International Journal of Mechanical and Industrial Technology ISSN 2348-7593 (Online)
Vol. 13, Issue 2, pp: (51-57), Month: October 2025 - March 2026, Available at: www.researchpublish.com

Characterization of Fine Natural Clay Powder for Reinforcement in Bio Composites for Food Packaging Applications

[1]Imadu C. Cedrick, [2]Abugh Ashwe, [3]Nyior G. Bem

Department of Mechanical Engineering

Joseph Sarwuan Tarka University Makurdi, Nigeria

DOI: https://doi.org/10.5281/zenodo.17708347

Published Date: 24-November-2025

Abstract: **Characterization of natural clay powder using spectroscopic techniques as reinforcement in bio composites for food packaging applications was studied. Natural clay was pulverized to fine clay powder of size 100 µm using ball mills. The fine clay powder was subjected to spectroscopy using energy dispersive X-ray fluorescence (EDXRF), Fourier transform infrared (FTIR) and scanning electron microscopy/energy dispersive X-ray (SEM/EDX). The study found that, EDXRF spectra of natural clay powder showed, silica (SiO_2 34.851 %), alumina (Al_2O_3 19.092 %) and Ferric oxide (Fe_2O_3 2.3475 %). Also, TiO_2 (1.2855 %), K_2O (1.1438 %) and MgO (1.04 %). FTIR spectra of the clay sample revealed eight peaks. Bands at 3690.06446 and 3622.97238 cm^{-1} showed presence of hydroxyl linkage. The peak at 3429.15081 cm^{-1} is assigned to OH stretching. Bands at 1640.02865 cm^{-1} indicates possibility of water of hydration or H-O-H bending of water in the adsorbent due to the hydrous nature of the clay materials. Bands at 998.92654 and 909.47043 cm^{-1} respectively correspond to the Al-OH bending vibrations. Si-O stretching vibrations were observed at around 790-678.37549 cm^{-1}. The results of the FTIR showed no toxic functional group in the sample. SEM of the clay sample showed particle size of 100 µm. EDX result showed silicon with the highest weight concentration of 58.67 %, aluminium 26.55 %. The results showed no toxic or heavy element of concern in the clay. This is a confirmation suggesting that, the clay is safe and could be used for reinforcements in bio composites food packaging applications.**

Keywords: **Natural Clay, Fine powder, toxicity, Spectroscopy and bio-composites.**

1. INTRODUCTION

Materials mostly used in food packaging include; synthetic plastics such as Poly ethylene terephthalate (PET), polyethylene, polyvinylchloride, polypropylene, and many others owing largely to their; inertness, resistance to microorganisms, ability to act as a barrier to gases, ease of shaping/moulding and their ability to retain shape for long periods (Shin and Selke, 2014; Lima *et al.*, 2018). However, synthetic materials used for food packaging are toxic and after completing their service life, plastic-based package films form significant amounts of waste that lead to environmental and economic challenges. Hence, the deployment of bio composite in packaging films is considered as a sustainable alternative for overcoming the challenges of plastic-based packaging materials. The use of low-cost and widely available natural materials has advantages such as reduction in weight, increasing biodegradability and hence could provide solutions to end-of-life issues (Seda *et al.*, 2020).

Utilization of natural fillers and bio-based polymers to develop bioplastics as substitutes for synthetic polymers has received much attention in recent years especially in food packaging applications (Coppola *et al.*, 2021). Bio composites are composed of natural fillers and biopolymers such as polysaccharides (starch). These kinds of composites are also referred to as green composites and apparently exhibit good strength to weight ratio, increased flexibility, no skin irritation, low cost, reduced weight, renewable resource, sound insulation and thermal recycling (Goyal *et al.*, 2014; Akarsh *et al.* 2018).

Several studies; Kusmono and Zainal (2013), and Pulikkalparambil *et al.* (2023) have confirmed utilization of natural materials such as; natural fibres, natural clay, natural starch and glycerol in food packaging films as reinforcements, fillers, matrices and plasticizers with excellent results. Najafi *et al.* (2012) and Makaremi *et al.* (2017) reported that, clay is widely available in nature and can be used as a filler in bio composite due to its high mechanical, thermal, barrier, high benignity, and stability, low cost, availability, and sustainability. It significantly enhances the mechanical, physicochemical and degradable properties of composite even at a lower filler load (< 10 wt%).

