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Elemental and Spectroscopic Analysis of Organic Matter Transformation during Forced Aeration of In-Vessel Composting

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Abstract: This work examines the spectral, elemental and SEM images of compost from different blends obtained from food waste. Four composting bins, each of 100 L capacity, were installed with holes at various depths for probes and leachate collection system. The bins (length 50 cm, width 40 cm, height 50 cm) were placed horizontally with a slight inclination to permit the collection of leachate. The blends were prepared from food waste, cow dung and saw dust as bulking agent. The evaluation studies included the operational indices such as temperature, pH, moisture content, compost maturity indices like carbon, nitrogen, C/N ratio, sulphur, Fourier Transform Infra-Red (FTIR) analysis and Scanning Electron Microscope (SEM) images. SEM images revealed a reduction in the easily assimilated peptide and carbohydrate components. The germination index for tomato seed was above 60% after dark room studies, showing that the final compost was not phytotoxic.

Key words: C/N ratio, Fourier Transform Infra-Red (FTIR), SEM observation.

Introduction

The necessity to preserve natural resources and the optimization of use of non-renewable energy had encouraged the recycling and recovery of organic waste as an alternative to dumping and incineration (Abdelmajid et al.). Composting could be defined as the controlled biological decomposition of organic substrates carried out by successive microbial populations combining both mesophilic and thermophilic activities, leading to the production of a final product sufficiently stable for storage and application to land without adverse environmental effects. Two types of composting system had been widely applied: In-vessel systems and Windrow systems. The technology called in-vessel composting comprised a number of integrally related components including: materials (sludge cake), amendment, recycle, handling, storage mixing, reactor system, odour-control system, aeration system, exterior curing/storage facilities as well as marketing (Marek et al.). The system had advantages over the windrow system as it required less space and provided better control than windrows. As it involved high process efficiency, food waste had been successfully composted for those purposes (Joung-Dae Kim et al.). This study had been conducted to evaluate the performance of a lab-scale in-vessel composting plant.

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Forced-aeration processes had been recognized as simple, economical and quick treatment processes for organic solid waste. It had also interested operators of other systems because of their low odour emission, which resulted from their turning operation and an adequate air supply (Masafumi Tateda et al.). The purpose of the investigation was to follow some of the physico-chemical changes occurring during the composting of food waste when mixed with bulking agents like saw dust. The evolution of compost maturity was assessed from data obtained by elemental analysis, physical analysis, FTIR spectroscopy and SEM images. Tomati et al. had suggested the use of different maturity indices such as C/N ratio, humification indices, and germination indices for the evaluation of compost.

Materials and Methods

Composting

The process of composting was studied using six different types of reactors, each simulating a different condition for the formation of compost. Bio-degradable solid waste from the transfer station was mixed with cow-dung slurry and the bins were loaded with saw dust. The cow dung was obtained from the cattle shed and was dried to remove odour and then 10% cow dung slurry was prepared as follows. 100 g of cow dung was mixed with 1000 ml of water. The fibres in the water were filtered using cloth filter. The characterization of food waste and the available bulking agent before composting was of primary importance to balance the recipe in terms of moisture

Table 1: Physico-chemical features of waste and other agents for composting

Characteristics	*Method/Instrument*	*Biodegradable waste*	*Cow dung*	*Saw dust*
pH	pH meter	4.4	7.8	4.7
Temperature °C	Stem Thermometer	30	24	27
Moisture Content (MC) (%)	Hydrometer	80	65	40
Carbon (C) (%)	Vario Elemental Analyzer	47.4	42	46.5
Hydrogen (H) (%)	Vario Elemental Analyzer	8.9	9.3	8.8
Nitrogen (N) ((%)	Vario Elemental Analyzer	2.0	0.65	0.22
Sulphur (S) (%)	Vario Elemental Analyzer	0.8	0.3	0.1
C/N	Carbon Nitrogen ratio	24	65	211
Phosphorous (%)	Acid digestion method	0.358	0.63	1.1
Potassium (K) (%)	Flame photo meter	0.9	0.36	0.04

