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Recent Research Assessment in Pharmacological Evaluation of Compounds Derived from Natural Precursors

Shailesh Kumar Singh[1], Dr. Laxmi Tripathi[2]

[1]*LTR Institute of Technology, Meerut, Uttar Pradesh, India*

[2]*MIT, Moradabad, Uttar Pradesh, India*

Abstract

A recent research assessment deals with the evaluation of Sesame seeds and oil synthetic compound derived from natural precursor which are used in treatment of various diseases and their safely use. All the Sesamol derivatives (1-6)A and (1-6)B were synthesized by the given schemes and reaction process was monitored by thin layer chromatography method using silica gel-G stationary phase, ethyl acetate: ethanol (2:3) as mobile phase, and detecting the spots with iodine vapours. All the synthesized derivatives were also confirmed by FTIR, 1H NMR spectroscopy and elemental analysis method. The FTIR spectrums were shown the significant peaks at 3270-3260 cm-1 (N-H stretch.), 1720- 1710 cm-1 (Cyclic C=O strech.), 1660-1640 cm-1 (Amide C=O stretch.), 1320-1310 cm-1 (C-N Strech), 695-685 cm-1 (C-S stretch) cm-1.The proton NMR spectrums were also confirmed the different Sesamol derivatives through significant signals due to change in environment of protons.

Keywords: Chromatography, Sesame, Silica.

1. Introduction

Sesame (Sesamum indicum L.) is one of the most important oilseed crops worldwide, and has been cultivated since ancient times for use as a traditional health food. (Nzikou JM) Sesame (Sesamum indicum) is a flowering plant in the genus Sesame. Numerous wild species of sesame were found in Africa, it is generally believed that sesame originated in Africa and a smaller number in India. (Nayar NM). Sesame oil has a mild odour and a pleasant taste and, as such, is a natural salad oil. It is used as a cooking oil, in shortening and margarine, as a soap fat, in pharmaceuticals and as a synergist for insecticides. (Hai Z, Budowsk P, Döker O). Sesame oil is composed of the Palmitic, Palmitoleic, Stearic, Oleic, Linoleic, Linolenic, and Eicosenoic fatty acids: Sesame oil is rich in unsaturated fatty acids where the fatty acids composition is 14% saturated 39% mono-unsaturated, and 46% poly-

unsaturated fatty acids (Toma and Tabekhia, 1979). Carbohydrates in sesame seed are composed of 3.2% glucose, 2.6% fructose and 0.2% sucrose while the remaining quantity is dietary fibers.

Figure 1 Sesame seeds and oil

Sesame oil have desirable physiological effects including antioxidant activity, blood pressure and serum lipid lowering potential as proven in experimental animals and humans (Sirato-Yasumoto et al., 2001). Sesame oil is mildly laxative, emollient and demulcent. Sesame in has been found to protect the liver from oxidative damage. The oil has been used for healing wounds for thousands of years. It is naturally antibacterial for common skin pathogens such as Staphylococcus and Streptococcus as well as common skin fungi such as athlete's foot fungus. It is anti-viral and anti-inflammatory used in the treatment of several chronic diseases including hepatitis, diabetes and migraines. Analgesic activity of the ethanolic extract of Sesamum indicum has been tested by acetic acid-induced writhing model in mice by Nahar and Rokonuzzaman (2009).

2. Material and Methodology

2.1 Synthesis of N'-(4-chlorobenzylidene)-2-(benzo[d][1,3]dioxol-5-yloxy)acetohydrazide (1A):

A mixture of Sesamol, anhydrous K2CO3, chloroethyl acetate and DMF were stirred at room temperature for 8 hours. The reaction mixture was diluted with ice cold water. A solid was separated and synthesis of compound was confirmed by TLC study. A mixture of obtained compound (0.019 M) and hydrazine hydrate (15 ml) was refluxed for 4 h. The excess of hydrazine hydrate was removed in vacuo and the residue was triturated with water, filtered off, dried and recrystallized from 70 % ethanol to give colourless crystals (Wang et al., 2010).

