

Original Research Article

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Industrial Waste Composts: Toxicity Tests and Decomposition Studies under Laboratory Conditions

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ABSTRACT

Enzyme industrial wastes viz. multiple effect evaporator salts, primary sludge, filter press feed were evaluated for their phytotoxic effects under laboratory conditions to understand their potential to use as a nutrient medium for supporting plant growth. It was found that the industrial waste-water extract recorded lower contents of essential nutrients and the presence of heavy metals viz. Ni and Cd. Germination studies revealed the inhibitory effects of industrial waste-water extracts on percentage and rate of seed germination and length of plumule and radicle. None of the seeds germinated in MEES: water extract and seed germination of tomato as indexed by rate 6.11 in PS: water extract exhibited the inhibitory effect by primary sludge on seeds. The length of radicle (5.79) and plumule (4.94) was relatively lesser in PS: water extract to that of FPF: water extract and control. The incubation study carried out in the laboratory conditions to understand the rate of decomposition of urban solid waste alone and three different industrial waste-composts prepared by combining urban solid waste with enzyme industrial wastes viz. multiple effect evaporator salts, primary sludge, filter press feed revealed that the carbon-di-oxide evolved was higher in incubating urban solid waste-multiple effect evaporator salts exhibiting a higher rate of decomposition due to the presence of more easily degradable compounds. This was 6.10 mg CO₂ 100 gc⁻¹ day⁻¹ on the 50th day of incubation in urban solid waste-multiple effect evaporator salts and 2.60 mg CO₂ 100 gc⁻¹ day⁻¹ in incubating urban solid waste alone at the 50th day of incubation. The cumulative CO₂ evolved ranged from 32.27 mg CO₂ 100 gc⁻¹ in urban solid waste alone to 89.48 mg CO₂ 100 gc⁻¹ in urban solid waste+ multiple effect evaporator salts on the 50th day of incubation.

Keywords

Enzyme Industrial wastes, Urban solid waste, compost, Phytotoxicity, C decomposition

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Introduction

Wastes are potential sources of nutrients which can be used in agriculture for the supply

of nutrients as well as a soil conditioner. Generally these wastes are considered to be rich in organic matter and essential nutrients for plants and microorganisms (Gendebein *et*

al., 2001; Cintya *et al.*, 2012). Enzyme industrial wastes are considered to be organic due to its origin. But the composition of these waste materials is variable which depends on the process or treatment involved in the industry. Urban solid waste is mainly heterogenous in nature. Urban solid waste and some of the industrial waste materials possess heavy metals and pose toxicity concerns on the environment. Large concentrations of toxic compounds are present in sewage sludge (Lerch *et al.*, 1992) which could be metals and other persistent pollutants (Natal-Da-Luz *et al.*, 2009) which may be toxic to plants (Martinez and McBride, 2000). However little is known about the nature of enzyme industry wastes, their toxic or beneficial effects. Hence the first objective of this study was to investigate the effect of enzyme industrial wastes on germination of seeds to identify the beneficial or phytotoxic effects.

Once separated to its compostable fractions, urban solid waste can be composted to manage problems of disposal. Industrial wastes generated from an enzyme production based industry, being organic in its source of origin makes it ideal to be converted to compost in mixing with segregated urban solid waste. Composting is the most common method involved in disposal of these wastes which otherwise will be landfilled and the nutrients go unutilized and unexploited for agricultural use. The process of composting involves the decomposition of organic matter and decomposition of any organic material depends on many factors like the C:N ratio, oxygen, temperature and moisture level maintained during composting. The decomposition process of organic matter includes an initial rapid mineralization of added substrates and derived microbial cells followed by slower mineralization of stabilized microbial products and undecomposed materials (Voroney *et al.*, 1989). Respiration activity or oxygen

consumption and heat production are indicative of the degradable organic matter present in the compost and are inversely related to the stabilization. Laboratory studies to evaluate compost stability include these (Zucconi and de Bertoldi, 1987). Respirometric studies, which determine the O₂ consumption or CO₂ production caused by mineralization of the compost's organic matter, have been carried out in pure composts and in compost mixed with soil in a proportion compatible with agricultural use (Morel *et al.*, 1979; Iannotti *et al.*, 1993). Thus an understanding on the relative magnitude of C mineralization is essential to identify the stabilization of organic matter. Keeping in this view, an incubation experiment was carried out under laboratory conditions to understand the rate of decomposition of the compost.