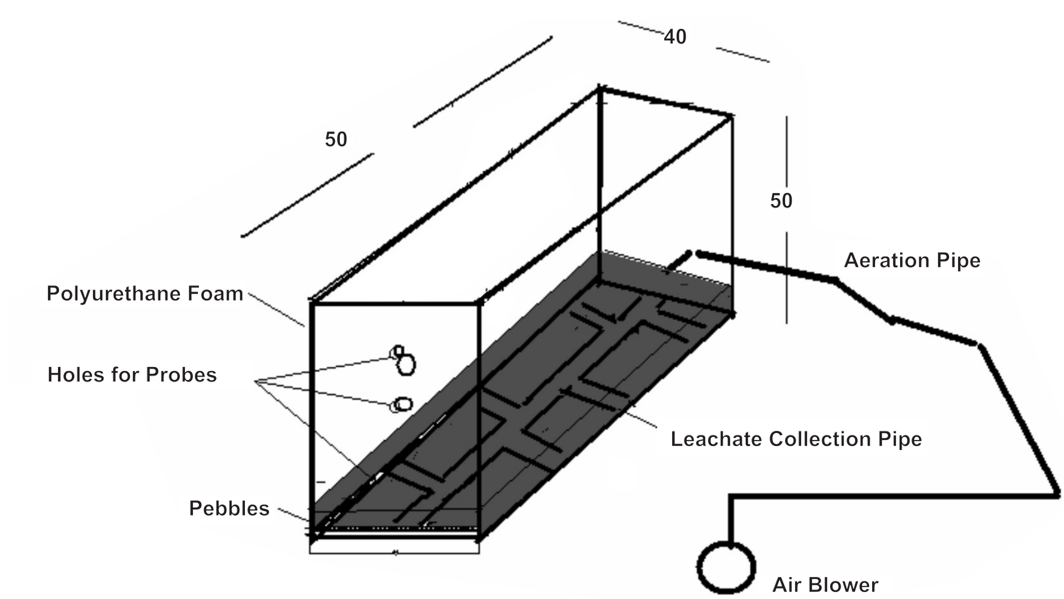

Figure 1: Diagram of rectangular bin composter.

content for aeration, pH for a proper microbial environment and carbon nitrogen for proper microbial development. Aeration was facilitated when the compost mixture offered a 30% free air space, which may vary depending on the substrates and the composting systems (Neklyudov et al.).

To allow optimization of the composting process, the food waste must be characterized for moisture content (MC), pH and C/N ratio (Pace et al.). Table 1 gives the characteristics of food waste, bulking agent and cow dung. Four experimental blends as in Table 2 were carried out in simulated aerobic degradation conditions under mesophilic temperature. The four blends used separately in each run were on a weight basis carried out using rectangular plastic bins (length 50 cm, width 40 cm, height 50 cm). A plastic mesh was fitted at the bottom of the receptacle to support the material and separate it from possible leachate. A 3-cm polyurethane foam layer provided thermal insulation. Several holes were perforated through the walls of the vessel to permit air movement, leachate removal and the insertion of different probes. Finally the germination study was done to find out seed germination and root elongation of blends for evaluating the compost quality. (Tiquia et al.). Forced aeration at a rate of 2.3 m^3/min was supplied to maintain adequate oxygen level and temperature inside the compost. It was operated for a period of one hour per day at a rate of 2 min 30 sec per hour with timer system using a blower.

Physico-chemical Analysis

The pH of a suspension of compost in water (1:2 v:v) was measured using Elico Instrument. The temperature was measured using multi stem thermometer. The probe of the thermometer was cleaned with an abrasive soft paper and planted at ¾ of its length. It was allowed to stand for one minute and the switch was slided ON. The temperature was noted in ^oC in the digital display. The moisture content (MC) was measured using hydrometer. The probe shaft of the instrument was cleaned with an abrasive soft paper and planted at ¾ of its length. It was allowed to stand for one minute and the needle showed the moisture content directly. All other parameters were measured by collecting the sample at centre after uniform mixing and drying the same at 105^o for one hour. The dried samples were powdered and used for analysis.

Vario EL Analyzer

Carbon (%C), hydrogen (%H), nitrogen (%N) and sulphur (%) were measured using Vario EL elemental analyzer. The samples after sieving in 4.75 mm mesh sieve were ground using mortar with pestle. The fine powder after drying was used for analysis. The analyzer works according to the catalytic tube in an oxygenated carbon dioxide atmosphere and high temperature. The combustion gas were free from foreign gases and the desired measuring components were separated from other with the help of specific adsorption columns. It was determined in succession with thermal conductivity detector consisting of two measuring chambers. The constantly tempered adsorption column 1 would remove first SO_2 quantitatively from the gas stream. Subsequently the reaction gas was dried in H_2O absorption column 2. The absorption column 3 finally would adsorb the CO_2. Now the measuring gas stream contains only N_2, which was directly measured in the thermal desorption. The measuring data was displayed on the screen after every analysis and stored in a temporary file.

