman (1993) guided the synthesis of the compound. To a solution of 8hydroxyquinoline (1.05g,7.23mmol) in acetone (15mL) was added solid potassium carbonate(1.0g,7.24mmol) and methyl iodide(0.45ml,7.23mmol).The reaction mixture was reflux for 24hours.It was allowed to cool to room temperature and followed by filtration and removal of the solvent in vacuo. Flash chromatography (acetone) of the residue afforded the product (815mg,71%) as a light oil with retardation factor (0.58) using (benzene/hexane 2:1



Preparation of 5-Nitro-8-Methoxyquinoline

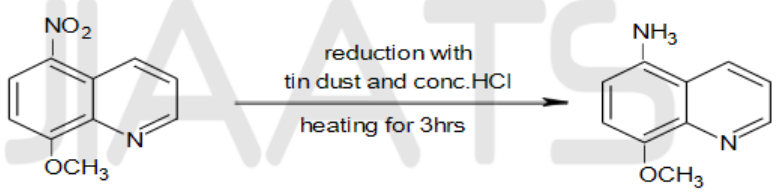
The method of Norman (1993) was adopted. 5cm^3 concentrated sulphuric acid and 4cm^{3}concentrated nitric acid were mixed with cooling. To the mixture in the cold,50mg of 8methoxquinoline was added with shaking to dissolve. The reaction reached completion about 10-15 minutes due to the strong activating methoxy group on the benzene ring. The mixture was poured into cold water and a yellow compound precipitated which was filtered in vacuum ,dried over anhydrous calcium chloride. The solids obtained were recrystallized with 50mL of 95% methanol giving a percentage yield of 77% and melts at 115^{0}C.



Preparation of 5-Amino -8-Methoxyquinoline

Reduction Process

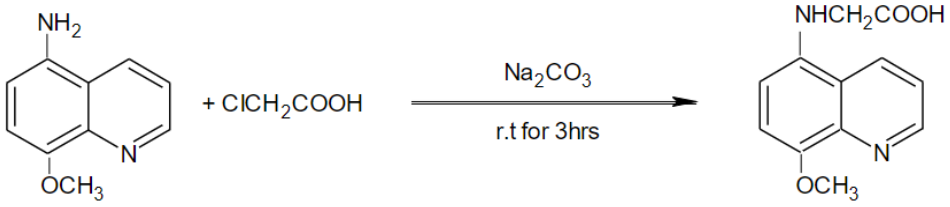
10ml concentrated hydrochloric acid was used to dissolved 0.050g of 5-nitro-8methoxyquinoline and 1g of tin dust was added with vigorous shaken. The mixture was heated on water bath for 1hr until all the nitro compound disappeared. The reaction mixture was subsided to about 30^{0}C and 40mL cold water was used to neutralize the medium. It was extracted with 40ml chloroform and a brownish solid was obtained which was further recrystallized and purified using column chromatography. The percentage yield obtained was 96%, it melt at 142^{0}C and R_f of 0.86 using benzene and chloroform in ratio 3:1 as a mobile phase.



Preparation of 8-Methoxy -5-Aminoacetic acid

Into 100cm^3 flask, 20mL of 5% sodium carbonate solution was used to dissolved 0.041g of 5amino-8-methoxyquinoline,0.025g of Monochloroacetic acid was coupled with mixture and the reaction medium was stirred for 3hrs.At the end of the reaction, the medium was neutralized with 10cm^3 concentrated hydrochloric acid and a whitish

solid precipitated. The precipitate was filtered, dried and recrystalized with 25ml of methanol. The percentage yield obtained was 60.97% and melt at 142°C.



FT/IR Analysis:
The IR of the compounds were recorded using spectroscopic techniques (Williams, 1993). These were done in University of Ibadan cenral research laboratory and University of New york,United kingdom. The model of the Infrared machine is spectrum Bruker Vortex 70 using Opus 2013 software, named as Ft-Ir system.

Herbicidal Activity:
For the herbicidal activity, 5mg of Na_2CO_3 was dissolved in 100ml of distilled water to give us Na_2CO_3 solution. 37mg of synthesized compound, 8-methoxy-5-amino acetic acid was dissolved in Na_2CO_3 solution. The solution was sprayed on the weeds using broadcast method (USDA Agricultural Research Service. 2010)

Results:
The FT/IR result is shown below.