Materials and Methods

Process involved in the production of industrial wastes: Multiple Effect Evaporator salts (MEES), primary sludge (PS) and filter press feed (FPF)

The enzyme industry basically uses wheat or barley as raw material. The endosperm is separated and subjected to treatment with various chemicals resulting in cell mass. Cell mass obtained from different organic sources is the main source for the industrial production of enzymes. This includes the process of fermentation of cell mass. Fermentation involves use of micro organisms, like bacteria and yeasts to produce the enzyme and is a common method of generating enzymes for industrial purposes.

During the process of enzyme production the cell mass is steam killed in the treatment plant. The steam killed product is separated out into solids and liquids. The solid obtained is termed as the ETP sludge or the primary sludge (PS). The liquid portion separated out

is treated with 0.05 per cent lime and other poly electrolytes to achieve coagulation. On coagulation, suspended solids are obtained. These suspended solids are pressed using a filter press and a solid portion separates out of it, which is referred to as the Filter Press Feed (FPF). The liquid portion obtained is subjected to three cycles of reverse osmosis to cause desalinization. At the end of reverse osmosis a concentrated liquid is obtained which is allowed through evaporator to reduce the water content. Water is then evaporated and a concentrated salt is obtained from the multiple effect evaporator system, which is referred to as the Multiple Effect Evaporator Salts (MEES).

These wastes were procured from an enzyme production based industry in Electronics city area of Bangalore district, Karnataka, India. These solid industrial waste samples were characterized for its total organic carbon content by dry combustion method.

Beneficial / Phytotoxicity studies

The industrial waste samples MEES, PS and FPF were tested under laboratory conditions to evaluate their phytotoxic or beneficial effects, if any. To test these for their phytotoxicity, water extracts were used. Industrial waste: water extracts (1:10) were prepared and analyzed for chemical constituents. These extracts were used to test the germination of selected seeds *viz.*, maize, finger millet, green gram and tomato. Germination sheets were used. Ten seeds were placed per sheet. The biosolid: water extract was applied to germination papers to maintain optimum moisture content. The germination percentage of seeds was recorded. Length of plumule and radicle was recorded to understand the nature and speed of growth on using the industrial waste- water extracts. The germination rate was calculated to evaluate the vigour of the seedlings. This was done by daily counting the

germinated seeds and determining the seedling vigour (Maguire, 1962). The mathematical expression used to calculate germination rate is given by

Rate= number of normal seedlings/ days to first count +...+ number of normal seedlings/ days to final count.

This was adapted from Throneberry and Smith (1955) that permits one to obtain the measurement for any intervals of time.

Incubation study to understand the evolution of CO₂ during decomposition of industrial waste composts

To utilize these industrial waste materials in agriculture, composting could be seen as a potential method. Composting could help in degrading the phytotoxic effects of the industrial waste materials as such (Bustamante *et al.*, 2008) and thus an attempt was made for the conversion of industrial wastes *viz.* multiple effect evaporator salts, primary sludge and filter press feed with urban solid waste as C source. To understand the rate of decomposition of industrial waste composts prepared by mixing urban solid waste with industrial wastes was determined in the laboratory by carbon-di-oxide evolution method. This rate of carbon di oxide evolution is also one of the indicators to assess compost maturity.