Fourier Transform Infra-red Analysis

KBr pellets of each sample to be analysed were prepared by compression under vacuum of a mixture of 2 mg dry sample (105 ^oC, 24 h) with 400 mg KBr. Spectra were obtained with a Bruker IFS66vFT-IR spectrophotometer by subjecting the pellets to a frequency of 4000–400 cm^{-1} at 16 nmsec^{-1}. The interference pattern obtained from a two-beam interferometer as the path difference between the two beams was altered and when Fourier transformed, gave rise to the spectrum. The transformation of the interferogram into spectrum was carried out mathematically with a dedicated on-line computer. Solid samples were dispersed in KBr or polyethylene pellets depending on the region of interest.

Table 2: Typical composition of blends based on weight

S.No	*Materials*	*Blend 1*	*Blend 2*	*Blend 3*	*Blend 4*
1	Cow dung (g)	500	500	500	500
2	Bio solid waste (g)	5000	5500	6250	7500
3	Saw dust (g)	4500	4250	3250	2000
	Total (g)	10000	10000	10000	10000

Scanning Electron Microscope

The Scanning Electron Microscope (SEM) was a type of electron microscope that imaged the sample surface by scanning it with a high-energy beam of electrons in a raster scan pattern. The electrons interact with the atoms which make up the sample producing signals that contain information about the sample surface topography, composition and other properties such as electrical conductivity. The Jeol JSM-7001F SEM characterizes nanostructures with a resolution of 1.2 nm at 30 kV and accommodates multiple detectors. The solid specimens were coated with a layer of ultra thin coating of gold using low vacuum sputter.

Seed Germination Studies

The seed germination and root elongation of *Lycopersicon esculentum* (tomato) species were used to evaluate quality of compost. The compost obtained from different blends were taken in plastic pots. It was spread in layer of about 3 cm height. A trace paper was placed in the top and sprinkled with water. After five days of incubation in the dark, the seed germination, root elongation and germination index (GI, a factor of relative seed germination and relative root elongation) were determined. A 5-mm primary root was used as the operational definition of germination (USEPA, 1982). The percentages of relative seed germination, relative root growth, and GI were calculated (Tam & Tiquia).

Relative seed germination (%) - Number of seeds germinated in compost/ Number of seeds germinated in control × 100 (1)

Relative root growth (%) - Mean root length in compost/ Mean root length in control × 100 (2)

GI - (% Seed germination) × (Root growth) × 100 (3)

Results and Discussion

Temperature

The variation in temperature recorded during the process was typical of a composting process (Tomati et al.). An initial stabilization phase was characterized by an increase in temperature reaching 60°C. This temperature rise resulted from intense microbial activities favoured by the high concentration of easily decomposable organic molecules. Afterwards during the maturation phase the temperature decreased steadily to reach an ambient 25° C at the end of the process as in Table 3.

Table 3: Variation of temperature

Day	*Blend 1*	*Blend 2*	*Blend 3*	*Blend 4*
0	30	30	30	31
5	62	65	64	60
10	60	63	61	58
15	55	58	57	54
20	52	54	53	52
30	42	45	42	45
45	36	38	35	36

pH

Table 4 displays the results of the monitored pH of the composting material. Composting proceeds most efficiently at the thermophilic temperature when the pH is approximately 8 (Liao et al.). Throughout the experiment, the pH level is found to be 8 or slightly more. A maximum pH value of 8.2 was observed on the tenth day, which further reduced to 7.9. Increase in the pH level during composting resulted from increase in volume of ammonia released due to protein degradation (Liao et al.).

Table 4: Variation of pH

Day	*Blend 1*	*Blend 2*	*Blend 3*	*Blend 4*
0	7.8	7.8	7.7	7.8
5	8	8	8.1	8
10	8.1	8.2	8.2	8.1
15	8.1	8.2	8.2	8.1
20	8	8.1	8.2	8
30	8	8	8	7.9
45	7.9	7.9	7.8	7.8

Moisture Content (MC)

In the experiments, the moisture content at the beginning was 60% and reduced to 50% within 20 days. During the initial days of composting, the moisture content was within an acceptable range of 50–65%. A small drop was observed at the end as the upper part was exposed to the environment, resulting in extra moisture loss which can

Table 5: Variation of moisture content in %

Day	*Blend 1*	*Blend 2*	*Blend 3*	*Blend 4*
0	60	60	60	60
5	58	57	58	58
10	56	55	57	56
15	54	54	55	55
20	52	53	54	54
30	52	52	53	54
45	50	52	52	55

be observed in Table 5. Leachate formation was not observed during the composting period.