According to Gustavo (2016) clay minerals are extremely fine materials that can be studied in detail by using X-ray techniques or sophisticated microscopic techniques, such as the electron scanning microscope. This study therefore, has characterized fine natural clay powder obtained from Benue State Nigeria by subjecting the natural clay samples to Fourier transforms infrared (FTIR) spectroscopy, energy dispersive X-ray (EDXRF) spectroscopy, and scanning electron microscopy attached with energy dispersive X-ray (SEM/EDX) spectroscopy, to explore the possibility of using the clay as a filler in bio composites. Clay reinforced polymers known as polymer–clay composites (PCNs) have demonstrated the potential to supplement the limitations of traditional food packaging solutions, especially cost effectiveness, environmental sustainability, and consumer safety.

2. METHODS

2.1 Preparation of fine clay powder

Bulk sample of natural clay was broken manually with a hammer to reduce the particle size and was Pulverized using Planetary Ball Mill (Thema ring mill) having 50 g of diameter 5 mm steel balls with a rotation speed fixed at 350 rotations per minute in the duration of 2hr. The fine clay powder was screened to pass through 100 µm sieve. Plate 1 shows the photograph of fine clay powder.

2.2 Characterization of Fine Clay Powder

Fine clay powder was subjected to the following spectroscopic analysis to determine its suitability for use in the reinforcement of bio composite for food packaging applications.

Energy Dispersive X-Ray Fluorescence (EDXRF) Spectroscopy

An energy dispersive XRF spectroscopy machine was employed as a primary source to determine the elemental composition in fine clay powder sample. This was done in line with Ipilakyaa *et al.* (2024). Natural clay was processed to fine powder of size 100 µm. The powdered sample was placed in a plastic sample cup and manually pressed to ensure a reasonable flat sample surface and presented to the XRF spectrometer system. An x-ray source was used to irradiate the specimen and cause the elements in the specimen to emit (fluoresce) their characteristic x-rays. The intensity of each characteristics radiation is directly related to the amount of each element in the material. The concentration of the elements present in the samples were obtained using AXIL-XRF software contained in the computer.

Fourier Transform Infra-red (FTIR) Spectroscopy

FTIR spectroscopy was carried out on fine clay powder of size 100 µm in accordance to ASTM 168-20. FTIR machine equipped with a focal plane array (FPA) detector at the mid-Infrared beamline was employed.

Plate 1: Photograph of Fine Clay Powder

A homogenous sample free of contaminants was loaded in a sample holder of the machine under vacuum pressure. The vacuum pressure caused the removal of moisture. IR spectrum bands were obtained when laser of infrared was projected onto the sample. The IR radiations impacts on the atomic vibrations of the molecules in the sample, resulting in the specific absorption and/or transmission of energy. The molecular components and structures contained in the specimen are displayed as absorption bands.

Scanning Electron Microscopy

Scanning Electron Microscopy attached with energy dispersive X-ray (SEM/EDX) was carried-out on fine clay powder. The test was conducted according to ASTM D2734 in line with Olaseinde *et al.* (2020) by placing fine clay on a sample holder (specimen stub) in the scanning electron microscope in vertical orientation. In this position, electron beam of 15 kV was focussed on the surface of the sample. The energy exchanged between the electron beam and the sample resulted in the reflection of high-energy electrons. The beam current absorbed by the specimen was detected and used to create images of the specimen and displayed on a computer monitor.

3. RESULTS AND DISCUSSION

3.1 Result of the Energy Dispersive X-ray Fluorescence (EDXRF) of Natural Clay

EDXRF spectra of fine clay powder is shown in Figure 1. Oxides found in clay and their percentage concentration are presented in Table 1. The result showed, major oxides present in clay in significant amounts include; silica (SiO_2 34.851 %), alumina (Al_2O_3 19.092 %) and Ferric oxide (Fe_2O_3 2.3475 %). While oxides in trace amounts include; TiO_2 (1.2855 %), K_2O (1.1438 %), MgO (1.04 %). According to Olaseinde *et al.* (2020) SiO_2 and $Al2O_3$ is a major component of common clay minerals such as bentonite. The presence of large amount of SiO_2 suggest that, the clay may be classified as silicate clay such as bentonite. This is consistent with the work of Ranjan *et al.* (2015) that has established that, SiO_2, $Al2O_3$ and Fe_2O_3 are the major constituents, while the remaining components could be found in trace amounts in natural clay samples. SiO_2 contributes to the hardness and durability of clay. It also, provides thermal stability, making the clay resistant to heat, thermal shock, chemical attack, particularly against acidic solutions. Aluminium oxide content increases the refractoriness (heat resistance) of clay, making it suitable for high temperature applications.

