

FUNCTIONALITY OF INOCULUMS IN FERMENTED GLUTINOUS RICE FOR THE PREPARATION OF CARBON NANOTUBES

S.F.Nik^{1,3}, N.F.A.Zainal^{1,3}, A.Azira^{1,3}, F. Mohamed^{2,3}, S. Abdullah^{1,3},
A. Z. Ahmed⁴ and M. Rusop^{2,3}

¹*Faculty of Applied Sciences
University Teknologi MARA, 40450 Shah Alam, Selangor, Malaysia*

²*Solar Cell Laboratory, Faculty of Electrical Engineering
University Teknologi MARA, 40450 Shah Alam, Selangor, Malaysia*

³*NANO-SciTech Centre, Institute Of Science
University Teknologi MARA, 40450 Shah Alam, Selangor, Malaysia*

⁴*Research Management Institute
University Teknologi MARA, 40450 Shah Alam, Selangor, Malaysia*

ABSTRACT

Inoculums are term used by microbiologists to describe a starter compound in composting process. Inoculums can be a combination of microbes; includes fungi, yeast and bacteria. In this paper, inoculums was introduced to steamed glutinous rice and a significant effect of the fermented glutinous rice can be observed using Fourier Transmission Infrared (FT-IR). The differences of spectrum before and after fermentation can be clearly seen in 1228 – 1420 cm⁻¹ range. The fermented glutinous rice was further used as a precursor in carbon nanotubes (CNTs) synthesis using thermal CVD. The CNTs was deposited on Ni coated silicon which acts as catalyst in deposition process. The CNTs was characterized using Scanning Electron Microscope (SEM) at various spots and magnification. Inoculums amount with 1.0 wt. % of glutinous rice showed the most favorable amount of inoculums as they can produced uniform CNTs rather than the other percentage. This new approach of starting material is expected to be commercialized due to its cheaper and renewable properties.

INTRODUCTION

Fermentation is one key process for the production of fermented glutinous rice, known as ‘tapai pulut’, an instant traditional food consumed in Asia especially in the South East region includes Indonesia, Malaysia, Philippine and China. It is popular in Asia-Pacific due to its subtropical humid climates, which is suitable for rice paddy cultivation and mould growth. Inoculums is a fermentation starters, also referred as ‘chu’ in Chinese, ‘nuruk’ in Korean, ‘koji’ in Japanese, ‘ragi’ in Southeast Asian countries and ‘bakhar ranu’ or ‘ marchaar’ in India (Batra and Millner, 1974). Inoculums is said to be a major role in reducing starch into glucose, ethanol and other

byproducts which usually happen almost simultaneously. The strains isolated from the inoculums were amylolytic, lipolytic and proteolytic. Glutinous rice fermentation in anaerobic condition produces ethanol, a mixture of residual glucose and other soluble matter [1].

CNTs can be described as cylindrical graphene sheets that are rolled up carbon compound in hexagonal structures. It can be single-walled nanotube (SWNT) or multi-walled nanotube (MWNT). They have special physical properties, depending on its chirality which gives different electrical, optical and structural properties. CNTs are usually synthesized using Arc-discharge technique, Laser ablation and Chemical Vapor Deposition (CVD) [2]. In this work, CVD was used as it was more practical and can produce both multi-walled CNTs and single-walled CNTs [3-4].

In this paper, the functionality of inoculums in fermented glutinous rice was investigated using FT-IR as it is less time-consuming than chemical methods, giving the same relevant information and allowing large screening experiment [5]. CNTs in this research were deposited from the prepared fermented glutinous rice, deposited on Ni coated silicon and further scrutinized using Scanning Electron Microscopy (SEM). The properties of using fermented glutinous rice as a starting material in CNTs synthesis was studied as different amount of starters or inoculums was used in the preparation of fermented glutinous rice. The consequence of utilizing different amount of inoculums in fermentation process for preparing the CNTs can be clearly seen in SEM micrograph.

EXPERIMENTAL

We adapted the traditional process for making fermented glutinous rice (*tapai* or *tah-peh*) for use at the laboratory scale as follows:

Preparation of saccharified glutinous rice

Significant amount of glutinous rice was blended for 1 minute. After blending the powdered rice was steamed using steamer (Pensonic PSM-121). The gelatinized glutinous rice was divided into 6 different beaker (in similar amount), and let the saccharified glutinous rice cooled to room temperature (27-28°C).