Table 1 showing Absorption peak (CM^{-1})

FUNCTIONAL GROUP	SPECTRUM A (cm^{-1})	SPECTRUM B (cm^{-1})	SPECTRUM C(cm^{-1})	SPECTRUM D(cm^{-1})	SPECTRUM E(cm-^{1})
O-H	3447.00	----------	----------	----------	----------
C=O	……..	.....	----------	----------	1614
NO_2	……..		1338		
Aromatic Absorption	3045.71, 882.90-481.08	3049,892-649	2914, 930-620	1019, 616	2453,889-655
N-H	----------	----------	----------	3348	3450

SPECTRUM A : 8-HYDROXYQUINOLINE
SPECTRUM B : 8-METHOXYQUINOLINE
SPECTRUM C : 5-NITRO-8-METHOXYQUINOLINE
SPECTRUM D : 5-AMINO-8-METHOXYQUINOLINE
SPECTRUM E : 8-METHOXYQUINOLINE-5-AMINO ACETIC ACID

Spectra 1-5 shows the infra-red spectra of newly synthesized compound and its derivatives.

Spectrum1

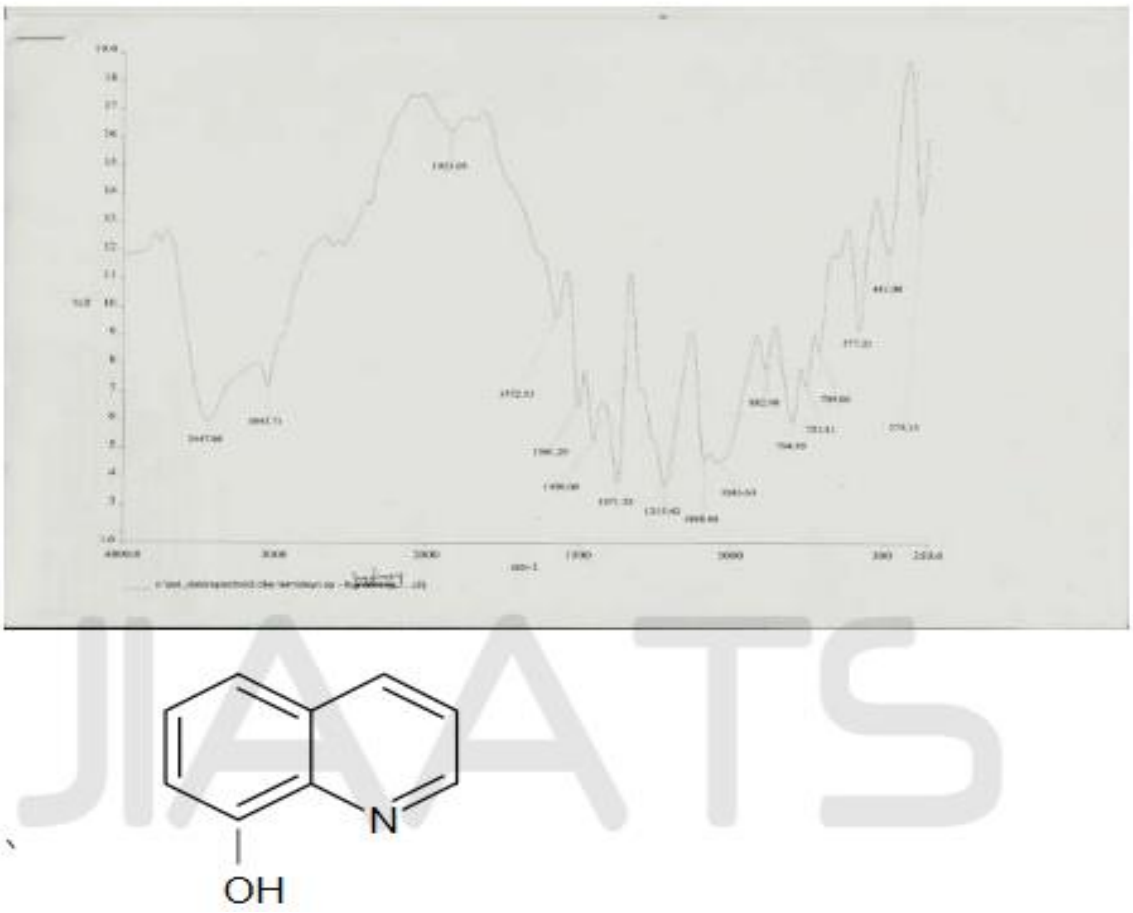


FIGURE 1: 8-HYDROXYQUINOLINE

Spectrum 2

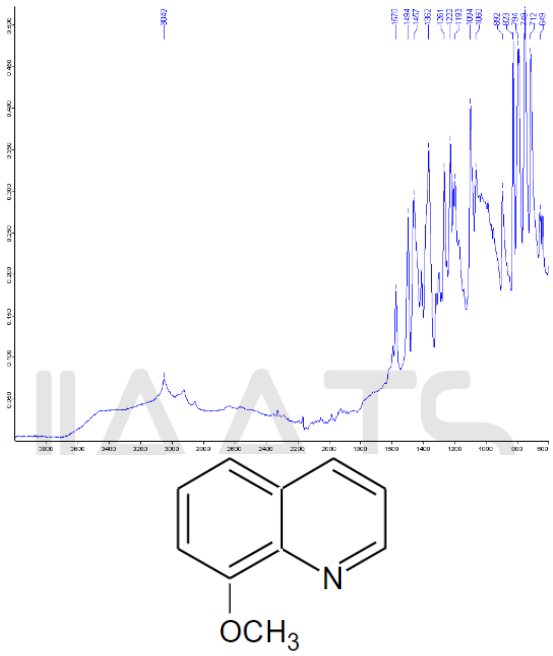
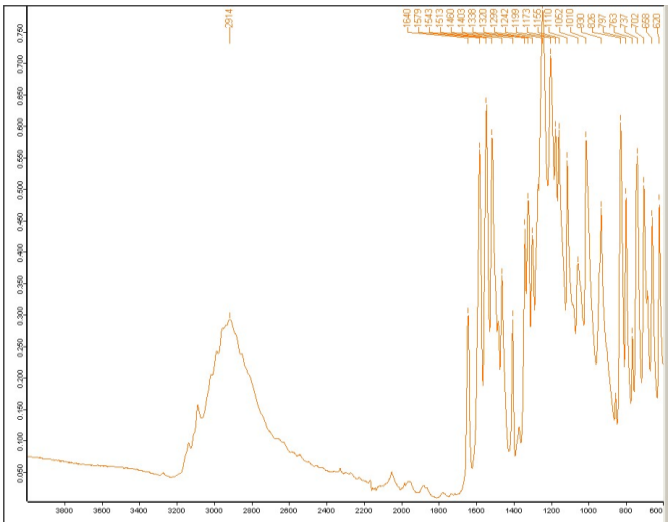


FIGURE 2:8-METHOXYQUINOLINE

Spectrum 3



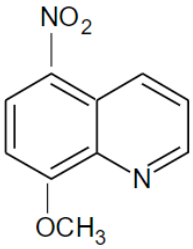
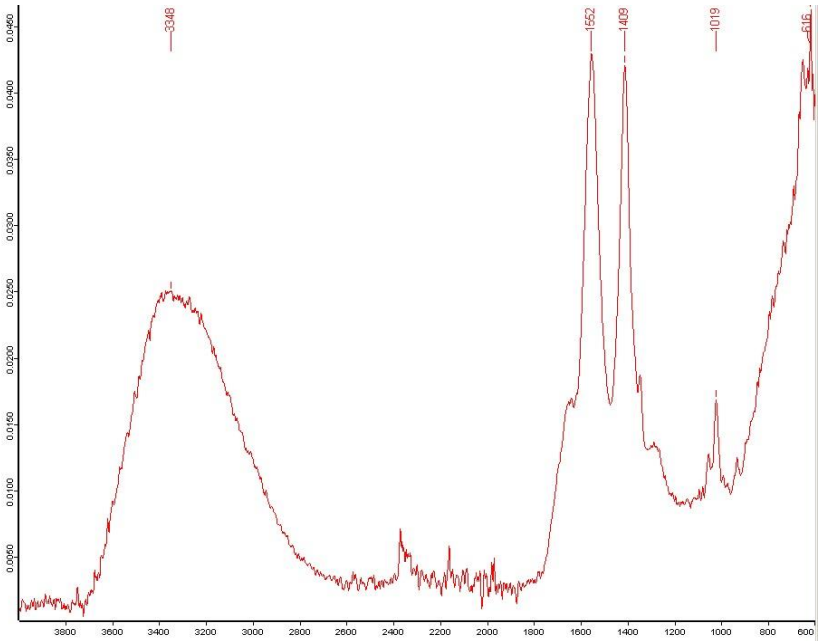