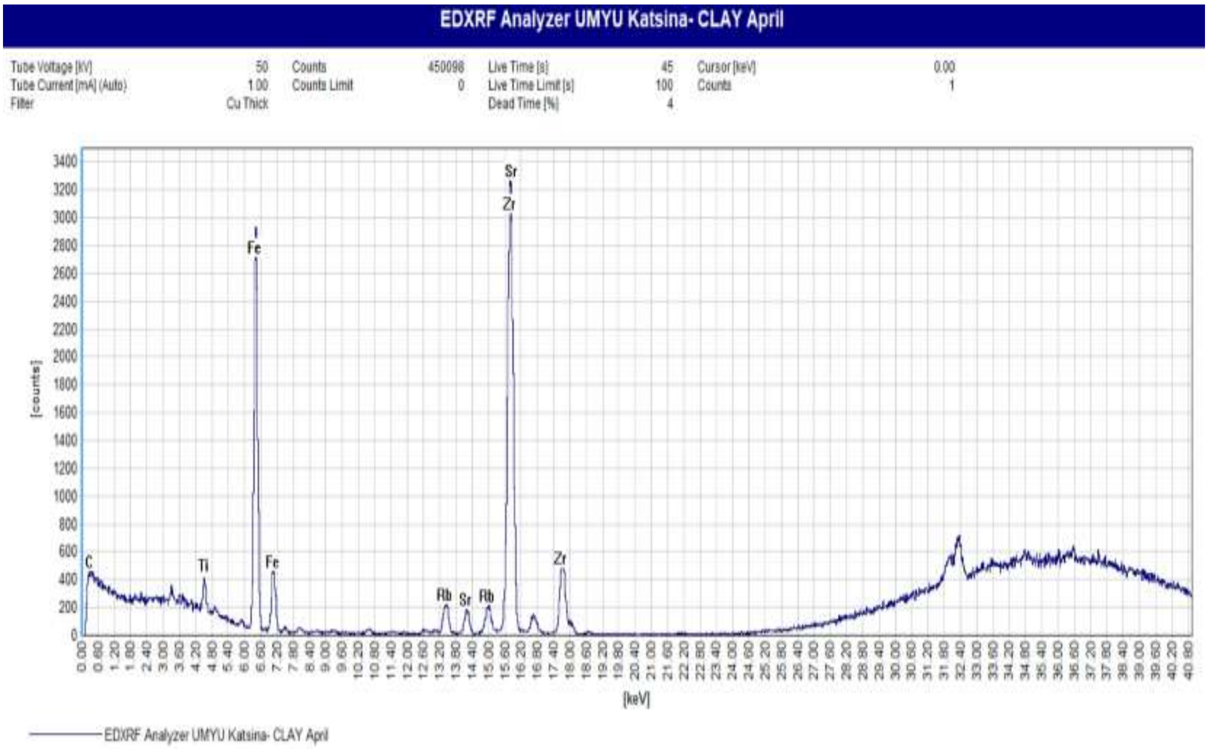


Figure 1: EDXRF Spectra of Fine Clay Powder

Results of the EDXRF has shown that, there is no toxic element of concern in the clay used in the study. Thus, the clay powder can be a potential source of reinforcement in bio composites especially, for food packaging applications.

3.2 Result of Fourier Transform Infrared (FTIR) Spectroscopy

FTIR spectroscopy was conducted on the natural clay powder to identify the functional groups and the finger prints present in the clay sample. The acceptability of clay in composite is dependent on the knowledge of its mineral content and chemical composition. The graph of transmittance versus wave number is shown in Figure 2. The FTIR spectra of clay powder shows eight peaks. Strong bands at around 3690.06446 and 3622.97238 cm^{-1} showed presence of hydroxyl linkage. The peak at 3429.15081 cm^{-1} is assigned to OH stretching which is close to 3424 to 3426cm^{-1} for bentonite spectra reported by Isci *et al.* (2006). Bands at 1640.02865 cm^{-1} in the clay spectrum indicates possibility of water of hydration or H-O-H bending of water in the adsorbent due to the hydrous nature of the clay materials (Nayak and Singh, 2007).