Preparation of inoculums

Significant amount of inoculums was measured (1, 3, 5, 7, 9 and 11 wt % of glutinous rice) and suspended in 4% ethanol. Standard inoculums were taken from Malaysian Agricultural Research and Development Institute (MARDI) as to ensure the standardization of inoculums used in this process.

Preparation of fermented glutinous rice

Saccharified glutinous rice was cooled to room temperature (27 – 28°C) and the prepared inoculums suspension was added and mixed well in each of the beaker. The prepared mixture was incubated in refrigerated incubator (Shel Lab LI 5) for 3 days at

30°C.

After incubation time, samples were centrifuged to separate the pellet and supernatant. Supernatant was further analyzed using FT-IR.

Screening test using FTIR

FT-IR Spectrometer Perkin Elmer Spectrum One 4035, bands between 4000-400cm⁻¹ at 16 cm⁻¹ resolution with 6 scans and data was collected and plotted using OriginPro7.5.

Preparation of substrate

Nickel (II) nitrate hexahydrate was spin coated on cleaned silicon wafer that has been washed meticulously. The coated silicon was dried for 24 hours.

Preparation of carbon nanotubes

Two system thermal-CVD was used in the preparation of CNTs. The prepared fermented glutinous rice was placed in quartz tube at furnace 1 and Ni coated silicon was placed in the same quartz tube at furnace 2. Argon was used as carrier gas. The CNTs deposited on Ni coated silicon was characterized using Scanning Electron Microscopy (SEM).

Screening test using SEM

Scanning Electron Microscopy Jeol 6360 LA was used as to characterize the surface morphology of the sample.

RESULTS AND DISCUSSION

FTIR spectra

All tested glutinous rice has successfully converted into *tapai* (*tah-peh*) due to the liquid of high glucose content released by the mass. Figure 1 shows the FTIR spectrum of 3.0 wt % of inoculums in sample before and after fermentation.