Synthesized compound (0.001 M) and p-chloro benzaldehyde (0.001 M) were dissolved in absolute ethanol (40 ml). Add few drops of glacial acetic acid and reflux for 6 hours. Then reaction mixture is poured in ice cold water and filtered the synthesized compound. Finally compound was washed with

water, dried at room temperature and recrystallized with ethanol. The reaction process is identified by TLC using ethyl acetate: n Hexane (2:3) as mobile phase and iodine as detecting agent (Rao et. al., 2012).

Molecular Weight: 332.06; Yield: 89.5%; M.P.: 213-215 0C. The purity of compound was checked by TLC using: Silica gel G, Solvent system; Ethyl acetate: ethanol (2:3) and detecting agent; iodine vapours. Only one sport was obtained (Rf, 0.43).

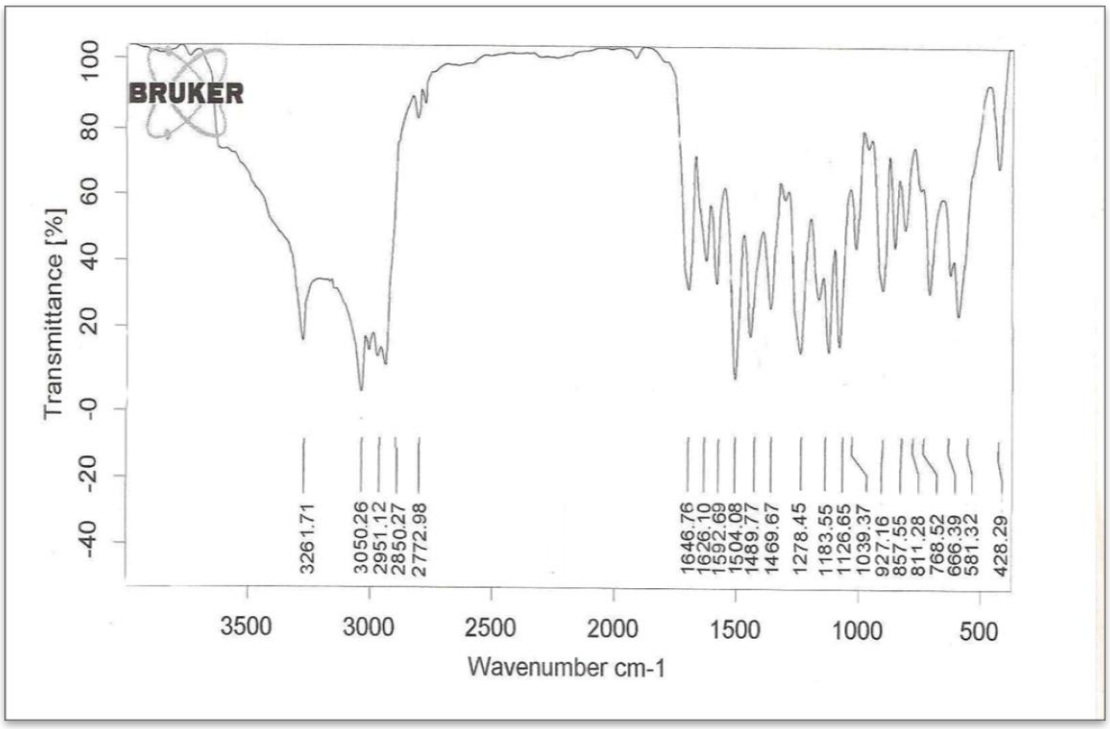

Figure 2 Transmittance v/s wave-number

Elemental analysis:

Calculated for $C_{16}H_{13}ClN_2O_4$: C, 57.75; H, 3.94; Cl, 10.0; N 8.42; O, 19.33%. Found: C, 57.78; H, 3.94; Cl, 10. 04; N 8.39; O, 19.29%

Spectral analysis:

FT-IR (vmax): 3261 (N-H), 3050 (Aromatic C-H), 2951 & 2850 (Aliphatic CH), 1646 (C=O), 1626 (Phenyl ring stretch.), 1592 (C=N), 1504 & 1489 (Phenyl C-H out of plane bending), 1469 (CH_2 bending), 1278 (C-O), 1183 (in plane aromatic bending), 1126 (C-O-C, asym), 1039 (C-O- C, sym), 927 (ethylene dioxide characteristic peak), 857 (C-Cl) cm^{-1}.