According to Wanyika *et al*. (2016) bands at 998.92654 and 909.47043 cm^{-1} respectively for natural-clays correspond to the Al-OH bending vibrations. Si-O stretching vibrations were observed at around 790-678.37549 cm^{-1} showed presence of bentonite.

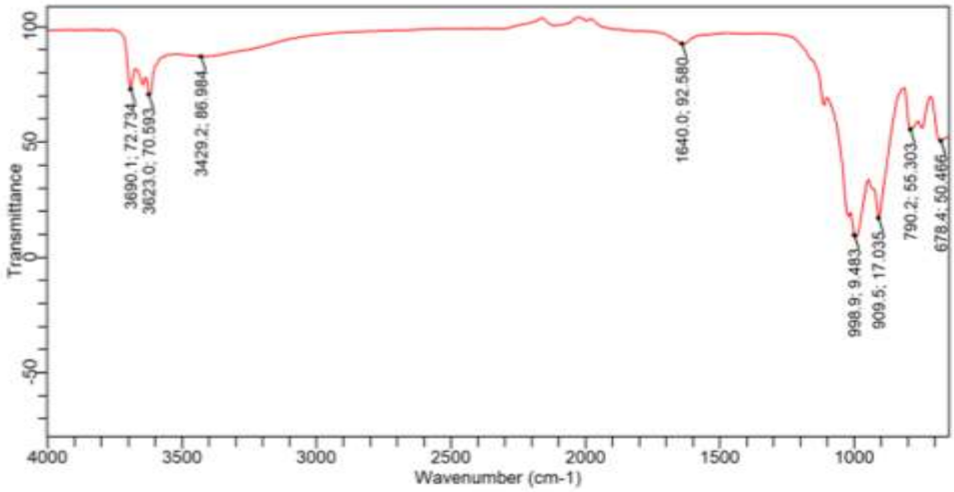


Figure 2: Fine Clay FTIR Spectra

The results of the FTIR on the natural clay shows no toxic functional group in the sample. This is a further confirmation of the safety of the clay used in the study.

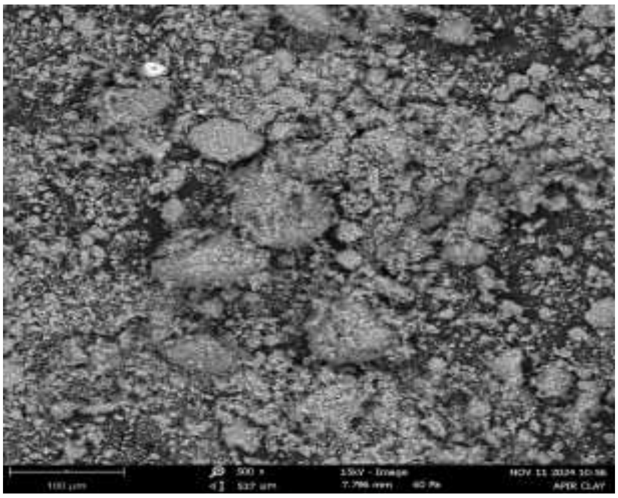
Table 1: Electron Dispersion X-ray Fluorescence (EDXRF) of Natural Clay Powder

Element	Concentration (%)	Peak(cps/mA)
Fe2O3	2.3475	8756
SiO2	34.851	2608
Al2O3	19.092	251
MgO	1.04	1
P2O5	0.0671	14
SO3	0.1484	81
TiO2	1.2855	4637
MnO	0.2703	818
CaO	0.2895	430
K2O	1.1438	1206
CuO	0.00889	28
ZnO	0.01072	58
Cr2O3	0.00633	51
V2O5	0.02528	169
As2O3	0	0
PbO	0.01197	5
Rb2O	0.00701	35
Ga2O3	0.00344	49

NiO	0.00610	30
Cl	0.01462	22
ZrO2	0.2453	377
Ta2O5	0.0096	10
Br	[0.000087]	0
SrO	0.491	27
Nb2O5	0.1419	15
Bi2O3	0.04672	2
Sb2O3	1	3
Co3O4	0	0
CdO	0	0
HfO2	0.00139	12
Ag2O	[0.00061]	1
CeO2	0.0459	49
BaO	1	2
Au	0	0
WO3	[0.06000]	10
MoO3	[0.0050]	3
La2O3	0	0
ThO2	0	0
SnO2	0	0
Y2O3	0.00348	21

