

Synthesis and Antimicrobial Property of 2-(2-nitrovinyl) Furan

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This work aimed at synthesis and antimicrobial evaluation of the potency of 2-(2-Nitrovinyl) Furan. The condensation of furfural with nitromethane was conducted in sodium tertiary butoxide. The product's characterization carried out with ^1H and ^{13}C NMR spectrometry and thermal analysis. The 2-(2-Nitrovinyl) Furan was tested with conventional antibiotics (Ridomil plus, Benomyl, Streptomycin, Tetracycline and Amphotericin) against pathogenic bacteria (*Escherichia coli*, *Pseudomonas aeruginosa*, *Staphylococcus aureus* and *Salmonella typhimurium*) and molds (*Fusarium solani* and *Cercospora cucurbitarum*) and yeast (*Candida albicans*) by agar well diffusion method. Characterization showed that the reaction product is 2-(2-Nitrovinyl) Furan and the product is crystalline yellow. According to the thermal analysis, the product's melting point was between 68°C and 70°C. The synthesized 2-(2-Nitrovinyl) Furan prevented the growth of the *S. typhimurium*, *C. cucurbitarum*, *F. solani* and *C. albicans* at 100%, *P. aeruginosa* and *Staph. aureus* at 96%, *E. coli* at 80% inhibition. In contrast, commercial antibiotics produced zones of growth inhibition in the range of 14% to 100%. Therefore, the synthesized 2-(2-Nitrovinyl) Furan appeared to be more powerful antimicrobial than the conventional antibiotics (Ridomil plus, Benomyl, Streptomycin, Tetracycline and Amphotericin).

Key words: 2-(2-Nitrovinyl) Furan, Synthesis, Chemical Structure, Antimicrobial efficacy.

Furfural is an intermediate chemical substance used in synthesizing a range of specialized chemical products, such as furfural alcohol, resin, adhesive, flavouring and as a precursor for many special chemicals^{1,2}.

The chemistry of furfural is that of an aromatic aldehyde, with other reactions due to the dienic character of the furan ring³. Furfural condensed with nitromethane in a weak basic

medium sodium tertiary butoxide to form 2-(2-Nitrovinyl) Furan. The presence of NO_2^- has been postulated to account for the strong antimicrobial activity of 2-(2-Nitrovinyl) Furan⁴. Historically, it was aromatic nitro compounds that were prominent in organic synthesis. In fact they have been extensively used as precursors of aromatic amines and their derivatives; and their great importance in industrial and laboratory applications has remained⁵. The most important progress in the chemistry of nitro compounds is the improvement of their preparations. In recent years, the importance of aliphatic nitro compounds has greatly increased due to the discovery of new selective transformations⁶. This work was proposed to synthesize furfural derivative and study its antimicrobial property.

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MATERIAL AND METHODS

Preparation of 2-(2-Nitrovinyl) Furan

Nitromethane and furfural were condensed in a mild basic medium to form 2-(2-Nitrovinyl) Furan⁷.

Chemical characterization of the 2-(2-Nitrovinyl) Furan

Purity of the synthesized Furan crystal was determined by thin layer chromatography method. The melting point was determined using capillary tube method.

Proton (¹H) and carbon-13 (¹³C) NMR spectrometer (Varian Germini 200 NMR with 3000.3Hz revolutions) was used in the elucidation of the structures⁸⁻¹⁰.

Determination of antimicrobial activity of the 2-(2-Nitrovinyl) Furan

The bacteria and fungi used for this experiment are *Escherichia coli*, *Pseudomonas aeruginosa*, *Staphylococcus aureus*, *Salmonella typhimurium*, *Fusarium solani*, *Cercospora cucurbitarium* and the yeast *Candida albicans*. All the bacteria and fungi were cultured aerobically at 37°C for 24 hours in peptone water. Antimicrobial activity of the synthesized compound was conducted on the microbes by agar well diffusion method of Norrel and Messely¹¹. Two millilitres of the synthesized compound was aseptically mixed with 15ml of sterile molten potato dextrose agar (PDA) that have cooled to 45°C before pour plating. It was allowed to solidify at ambient temperature. The fungi were inoculated at the centre of the plates with 4mm cork borer. Methanol, Benomyl and Ridomil plus at 0.25g/ml respectively were used

as standard antifungal agents. Another control agar media lacking any of the compounds and antibiotics were set up. All inoculated agar media were incubated at 37°C (bacteria) and 27°C (fungi) for 24-144 hours. Zones of mycelia and bacterial growth were measured in millimetres at 24 hours interval and calculated in percentage.