Carbon (%C)

The change in the TOC content during the composting period is detailed in Table 6. The content of organic carbon decreased as the decomposition progressed. Initially, the amount of organic carbon in the material was 48%, which then reduced to 29% in blend 1. In the case of blend 4, the carbon in the material reduced from 47% to 18% in the end of 45 days. Around 30% of the available carbon was utilised by micro-organisms as a source of energy (Polprasert).

Table 6: Variation of carbon (%)

Day	*Blend 1*	*Blend 2*	*Blend 3*	*Blend 4*
0	44.62	45.83	44.73	44.85
5	40.1	42.8	31.27	30.27
15	38.3	38.5	24.1	23.97
30	34.05	32.9	22.82	20.61
45	32.3	27.19	18.65	18.8

Nitrogen (%N)

Mineralization of organic nitrogen in compost was dependent on many factors including C/N ratio of raw material, composting conditions, compost maturity, time of application and compost quality (i.e., C/N ratio and C- and N-fractions). The nitrogen content had increased in blends 1, 2 and 3 probably by mineralization of organic matter as well as loss of carbon. The increase of nitrogen shows the good quality of bio compost as in Table 7.

Table 7: Variation of nitrogen (%)

Day	*Blend 1*	*Blend 2*	*Blend 3*	*Blend 4*
0	1.10	1.19	1.32	1.54
5	1.06	1.38	1.36	1.14
15	1.27	1.25	1.42	1.17
30	1.3	1.08	1.39	1.05
45	1.36	1.02	1.18	1.11

C/N Ratio

The compost from blends 1 and 2 would not be suitable for land application since the excess carbon would tend to utilize nitrogen in the soil to build cell protoplasm, consequently resulting in loss of nitrogen in the soil on which it would be applied. On the other hand, the C/N in the compost formed in blends 3 and 4 would help to improve the soil structure as in Table 8 (Bhattacharyya et al.).

Table 8: Variation of C/N ratio

Day	*Blend 1*	*Blend 2*	*Blend 3*	*Blend 4*
0	40.81	38.40	28.90	33.85
5	37.83	31.01	26.55	22.99
15	30.16	30.80	20.49	16.97
30	26.19	30.46	19.63	16.42
45	23.75	26.66	16.94	15.81

Sulphur

Sulphur had been recognized as the fourth major plant nutrient along with nitrogen, phosphorous and potassium (NPK). In sulphur deficit soil, applying NPK rich manure cannot ensure high yields unless sulphur was also applied. Hence the sulphur content in the mulch was also analyzed along with the other parameters (Tandon). The sulphur content shows an increasing trend as in Table 9.

Table 9: Variation of sulphur (%)

Day	*Blend 1*	*Blend 2*	*Blend 3*	*Blend 4*
0	0.37	0.46	0.55	0.64
5	0.104	0.074	0.127	0.284
15	0.72	0.151	0.168	0.125
30	0.53	0.62	0.85	0.63
45	0.53	0.62	0.85	0.63

Germination Study

The results of the seed germination and root elongation test in the case of saw dust showed that blends 3 and 4

Table 10: Germination studies

Blend. No	*Number of seeds germinated in blends*	*Number of seeds germinated in control*	*Relative seed germination %*	*Mean root length in blends (cm)*	*Mean root length in control (cm)*	*Relative root growth %*	*Germination index*
1	6	20	30	3.8	5	76	54
2	6	20	30	4.1	5	82	56
3	10	20	50	4.2	5	84	61
4	10	20	50	4.1	5	82	62

could be used for agricultural application. The relative germination percentage of tomato was as high as 60% at the end of the germination period for blends 3 and 4. On the other hand, blends 1 and 2 had an initial relative germination percentage around 50%. The GI values for the compost-based substrates were practically the same, especially after the germination period, which showed the absence of detrimental effects derived from high salt or toxic substances presence in these substrates. As pointed out by Zucconi et al., a GI value lower than 60% would indicate incomplete stabilisation of the organic matter and consequently the presence of toxic substances. The GI values for the compost-based substrates were in the range of 30–60%.