The treatment combinations include T₁: urban solid waste + MEE Salts, T₂: urban solid waste + primary sludge, T₃: urban solid waste + filter press feed and T₄: urban solid waste alone. The waste materials in each treatment combination were thoroughly mixed. A total of 200 g of industrial waste material were placed in one litre conical flask. Moisture was maintained at 60 per cent by adding distilled water. The flasks were closed with rubber cork and sealed with wax. These were incubated at

28° C. Carbon mineralized was determined by titrimetric method. A vial containing 10 ml of 2 M NaOH was placed in the flask with the help of thread, and flasks were sealed air-tight. The vials were taken out in 5, 10, 15, 20, 30, 40 and 50 days from the day of initiation of incubation study and titrated with standardized 0.5 N hydrochloric acid after addition of 1 ml of saturated barium chloride using phenolphthalein as indicator. The amount of C-CO₂ evolved was calculated as outlined by Wilde *et al.*, 1972. The samples were incubated for a period of 50 days under laboratory conditions.

Results and Discussion

Chemical properties of industrial waste: water extracts

Results in Table 1 indicate the chemical properties of industrial waste: water extracts. The highest pH was recorded in 1:10 dilution of PS: water (7.00). The extract from MEES: water recorded highest EC of 60 dSm⁻¹; nitrogen and potassium recorded 1.5 and 0.33 per cent, respectively. Zinc (2.8 mg kg⁻¹) was detected in 1:10 extract of MEES: water. The other micronutrients recorded lower levels. Nickel and cadmium were present in industrial waste: water extracts, whereas lead and chromium were not detected. Primary Sludge: water extract recorded a Ni content of 9.8 mg kg⁻¹ and Filter press feed: water extract recorded 9.7 mg kg⁻¹.

Germination test

The effect of industrial waste-water extracts on seed germination of maize, finger millet, green gram and tomato was determined. The percentage and rate of germination of seeds using water extract of industrial wastes is presented in Table 2. Control (tap water) recorded hundred per cent germination in two/three days in case of all seeds. Maize

seeds took four days to record 100 per cent germination compared to others which recorded 100 per cent germination in three days. The important observation here is that none of the seeds germinated in MEES: water extract. There was a noticeable inhibition of germination of seeds in 1:10 water extract of MEE salts compared to primary sludge, filter press feed. This inhibition of germination may be due to the presence of salts as evidenced by the high EC value of 60 dS m⁻¹ in case of 1: 10 MEES: water extract. Primary sludge: water and FPF: water extracts inhibited germination of seeds which is indicated by the lower germination percentage of tomato seeds on the fourth day, whereas the control recorded hundred percentage of germination. Tomato being a good indicator plant exhibited inhibition of germination due to industrial waste: water extract. This inhibitory effect was relatively higher in PS: water extract than filter press feed: water extract, which was also supported by the lower rate of germination (6.11) in PS: water extract than (7.34) FPF: water extract.

The control (tap water) recorded higher rate of germination than industrial waste: water extracts giving a clear indication of toxic effect of the industrial wastes. Germination rate as given by Maguire (1962) presented the germination rate for field or laboratory conditions to evaluate the seedlings vigour. The higher rates of germination mean the higher the seedling vigour of one sample in comparison to the other. In seed technology this value, named index of velocity of germination or emergence, is used to predict the relative vigour of samples, especially for cultivated species, since samples with the same quantity of seeds germinated can present different values for this index. Although Maguire (1962) had not presented the unit of this measurement, the value calculated using the expression proposed denotes a number of normal seedlings per day.

While conducting the germination test of seeds to understand the beneficial or phytotoxic effects if any, similar observations were recorded in the case of length of radicle as well as plumule (Table 3) of the seeds when placed in 1:10 PS: water extract and control (tap water). The length of radicle (5.79) and plumule (4.94) in case of tomato, an indicator plant, was relatively lesser in PS: water extract to that of FPF: water extract and control. Thus FPF: water extract recorded better elongation of radicle and plumule in case of tomato and green gram. Toxicity of the extract affects germination, therefore length of radicle and plumule was less in industrial waste: water extract in comparison to control.

Incubation study: rate of decomposition by CO₂ evolution

Carbon-di-oxide evolution is estimated to understand the rate of decomposition and the data on the amounts of carbon di oxide evolved at different intervals during decomposition are represented in Table 4.