RESULTS AND DISCUSSION

Chemical Characteristics of the Prepared 2-(2-Nitrovinyl) Furan

The R_f was 0.88 and the melting point was between 68 to 70°C. The building units of the synthesized 2-(2-Nitrovinyl) Furan are shown in proton and carbon NMR spectra (Figures 1 and 2). The molecular formulae of 2-(2-Nitrovinyl) Furan is C₆H₅NO₃ and the molecular weight obtained is 139 g. The furan ring appeared between δ 7.4 and δ 7.8 while the 2 CH showed up at δ 6.5 and δ 6.9 respectively (Figures 1, 2 and 3). The complete structure of the 2-(2-Nitrovinyl) Furan is presented in figure 3. The quaternary carbon numbered 2 appeared at δ 146.8, the 3 CH numbered 3, 4 and 5 appeared at δ 113.6, δ 120.3 and δ 147.1 respectively (Figures 1, 2 and 3). The CH that attached to furan ring at carbon 2 appeared at δ 135.0 while that CH that attached to the nitro (NO₂) group appeared at δ 125.7. This result confirmed that 2-(2-Nitrovinyl) Furan was actually synthesised by condensing furfural with nitromethane in a basic medium (sodium *tert* butoxide)⁸.

Antimicrobial activity of 2-(2-nitrovinyl) furan

Table 1 showed the zones of inhibition created by the synthesized compound and the

Table 1. Percentage Growth Inhibitory Effect of the synthesized 2-(2-Nitrovinyl) Furan relative to standard antibiotics

Microbial Strains	2-(2-Nitrovinyl) Furan	Strep.	Tet.	Amph.	Benomyl	Ridomil plus
<i>Staphylococcus aureus</i>	96	56	48	48	NA	NA
<i>Escherichia coli</i>	80	40	40	14	NA	NA
<i>Salmonella typhimurium</i>	100	64	56	44	NA	NA
<i>Pseudomonas aeruginosa</i>	96	70	60	28	NA	NA
<i>Cercospora cucurbitarium</i>	100	NA	NA	NA	95	85
<i>Fusarium solani</i>	100	NA	NA	NA	100	90
<i>Candida albicans</i>	100	NA	NA	NA	90	90

Legend: Strep: Streptomycin, Tet: Tetracycline, Amph: Ampicillin, NA: Not applicable.