2.2 Synthesis of N'-(4-hydroxybenzylidene)-2-(benzo[d][1,3]dioxol-5-yloxy) acetohydrazide (1B):

A mixture of Sesamol, anhydrous K2CO3, chloroethyl acetate and DMF were stirred at room temperature for 8 hours. The reaction mixture was diluted with ice cold water. A solid was separated and synthesis of compound was confirmed by TLC study. A mixture of obtained compound (0.019

M) and hydrazine hydrate (15 ml) was refluxed for 4 h. The excess of hydrazine hydrate was removed in vacuum and the residue was triturated with water, filtered off, dried and recrystallized from 70 % ethanol to give colourless crystals (Wang et al., 2010).

Synthesized compound (0.001 M) and p-hyroxybenzaldehyde (0.001 M) were dissolved in absolute ethanol (40 ml). Add few drops of glacial acetic acid and reflux for 6 hours. Then reaction mixture is poured in ice cold water and filtered the synthesized compound. Finally compound was washed with water, dried at room temperature and recrystallized with ethanol. The reaction process is identified by TLC using ethyl acetate: n Hexane (2:3) as mobile phase and iodine as detecting agent (Rao et. al., 2012).

Molecular Weight: 314.09; Yield: 92.4 %; M.P.: 216-218 0C. The purity of compound was checked by TLC using: Silica gel G, Solvent system; Ethyl acetate: ethanol (2:3) and detecting agent; iodine vapours. Only one sport was obtained (Rf, 0.92).

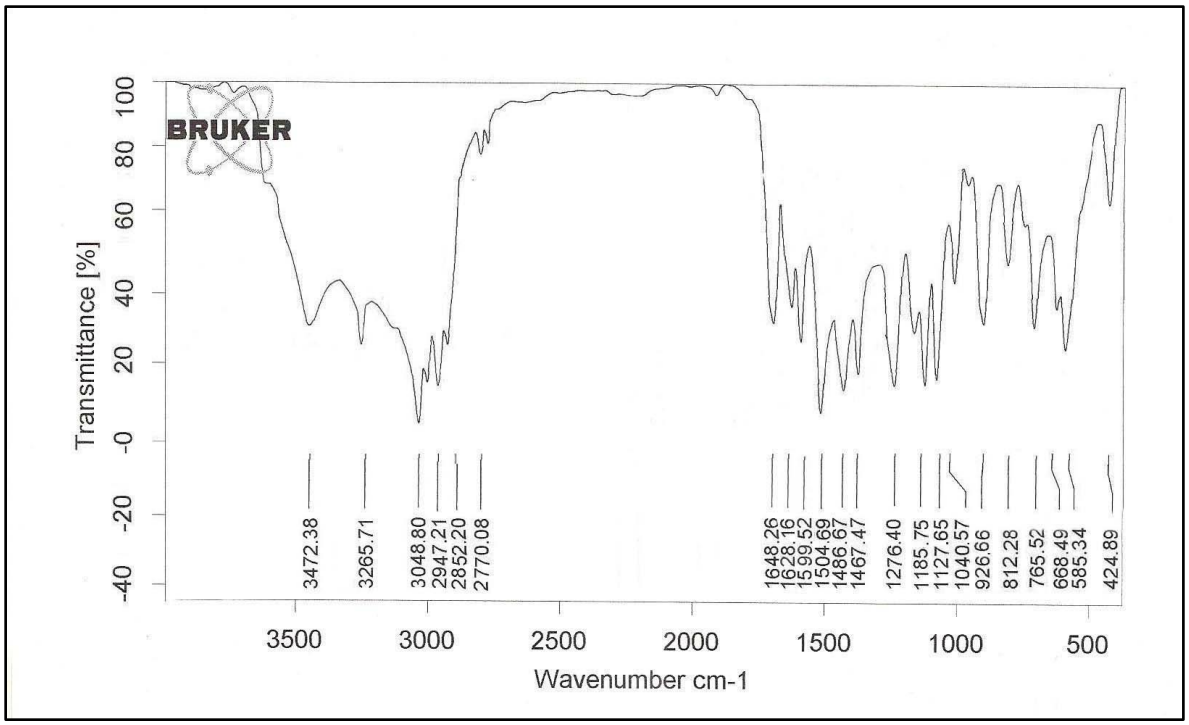

Figure 3 Transmittance v/s wave-number

Elemental analysis:

Calculated for $C_{16}H_{14}N_2O_5$: C, 61.14; H, 4.49; N, 8.91; O, 25.45%. Found: C, 61.18; H, 4.46; N, 8.88; O, 25.47%.