The organic carbon content in urban solid waste + multiple effect evaporator salts, urban solid waste + primary sludge, urban solid waste + filter press feed and urban solid waste alone was 58.0, 57.0, 55.0 and 45.3 per cent respectively on incubation.

The carbon di oxide evolved during the fifth day in different treatments ranged from 9.35 mg CO₂ 100 gc⁻¹ day⁻¹ in treatment T₃ (urban solid waste+ filter press feed) to 20.36 mg CO₂ 100gc⁻¹ day⁻¹ in treatment T₁ (urban solid waste+ multiple effect evaporator salts). The treatment which received urban solid waste alone (T₄) evolved least amount of CO₂ (6.05 mg CO₂ 100 gc⁻¹ day⁻¹). During the tenth day, as well, evolution of carbon-di-oxide showed a similar trend. Maximum evolution of 19.58 mg CO₂ 100 gc⁻¹ day⁻¹ was observed in treatment T₁ (urban solid waste+ multiple

effect evaporator salts) and lower evolution of 5.72 mg CO₂ 100 gc⁻¹ day⁻¹ in treatment T₄ (urban solid waste alone). But, there was not much reduction in the amount of carbon-di-oxide evolved between 5th and 10th day. From the fifteenth day onwards a decrease was observed in the CO₂ evolved till the 50th day. At the end of the 50th day of incubation, the CO₂ evolution ranged from a minimum of 2.60 mg CO₂ 100 gc⁻¹ day⁻¹ in treatment T₄ (urban solid waste alone) to 6.10 mg CO₂ 100 gc⁻¹ day⁻¹ in treatment T₁ (urban solid waste+ multiple effect evaporator salts).

In general as the days of incubation increased a decreasing trend was noticed with respect to carbon-di-oxide evolution in all the treatments. The amount of CO₂ evolved was high in the initial stages of composting. This may be attributed to the high availability of carbon sources, which are high at the beginning of incubation and such compounds are readily utilized by the decomposers, resulting in microbial activity with higher evolution of CO₂. The enhancement of decomposition of organic matter in the initial stages could be due to the presence of soluble substances in the industrial waste-urban solid waste combination thus providing readily available source of energy for microbial growth and activity. The higher microbial activity helps in increased oxidation of carbon to carbon-di-oxide resulting in higher evolution. Similar results have been reported by Sarmah and Bordoloi (1994); Krishnamurthy *et al.*, (2010). The decreased evolution of CO₂ with time may be due to reduction in the amount of easily decomposable labile carbon compounds.

Among the treatments, T₁ (urban solid waste+ multiple effect evaporator salts) recorded the highest CO₂ production during initial periods followed by a gradual decrease over the period. All the decomposing systems showed decrease in the rate of CO₂ evolution, though

the fluctuations over time were less pronounced at later stages. This is mainly due to the differences in the bio-chemical compositions of the decomposing systems. If labile fractions are predominant they undergo rapid decomposition and will be evident during the initial decomposition period. Hence, larger fluctuations among treatments are expected during the initial period (Stevenson, 1982). The maturity of compost may also be assessed by CO₂ evolution studies. Insufficiently mature compost has a strong demand for O₂ and high CO₂ production rates due to intense development of microorganisms as a consequence of the abundance of easily biodegradable compounds in the raw material. For this reason, O₂

consumption or CO₂ production are indicative of compost stability and maturity (Hue and Liu, 1995). The data on the cumulative CO₂ evolution from different treatments at fixed intervals during 50 days of incubation are presented in Table 5. The cumulative CO₂ evolved over first ten days ranged from 11.77 mg CO₂ 100 gc⁻¹ in urban solid waste alone to 39.94 mg CO₂ 100 gc⁻¹ in treatment T₁ (urban solid waste+ multiple effect evaporator salts). The incubation of urban solid waste alone recorded lower cumulative CO₂ evolution until the end whereas higher cumulative CO₂ evolution was recorded in urban solid waste+ multiple effect evaporator salts and urban solid waste+ primary sludge treatments.