3.3 Results of the Scanning Electron Microscopy/Energy Dispersive X-ray (SEM/EDX) of Natural Clay Powder

Results of the scanning electron microscopy SEM/EDX on natural clay samples are presented in Figures 3 and 4 respectively. The results show clay powder of size 100 µm. According to Karayannis *et al.*, (1997) utilization of fine clay particle size of 100 µm gave good mechanical properties and reduced water absorption of composites. Also, Onyedika *et al.* (2020) confirmed that fine clay particle size enhanced the mechanical properties of clay powder filled recycled low-density polyethylene composites.



Element Number	Element Symbol	Element Name	Atomic Conc.	Weight Conc.
14	Si	Silicon	60.63	58.67
13	Al	Aluminium	28.56	26.55
26	Fe	Iron	3.37	6.49
22	Ti	Titanium	1.23	2.02
15	P	Phosphorus	1.35	1.44
11	Na	Sodium	1.42	1.13
16	S	Sulfur	1.02	1.13
12	Mg	Magnesium	1.28	1.07
19	K	Potassium	0.51	0.68
17	Cl	Chlorine	0.43	0.53
20	Ca	Calcium	0.20	0.28

FOV: 537 µm, Mode: 15kV - Image, Detector: BSD Full, Time: NOV 11 2024 11:56

(a)

Figure 3: Scanning Electron Microscopy (SEM/EDX) of Natural Clay Powder

Figure 3 show the EDX of fine clay, the result shows the weight concentration of the elements found in the clay; silicon and aluminium are the major elements found in the clay. Silicon has the highest weight concentration of 58.67 %, aluminium has weight concentration of 26.55 %.

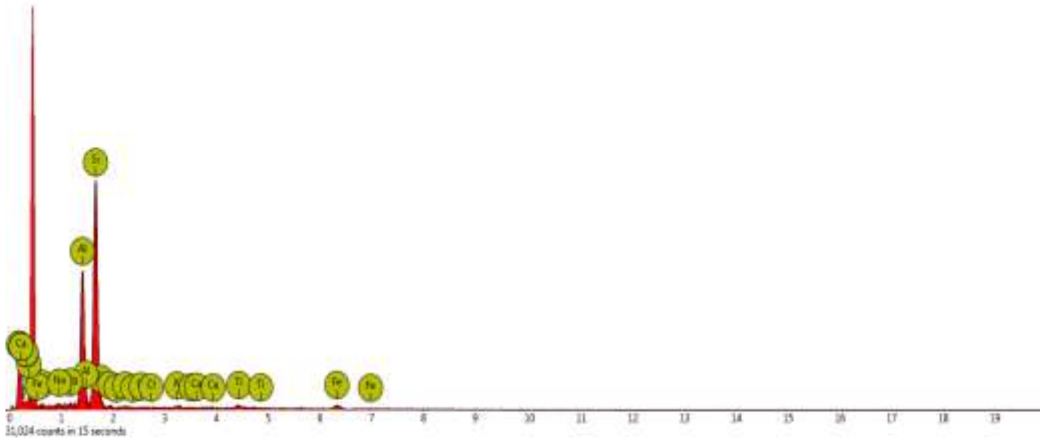


Figure 4: SEM/EDX Spectra

According to Najafi *et al.* (2012) and Makaremi *et al.* (2017) clay used as filler in composite enhances the mechanical, physicochemical and degradable properties of composite even at a lower filler load (< 10 wt%). Clay powder can improve the mechanical properties of composites more than the composites prepared from mineral/inorganic fillers such as glass fiber, talc, metal oxides and others (Onyedika *et al.*, 2020). The results show no toxic or heavy elements in the clay. This is an indication that, the clay is safe and could be used as reinforcement in bio composites production.