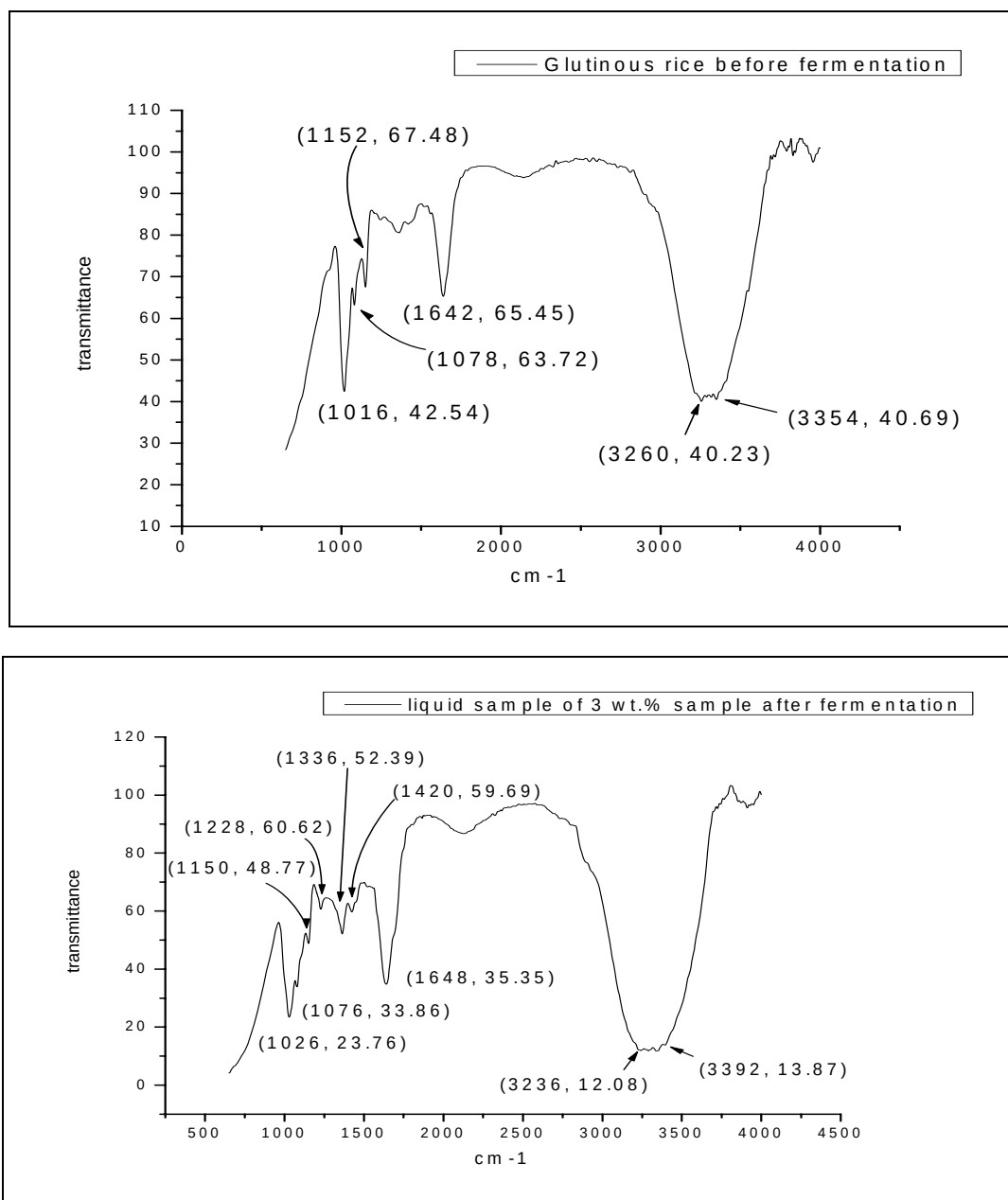


Figure 1: FT-IR spectrum of a) before fermentation of glutinous rice, b) after 3 days glutinous rice fermentation.

Peaks at 1078-1152 cm⁻¹ (fig.1a) range shows the C-O-C group in a six-membered ring glucose structure. After 3 days fermentation, the asymmetrical C-O-C stretching vibration moves progressively to lower wavenumbers as the ring structure becomes smaller [6], as can be seen in figure 1b. These indicate that during fermentation process, starch molecular structures had degraded to glucose, monomer structures of starch.