Spectral analysis:

FT-IR (νmax): 3472 (O-H), 3265 (N-H), 3048 (Aromatic C-H), 2947 & 2852 (Aliphatic C-H), 1648 (C=O), 1628 (Phenyl ring stretch.), 1599 (C=N), 1504 & 1486 (Phenyl C-H out of plane bending), 1467 (CH2 bending), 1276 (C-O), 1185 (in plane aromatic bending), 1127 (C-O-C, asym), 1040 (C-O-C, sym), 926 (ethylene dioxide characteristic peak) cm-1.

2.3 Synthesis of N-((4-(benzo[d][1,3]dioxol-5-yloxy)phenyl)methylene)-4-chloro benzenamine (2A):

A mixture of Sesamol, anhydrous K2CO3, 4-hydoxybenzaldehyde and acetone were refluxed at 60 0C for 6 hours. The reaction mixture was diluted with ice cold water. A solid was separated and synthesis of compound was confirmed by TLC study. The solid was washed with water, filtered off and dried at room temperature.

Synthesized compound (0.001 M) and p-chloro aniline (0.001 M) were dissolved in absolute ethanol (50 ml). Add few drops of glacial acetic acid and reflux for 4 hours. The reaction mixture is poured in ice cold water and filtered the synthesized compound. Finally compound was washed with water, dried at room temperature and recrystallized with ethanol. The reaction process is identified by TLC using ethyl acetate: n Hexane (2:3) as mobile phase and iodine as detecting agent.

Molecular Weight: 351.78; Yield: 83.7 %; M.P.: 164-166 0C. The purity of compound was checked by TLC using: Silica gel G, Solvent system; Ethyl acetate: ethanol (2:3) and detecting agent; iodine vapours. Only one sport was obtained (Rf, 0.61).