FTIR Analysis

FT-IR spectroscopy was used to monitor the composting process, evaluate the degradation rate and thus determine the maturity. In the case of saw dust, the main absorbance in the FT-IR spectra of blends 3 and 4 based on C/N ratio around 20 were considered. The characteristic stretching for important functional groups such as OH, NH and C=O occur in this region. There is a very large band located between 4000 and 3000 cm^{-1} centred around 3400 cm^{-1} which is due to hydrogen vibrations of the OH groups of alcohols, phenols or carboxylic acids, as well as to amide hydrogen vibrations. In blends 3 and 4 absorption bands at 2925 cm^{-1} are attributed to aliphatic methylene groups and assigned to fats and lipids. Lipids are an important fraction of compost that could influence the water retention capacity of amended soils, their structural stability and the biodegradation–humification balance in soils as in Figures 2 and 3.

SEM Observation

Studies on the macroscopic properties of compost seem to be of especially great importance to research their multiscale micro- and nano-structures (Ying et al.). The SEM micrographs of the internal structure of the blends are shown in Figures 4 and 5. It shows a porous structure, as a result of which a large number of bacteria cells grow on the surface. The presence of various functional groups as discussed previously by FTIR analysis in the blends probably have an important part in initiating the biodegradation of raw blends by enabling the colonization and multiplication of cells of microorganisms on the film surfaces.

Conclusion

Performance of lab-scale in-vessel composting for biodegradable solid waste was valuated in this study. The

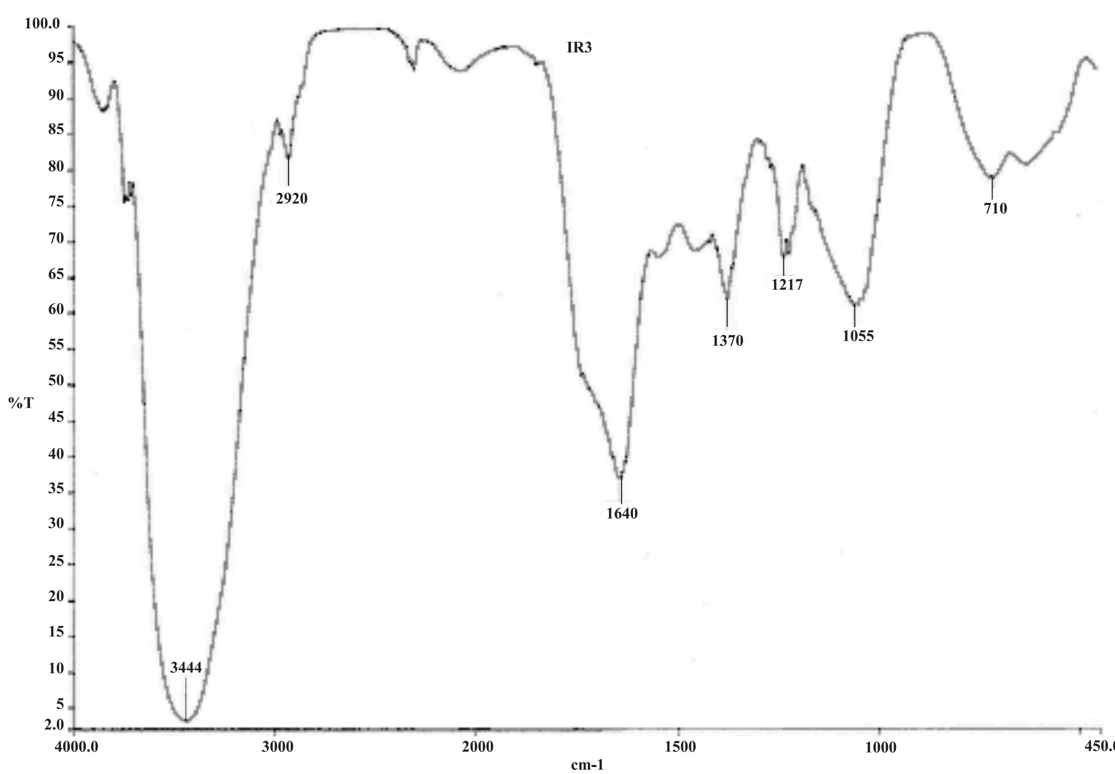

Figure 2: FTIR showing the characteristics of compost blend 3.