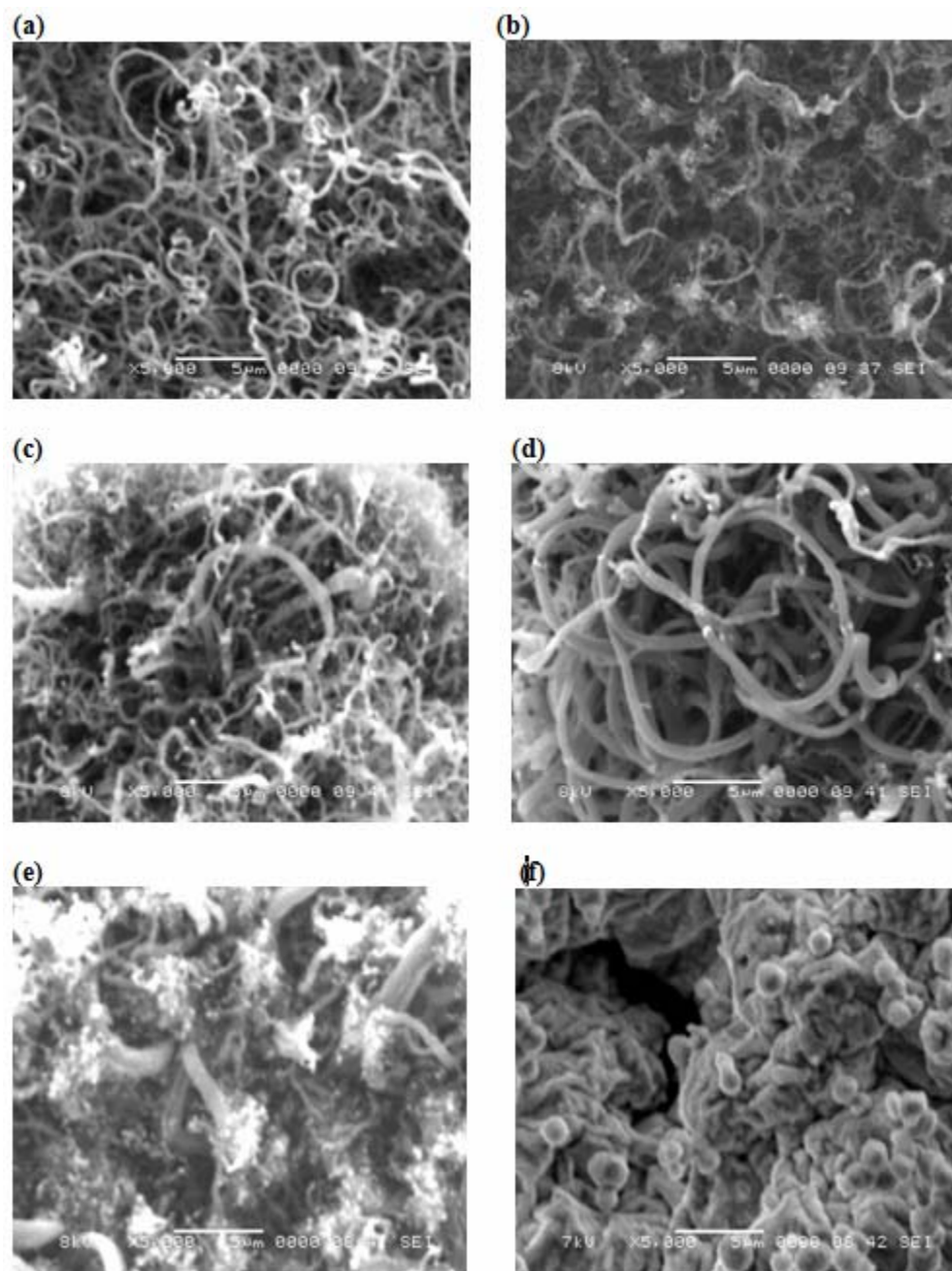


Figure 2: SEM micrographs of CNTs deposited on silicon using fermented glutinous rice at different amount of inoculums; a) with 1 wt% inoculums, b) 3 wt% inoculums, c) 5 wt% inoculums, d) 7 wt% inoculums, e) 9 wt% inoculums and f) 11 wt% inoculums.

The intensity of C-O vibration also changed in which it showed an increment to significant value after 3 days of fermentation process. This suggests the presence of new C-O alcohol structure bonding compound that existed as product in the fermentation process.

The obvious transformation of the spectrum can be seen in 1228-1420 cm^{-1} range (figure 1b) wherein the 1228 cm^{-1} indicates the formation of C-O stretching vibration of alcohol structure, whereas the existence of sharper peaks at 1336 cm^{-1} and 1420 cm^{-1} reflect the O-H in plane bending couples in alcohol with the C-H wagging vibrations that produces two bands; the first near 1420 cm^{-1} , the second near 1330 cm^{-1} [6].

Morphological detail

Figure 2 shows SEM images of CNTs deposited on Ni coated silicon using different amount of inoculums in fermented glutinous rice. This micrographs show that the most favorable inoculums amount for CNTs synthesis was fermented glutinous rice with 1 wt% of inoculums as it produces uniform with less defect structures of CNTs. The size of CNTs increases with the percentage rises of the inoculums amount, as can be seen in figure 2(a – d). The CNTs structures became destructed when higher inoculums amount were used in the process (figure 2e) meanwhile inoculums with 11 wt% could not produce CNTs at all. This indicates that the optimum amount of inoculums suitable used for nanotechnology application was 1 wt% of the glutinous rice.

CONCLUSION

Alcohol compound has been produced in the fermentation process as shown in FTIR spectra. New approach of starting material for CNTs fabrication was successfully developed from the prepared fermented glutinous rice. The structures quality of CNTs produced changed with different amount of inoculums used in the process. The optimum inoculums amount was 1 wt% of glutinous rice as it can produce the most favorable structures of CNTs.

ACKNOWLEDGEMENT

We were indebted to staff members of Biotechnology & Food Technology Research Institute, MARDI, for inoculums supplied, staffs of Chemistry Laboratory, Faculty of Applied Sciences and Material Science Laboratory, Faculty of Electrical Engineering, UiTM, for FT-IR test and SEM.

REFERENCES

- [1]. N. T. P. Dung et al., Characteristics of Some Traditional Vietnamese Starch-based Wines Fermentation Starters, *LWT* **40** (2007) 130-135.
- [2]. R. Saito, G. Dresselhaus, M. S. Dresselhaus, *Physical Properties Of Carbon Nanotubes*, Imperial College Press, London, 1998.
- [3]. K. Mukhopadhyay, A. Koshio, N. Tanaka, H. Shinohara, Jpn. A Simple and Novel Way To Synthesize Aligned Nanotube Bundles At Low Temperature, *J. Appl. Phys.* **37** (1998) L1257.
- [4]. S. Maruyama, R. Kojima, Y. Miyauchi, S. Chiashi, M. Kohno, *Chem. Low Temperature Synthesis Of High-Purity Single-walled Carbon Nanotubes From*

- Alcohol, Phys. Lett. **360** (2002) 229.
- [5]. I. Correia, A. Nunes, F. I. Duarte, A. Barros, I. Delgadillo, Food Chem., 90 (2005) 853-859.
- [6]. R. M. Silverstein, F. X. Webster, Spectrometric Identification of Organic Compound, **6** (1998) 79-91.