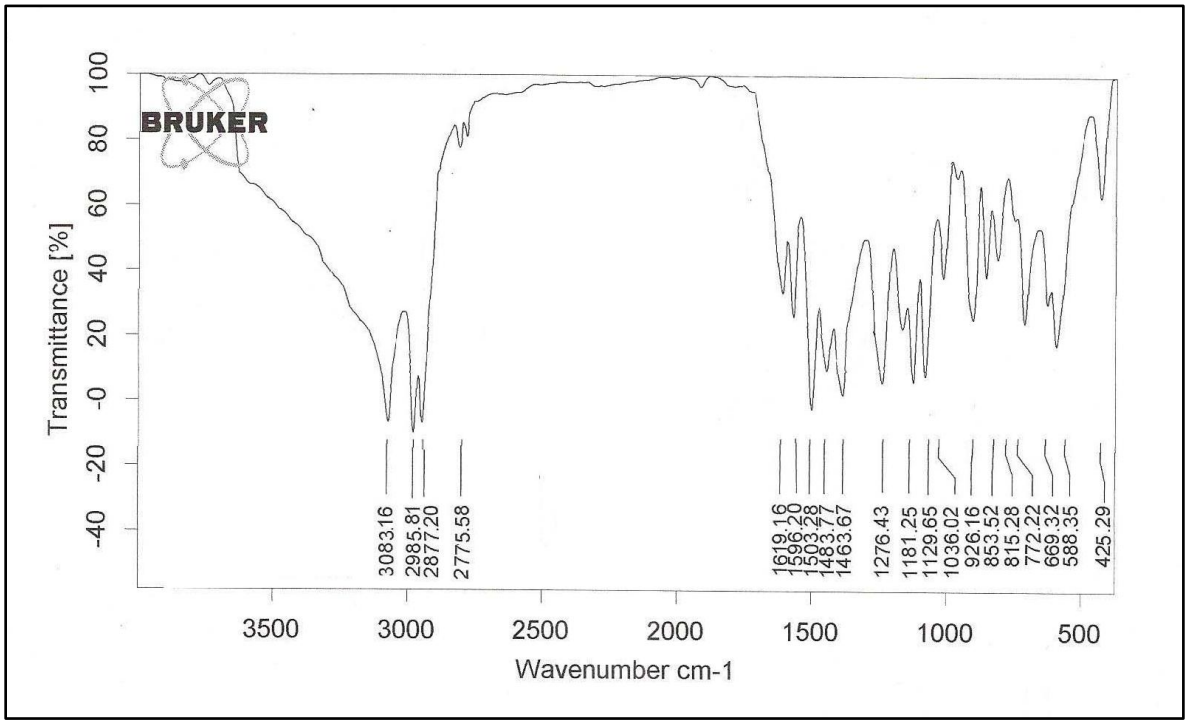

Figure 4 Transmittance v/s wave-number

Elemental analysis:

Calculated for $C_{20}H_{14}ClNO_3$: C, 68.28; H, 4.01; Cl, 10.08; N, 3.98; O, 13.64 %. Found: C, 68.28; H, 3.97; Cl, 10.04; N, 4.03; O, 13.67 %.

Spectral analysis:

FT-IR (vmax): 3083 (Aromatic C-H), 2985 & 2877 (Aliphatic CH), 1619 (Phenyl ring stretch.), 1596 (C=N), 1503 & 1483 (Phenyl C-H out of plane bending), 1463 (CH2 bending), 1276 (C-O), 1181 (in plane aromatic bending), 1129 (C-O-C, asym), 1036 (C-O-C, sym), 926 (ethylene dioxide characteristic peak), 853 (C-Cl) cm-1.

2.4 Synthesis of N-((4-(benzo[d][1,3]dioxol-5-yloxy)phenyl)methylene)-4-methyl benzenamine (2B):

A mixture of Sesamol, anhydrous K2CO3, 4-hydoxybenzaldehyde and acetone were refluxed at 60 0C for 6 hours. The reaction mixture was diluted with ice cold water. A solid was separated and synthesis of compound was confirmed by TLC study. The solid was washed with water, filtered off and dried at room temperature.

Synthesized compound (0.001 M) and p-toluidine (0.001 M) were dissolved in absolute ethanol (50 ml). Add few drops of glacial acetic acid and reflux for 4 hours. The reaction mixture is poured in ice cold water and filtered the synthesized compound. Finally compound was washed with water, dried at room temperature and recrystallized with ethanol. The reaction process is identified by TLC using ethyl acetate: n Hexane (2:3) as mobile phase and iodine as detecting agent.

Molecular Weight: 331.36; Yield: 78.8 %; M.P.: 165-167 0C. The purity of compound was checked by TLC using: Silica gel G, Solvent system; Ethyl acetate: ethanol (2:3) and detecting agent; iodine vapours. Only one sport was obtained.