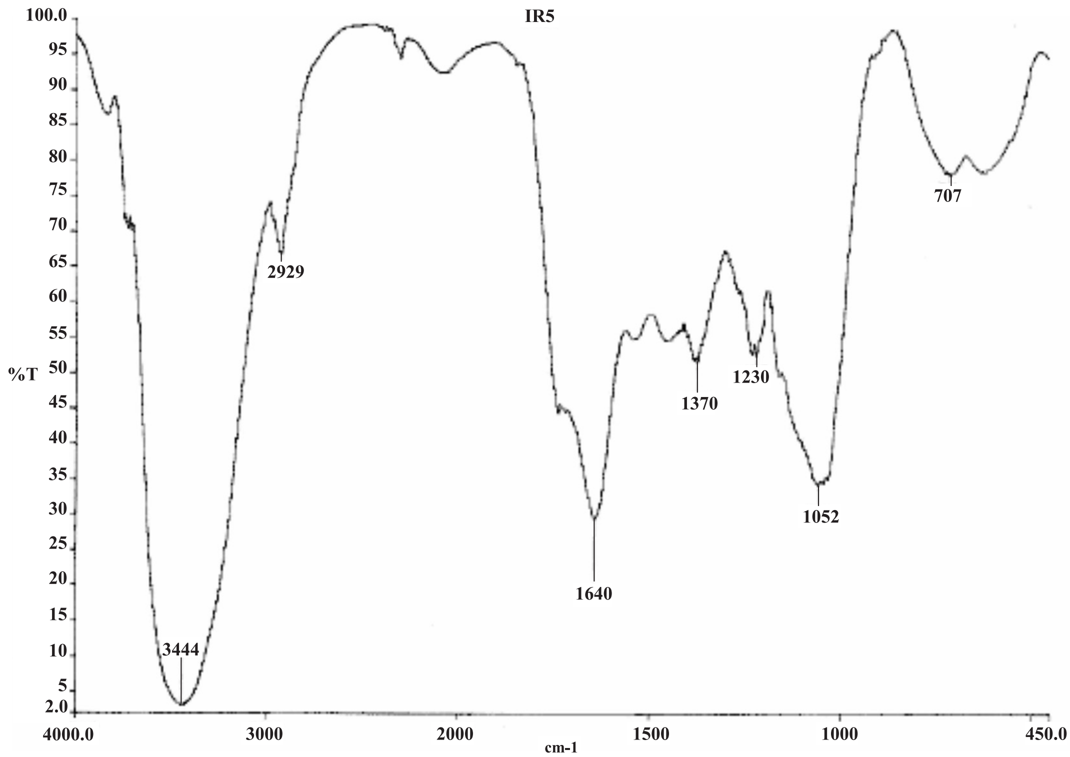

Figure 3: FTIR showing the characteristics of compost blend 4.