FIGURE 3: 5-NITRO-8-METHOXYQUINOLINE

Spectrum 4



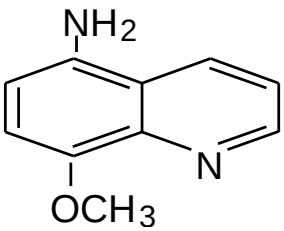
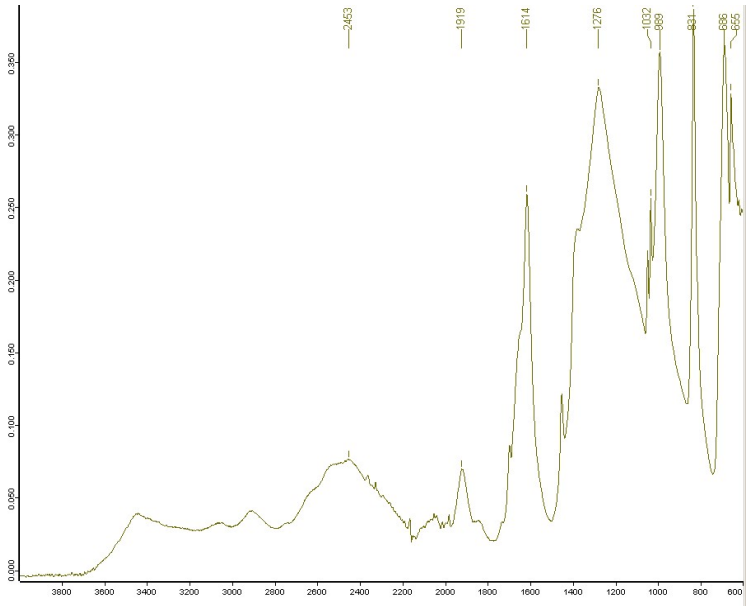


FIGURE 4: 5-AMINO-METHOXYQUINOLINE

Spectrum 5



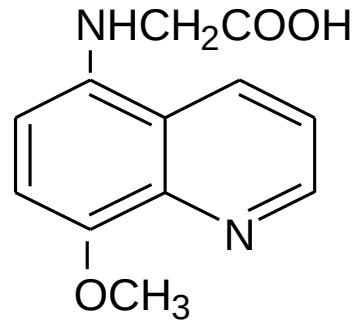


FIGURE 5: 8-METHOXY-5-AMINOACETIC ACID

Application of 8-methoxyquinoline-5-amino acetic acid on weeds;

Effect after 11days of application;

Discussion

The IR of figure 1 was run in the University of Ibadan, Nigeria while the IR of Figure 2 and 3 were run in the University of New York, London. From table 1, which showed the result of Infra red and figure1 showed the spectrum of 5-hydroxyquinoline showing the functional groups present on the ring. Absorption at 3447.60cm^{-1} depicts for –OH, a –CH stretch absorption at 3045cm^{-1}, weak overtone with associated aromatic compound was seen at 1923cm^{-1}, C=C stretch was seen at 1572cm^{-1}. In addition, from table 1 also and figure 2 showed spectrum of 8-methoxyquinoline,the product form from reaction of 8hydroxyquinoline with methylodide with peak at 3049cm^{-1} (-CH stretch), C=C stretch 1570cm^{-1}, -CH bend in and out plane help in suggesting aromatic compound at 1094-712cm^{1}. Moreover, from table 1which showed the IR and figure 3 showed spectrum of 5-nitro-8methoxyquinoline formed from the reaction of 8-methoxyquinoline with mixed acid (conc. Sulphuric and Nitric acid). Absorption at 2914cm^{-1} depicts for –CH stretch, 1460cm^{-1} for nitro group (-NO), aromatic absorption C=C stretch at 1579cm^{-1} and other bonds like –CH bend in/out of plane on the fingerprint region help to suggest presence of aromatic ring at 1110-558cm^{-1}.

Figure 4 show spectrum of 5-amino-8-methoxyquinolie from the reduction of 5-nitro-8methoxyquinoline with tin dust and concentrated hydrochloric acid. Absorption at 3348cm^{-1} amino group(-NH_2), C=C stretch at 1552cm^{-1}, -CH bend at 1019-616cm^{-1}. Figure 5 show spectrum of 8-methoxyquinoline-5-amino acetic acid from coupling of monochloroacetic acid with 5-amino-8-methoxyquinoline. –OH stretch absorption and amino group(-NH) which is not too prominent at 3450cm^{-1} and 3300cm^{-1}, carbonyl (C=O) absorption depicts at 1614cm^{-1} and -CH bend helps to show the presence of aromatic ring. An important characteristic feature which increases the likelihood of an aromatic compound present is the C-H observed in the finger print region.

In addition, the compound, 8-methoxyquinoline-5 amino acetic acid exhibited strong herbicidal activities as shown in the pictures and proved effective as agro-chemical compound.

Recommendation

The synthesized compound still needs to be evaluated for its herbicidal activities using different concentration of the solution of the compound and also carry out laboratory investigation with other herbicides. Effect on plant crops needs to be investigated also.

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