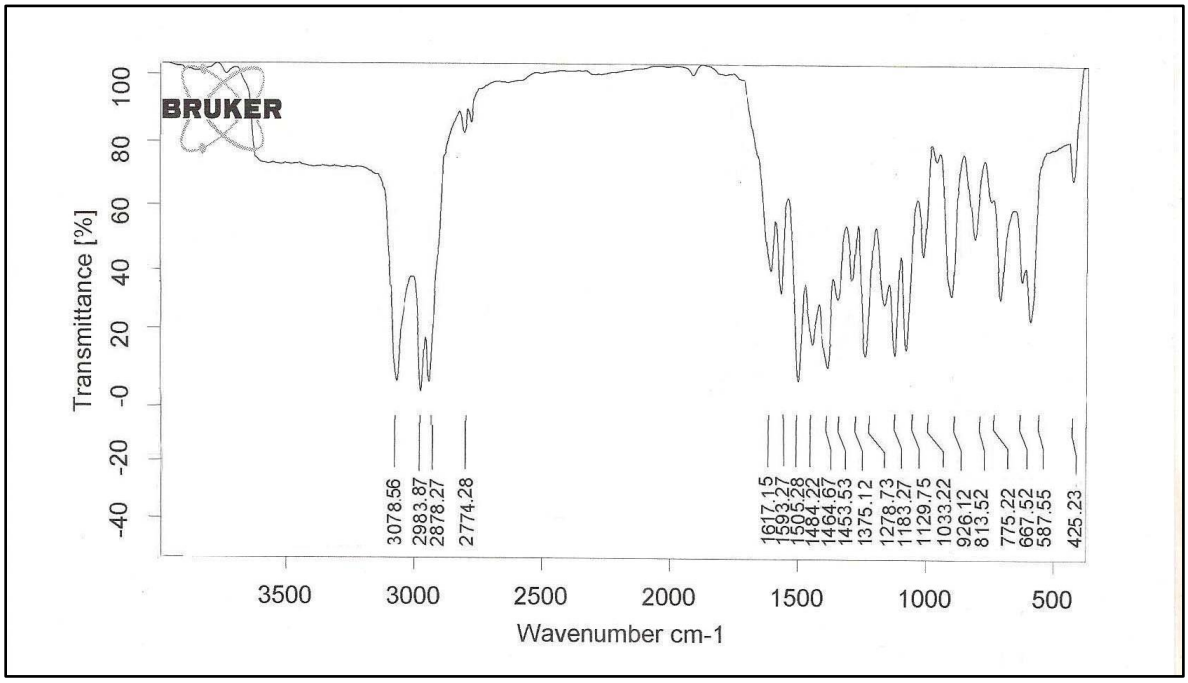

Figure 5 Transmittance v/s wave-number

Elemental analysis:

Calculated for $C_{21}H_{21}NO_3$: C, 76.12; H, 5.17; N, 4.23; O, 14.49 %. Found: C, 76.15; H, 5.13; N, 4.25; O, 14.48 %.

Spectral analysis:

FT-IR (νmax): 3078 (Aromatic C-H), 2983 & 2878 (Aliphatic CH), 1617 (Phenyl ring stretch.), 1593 (C=N), 1505 & 1484 (Phenyl C-H out of plane bending), 1464 (CH2 bending),
1453 (CH2 Bending), 1375 (CH3 Bending), 1278 (C-O), 1183 (in plane aromatic bending), 1129 (C-O-C, asym), 1033 (C-O-C, sym), 926 (ethylene dioxide characteristic peak) cm-1.

2.5 Synthesis of 1-((4-(benzo[d][1,3]dioxol-5-yloxy)phenyl)methylene)-2-propyl hydrazine (3A):
A mixture of Sesamol, anhydrous K2CO3, 4-hydoxybenzaldehyde and acetone were refluxed at 60 0C for 6 hours. The reaction mixture was diluted with ice cold water. A solid was separated and synthesis of compound was confirmed by TLC study. The solid was washed with water, filtered off and dried at room temperature.
Synthesized compound (0.001 M) and propyl hydrazide (0.001 M) were dissolved in absolute ethanol (50 ml). Add few drops of glacial acetic acid and reflux for 4 hours. The reaction mixture is poured in ice cold water and filtered the synthesized compound. Finally compound was washed with water, dried at room temperature and recrystallized with ethanol. The reaction process is identified by TLC using ethyl acetate: n Hexane (2:3) as mobile phase and iodine as detecting agent.
Molecular Weight: 298.13; Yield: 88.6 %; M.P.: 182-184 0C. The purity of compound was checked by TLC using: Silica gel G, Solvent system; Ethyl acetate: Ethanol (2:3) and detecting agent; iodine vapours. Only one sport was obtained.