4. CONCLUSION

Fine natural clay powder was characterized using spectroscopy techniques and evaluated as a potential reinforcement material for bio composites in food packaging applications. The results showed that clay powder is safe and possesses desirable properties; EDXRF spectra of natural clay powder showed, silica (SiO_2 34.851 %), alumina (Al_2O_3 19.092 %) and Ferric oxide (Fe_2O_3 2.3475 %) in significant amounts. While TiO_2 (1.2855 %), K_2O (1.1438 %), MgO (1.04 %) in trace amounts. FTIR spectra of natural clay powder shows eight peaks. Strong bands at around 3690.06446 and 3622.97238 cm^{-1} showed presence of hydroxyl linkage. The peak at 3429.15081 cm^{-1} is assigned to OH stretching which is close to 3424 to 3426cm^{-1} for bentonite spectral. Bands at 1640.02865 cm^{-1} in the clay spectrum indicates possibility of water of hydration or H-O-H bending of water in the adsorbent due to the hydrous nature of the clay materials. Bands at 998.92654 and 909.47043 cm^{-1} respectively for natural-clays correspond to the Al-OH bending vibrations. Si-O stretching vibrations were observed at around 790-678.37549 cm^{-1}. The results of the FTIR on the natural clay shows no toxic functional group in the sample. SEM on natural clay sample showed clay powder of size 100 μm. EDX result showed silicon having the highest weight concentration of 58.67 %, aluminium has weight concentration of 26.55 %. The results showed no toxic or heavy element of concern in the sample. This is a further indication that, the clay is safe and could be used for reinforcements in bio composites food packaging applications.

REFERENCES

[1] Akarsh Verma, Kamal Joshi, Amit Gaur and V. K. Singh (2018). Starch-Jute Fiber Hybrid Biocomposite Modified with Epoxy Resin Layer: Fabrication and Experimental Characterization. doi:10.20944.

[2] Coppola, G.; Gaudio, M.T.; Lopresto, C.G.; Calabro, V.; Curcio, S.; Chakraborty, S. (2021). Bioplastic from Renewable Biomass: A FacileSolution for a Greener Environment. Earth Syst. Environ5, 231–251.

[3] Goyal, K. K., Jain, V., &Kumari, S. (2014). Prediction of Optimal Process Parameters for Abrasive Assisted Drilling of SS304. Procedia Materials Science, 6, 1572-1579.

[4] Ipilakyaa T, Daniel, Tile S. Emmanuel, Nyior G. Bem, Gundu D. Terfa (2024). Characterization of alkaline treated raffia palm fibres as reinforcement in polymer composite. Engineering and Technology Journal. Journal homepage: https://etj.uotechnology.edu.iq.

[5] Kusmono and Zainal Arifin Mohd Ishak (2013). Effect of Clay Addition on Mechanical Properties of Unsaturated Polyester/Glass Fiber Composites. International Journal of Polymer Science. ArticleID797109, 7pages http://dx.doi.org/10.1155/2013/797109

[6] Lima EMB, L AM, Mungiata APS, Santos NRR, Preira ICS, Neves TTM, Gonçalves LFC, Moreira APD, Middea A, Neuman R, Tavares MIB, Oleveira RN, (2018). Poly(lactic acid) biocomposites with mango waste and organo-montmorillonite for packaging. Journal of Applied Polymer Science, 1-11.

[7] Makaremi, M., Pasbakhsh, P., Cavallaro, G., Lazzara, G., Aw, Y.K., Lee, S.M., Milioto, S. (2017). Effect of morphology and size of halloysite nanotubes on functional pectin bionanocomposites for food packaging applications. ACS Appl. Mater Interfaces 9, 17476–17488. https:// doi. org/ 10. 1021.

[8] Najafi, N., Heuzey, M.C., Carreau, P.J. (2012). Polylactide (PLA)-clay nanocomposites prepared by melt compounding in the presence of a chain extender. Compos. Sci. Technol. 72, 608–615.

[9] Pulikkalparambil, H, Varghese, S.A.; Chonhenchob, V., Nampitch, T., Jarupan, L., Harnkarnsujarit, N. (2023). Recent Advances in Natural Fibre-Based Materials for Food Packaging Applications. Polymers 15, 1393. https:// doi.org/10.3390/polym15061393.

[10] Seda ERSUS (2020). Production Of Biocomposite Packaging Materials From Fruit Juice Processing Wastes. Journal of the Institute of Science and Technology, 10(1): 250-259.

[11] Shin J, Selke SEM, 2014. Food packaging, Food Processing: Principles and Applications, Second Edition. Edited by Stephanie Clark, Stephanie Jung, and Buddhi Lamsal. © 2014 John Wiley & Sons, Ltd. Published 2014 by John Wiley & Sons, Ltd.