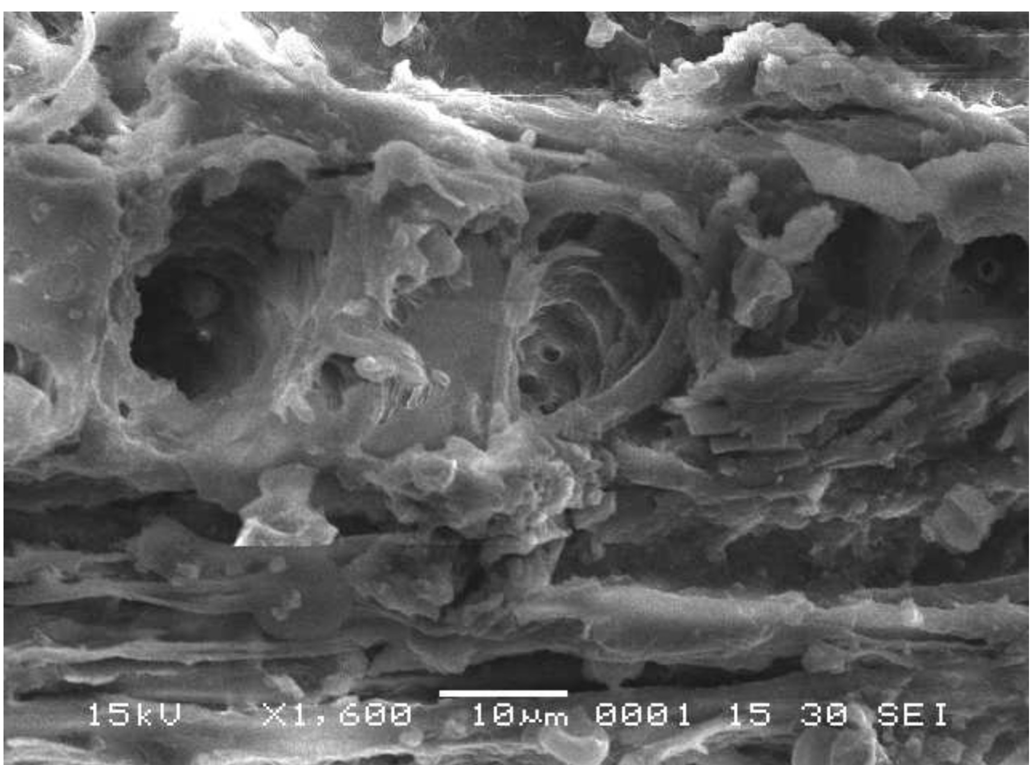

Figure 4: SEM observation of compost blend 3.

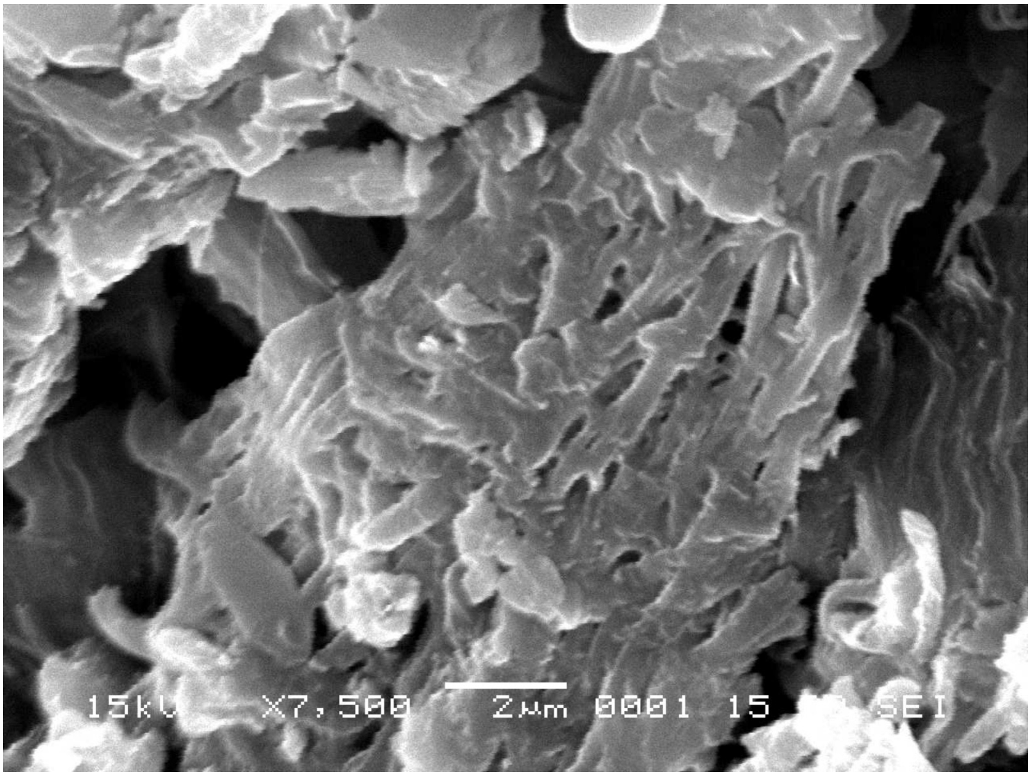

Figure 5: SEM observation of compost blend 4.

results of the investigations enabled one to prove the usefulness of the in-vessel composting method for utilizing bio degradable solid waste. Specific conclusions can be drawn from moisture content, pH, temperature and C/N ratio. The bulking agents like saw dust were found to offer the best properties with neutral pH and a moderately C/N ratio and could be recycled. The blends 3 and 4 had a C/N ratio around 20 which proved to be ideal for agricultural application. The test of sprouting in blends 3 and 4 proved that the mature compost had a better quality than garden soil that was used for comparison. However, the SEM micrographs showed that the amount of bacteria on the surface of the films continued. Hence the technology could be introduced in residential areas and the compost used to fertilize plants.

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