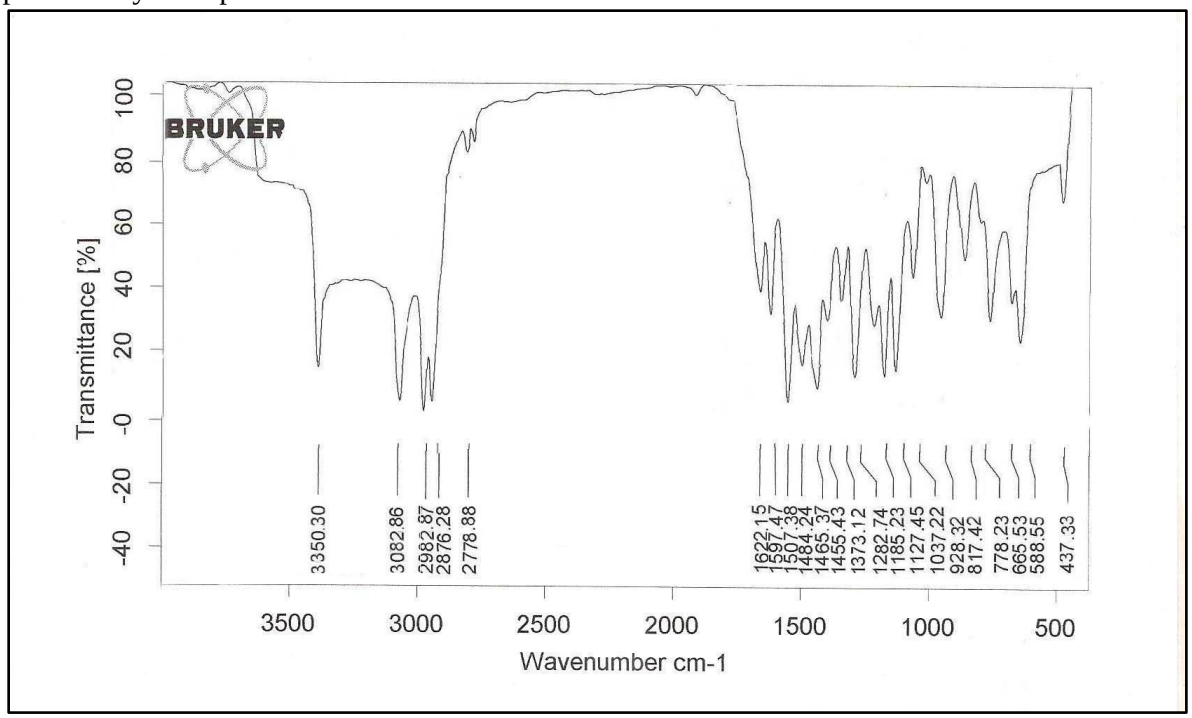

Figure 6 Transmittance v/s wave-number

Elemental analysis:
Calculated for C17H18N2O3: C, 68.44; H, 6.08; N, 9.39; O, 16.09 %. Found: C, 68.47; H, 6.02; N, 9.41; O, 16.08 %.

Spectral analysis:
FT-IR (νmax): 3350 (N-H), 3082 (Aromatic C-H), 2982 & 2876 (Aliphatic CH), 1622 (Phenyl ring stretch.), 1597 (C=N), 1507 & 1484 (Phenyl C-H out of plane bending), 1465(CH2 bending), 1455 (CH3, Asym Bending), 1373 (CH3, Sym Bending), 1282 (C-O), 1185 (in plane aromatic bending), 1127 (C-O-C, asym), 1037 (C-O-C, sym), 928 (ethylene dioxide characteristic peak) cm-1.

3. Results

The present study deals with the preparation of Sesamol derivatives N'-(4chlorobenzylidene)-2-(benzo[d][1,3]dioxol-5-yloxy)acetohydrazide-(1A), N'-(4-hydroxybenzylidene)-2 (benzo[d][1,3]dioxol-5-yloxy) acetohydrazide-(1B), N-((4-(benzo[d][1,3]dioxol-5yloxy)phenyl)methylene)-4-chloro benzenamine-(2A), N-((4-(benzo[d][1,3]dioxol-5-yloxy)phenyl)methylene)-4-methyl benzenamine-(2B), 1-((4-(benzo[d][1,3]dioxol-5-yloxy)phenyl)methylene)-2-propyl hydrazine-(3A) are prepared by reflection and extraction method. Characterization was done by element analysis and FTIR. There yields was found respectively (89.5), (92.4), (83.7), (78.8), (88.6).

4. CONCLUSION

The sesam oil derivatives are prepared was successfully from sesame oil by reflection and extraction method and found different yield successfully.

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