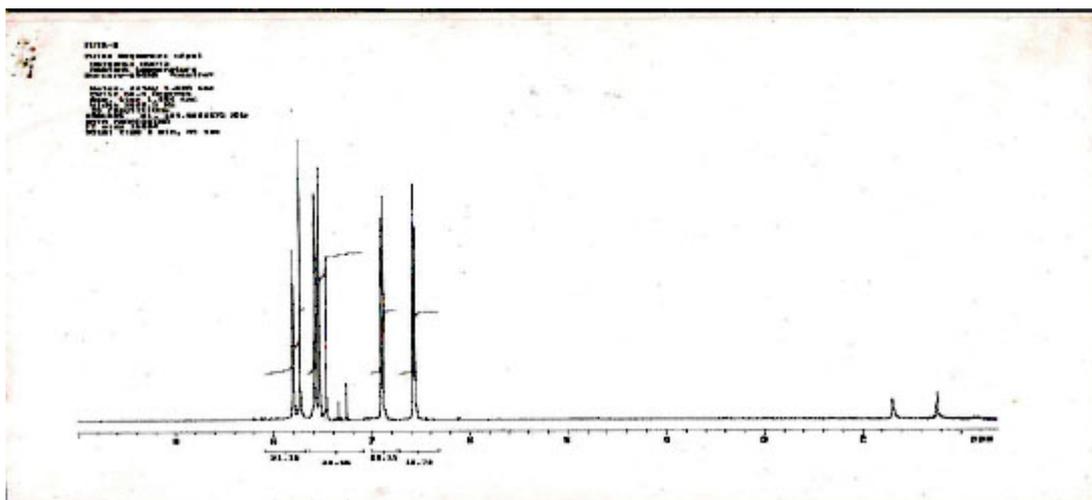


Fig. 1. Proton NMR of 2-(2-Nitrovinyl) Furan

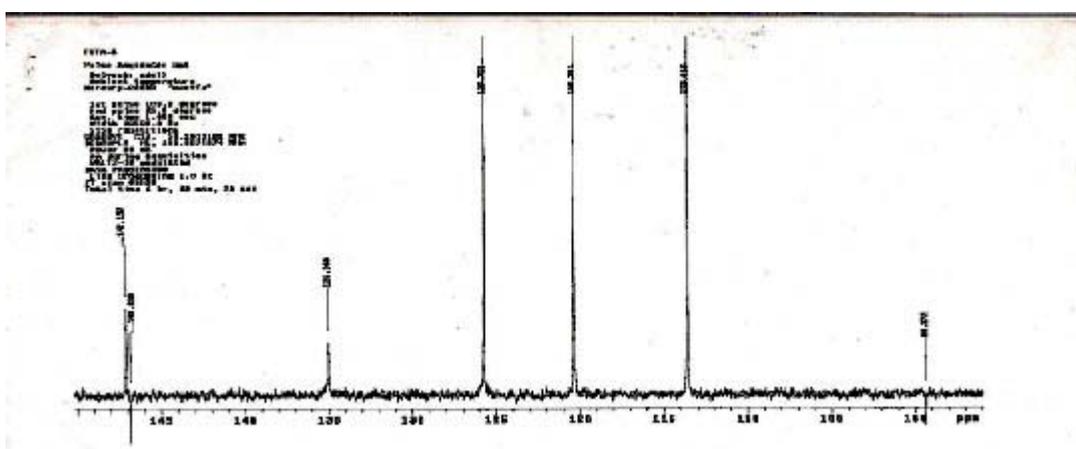
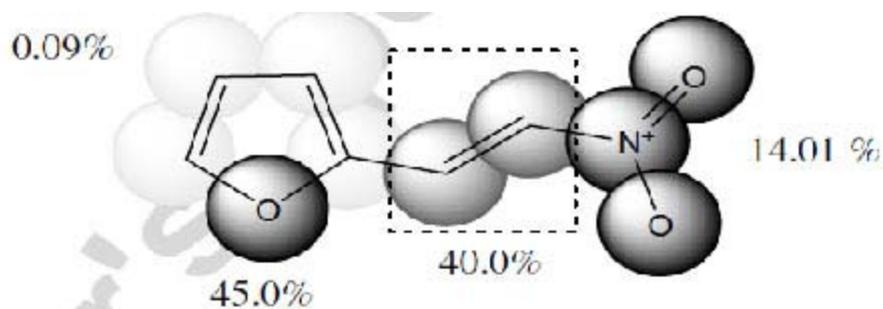
Fig. 2. Carbon 13 NMR (¹³C) of 2-(2-Nitrovinyl) Furan

Fig. 3. Structure of 2-(2-Nitrovinyl) Furan

control antibiotics. The zones of growth inhibition of the 2-(2-Nitrovinyl) furan almost double that of the standard antibiotics in the antibacterial assay. For instance, where the synthesized product had 96%, Streptomycin had 56%, Tetracycline had 48% and Amphotericin also had 48% zone of bacterial inhibition.

The 2-(2-Nitrovinyl) Furan restricted fungal growth at 100% level, where Benomyl and Ridomil plus inhibited the growth at less than 100%. Therefore, the 2-(2-Nitrovinyl) furan is more powerful antimicrobial than the standard antibiotics used.

CONCLUSION

It can be concluded that the 2-(2-Nitrovinyl) Furan can be synthesized by condensation reaction of the furfural with active methyl groups like nitromethane and because of the presence of nitro-group in the synthesized compound 2-(2-Nitrovinyl) Furan, it exhibited very high antimicrobial activity.

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REFERENCES

1. Akitt, J.W., Mann, B.E. NMR and Chemistry. Cheltenham, UK: Stanley Thornes. 2000; 273: 287.
2. Brady, J.E., Russell, J.W., Holum, J.R. Chemistry-Matter and its Changes, John Wiley, New York. NY, USA. 3rd edition. 2000; 1055-7.
3. Dunlop, A.P., Peters, F.N. The furans, Reinhold publication. Corp. New York 1953; 1055-1057.
4. Gonzalez-Diaz, H., Olazabal, E., Santana, L., Uriarte, E., Gonzalez-Diaz, Y., Castanedo, N. QSAR study of anticoccidial activity for diverse chemical compounds: Prediction and experimental assay of trans-2-(2-nitrovinyl)furan, *Journal of Biorganic and Medicinal Chemistry*, 2007; 962-968.
5. Wondu Business and Technology Service. Furfural Chemicals and Biofuels from Agriculture. *RIRDC publication*. 2006; ix.
6. Hornak, J.P. "The Basics of NMR". <http://www.cis.rit.edu/htbooks/nmr/>. 2009.
7. Ono, N., The Nitro Group in Organic Synthesis. A John Wiley and Sons, INC., Publication, New York 2001; 222.
8. Kalinowski, H., Berger, S., Braun, S. Carbon-13 NMR Spectroscopy, John Wiley and sons. Federal Republic of Germany. 1984; 251-300.
9. Keeler, J. Understanding NMR Spectroscopy. John Wiley and Sons. 2005; 100.
10. Martin, G.E., Zekter, A.S. Two-Dimensional NMR Methods for Establishing Molecular Connectivity. VCH Publishers. New York: 1988; 59.
11. Norrel, S.A. and Messley, K.E. Microbiology Laboratory Manual: Principles and Applications. Prentice Hall. Upper Saddle River. New Jersey. 1997;
12. Brenkem Consultants Asia Co Ltd. Info:info@brenkem.com. Sales. <http://www.brenken.com/furfural.htm> 19/87 soi Rewadi 64, Muang Nonthaburi, Nonthaburi, Thailand 11000. 2004.