Table.1 Chemical characteristics of 1: 10 water extract of MEE Salts, Primary Sludge and Filter Press Feed used for germination test

Parameter	MEE salts	Primary Sludge	Filter press feed
pH	5.35	7.00	5.66
EC (dS m⁻¹)	60.00	3.00	1.00
Nitrogen (%)	1.52	0.08	0.02
Phosphorous (%)	0.004	0.003	0.00
Potassium (%)	0.326	0.006	0.00
Sodium (%)	0.036	0.018	0.019
Sulphur (%)	0.016	0.009	0.002
Micronutrients (mg kg ⁻¹)			
Iron	ND	ND	ND
Manganese	0.82	0.92	0.07
Zinc	2.78	ND	ND
Copper	ND	ND	ND
Heavy metals (mg kg ⁻¹)			
Nickel	1.11	9.82	9.74
Cadmium	0.80	0.76	0.79
Lead	ND	ND	ND
Chromium	ND	ND	ND

ND: Not Detected

Table.2 Per cent and rate of germination of seeds as influenced by 1:10 water extract of biosolids

Treatment	Maize						Rate	Finger millet						Rate	Green gram					Rate	Tomato						Rate
	%							%							%						%						
	1	2	3	4	5	6		1	2	3	4	5	6		1	2	3	4	5		1	2	3	4	5	6	
	Days							Days							Days						Days						
Control (tap water)	-	40	60	100	100	100	10.16	-	60	100	100	100	100	12.43	-	60	100	100	100	12.43	-	40	70	100	100	100	10.43
MEES water extract (1:10)	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
PS water Extract (1:10)	-	-	76	85	100	100	8.33	-	-	100	100	100	100	9.43	-	-	100	100	100	9.43	-	-	34	55	100	100	6.11
FPF water Extract (1:10)	-	-	65	100	100	100	8.27	-	-	100	100	100	100	9.43	-	-	100	100	100	9.43	-	-	56	75	100	100	7.34

Table.5 Effect of different treatments on carbon dioxide evolution (cumulative) at different intervals of incubation

Treatments	Cumulative CO ₂ evolution (mg CO ₂ 100 gc ⁻¹)						
	Days after incubation						
	5	10	15	20	30	40	50
T ₁	20.36	39.94	54.13	64.41	73.92	83.38	89.48
T ₂	10.23	18.26	26.08	33.29	39.57	45.28	50.28
T ₃	9.35	17.69	24.62	30.55	35.60	40.10	43.43
T ₄	6.05	11.77	16.81	21.69	26.08	29.67	32.27

T₁: Urban solid waste + MEE Salts
 T₂: Urban solid waste + Primary sludge
 T₃: Urban solid waste + Filter press feed
 T₄: Urban solid waste alone

Table.3 Length of radicle and plumule as influenced by 1:10 water extract of biosolids

	Maize						Finger millet						Green gram						Tomato					
	Days						Days						Days						Days					
	1	2	3	4	5	6	1	2	3	4	5	6	1	2	3	4	5	6	1	2	3	4	5	6
	Length of radicle (cm)																							
Control (tap water)	-	1.00	1.4	1.50	10.00	16.00	-	1.11	2.50	4.52	6.00	7.50	-	1.60	2.00	4.55	6.85	12.10	-	0.60	1.00	2.50	4.50	6.50
MEES water extract (1:10)	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
PS water extract (1:10)	-	-	1.56	5.90	10.65	16.68	-	-	1.02	2.12	5.60	7.78	-	-	1.55	3.40	5.45	7.65	-	-	0.50	2.46	4.40	5.79
FPF water extract (1:10)	-	-	1.11	4.24	09.34	14.75	-	-	0.63	1.67	5.20	7.20	-	-	2.23	5.57	7.89	14.17	-	-	0.60	2.56	4.33	6.32
	Length of plumule (cm)																							
Control (tap water)	-	0.69	1.24	4.50	10.50	15.00	-	0.87	0.90	1.50	1.60	4.50	-	0.50	0.80	5.68	8.86	12.56	-	0.59	0.96	2.50	4.00	6.26
MEES water extract (1:10)	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
PS water extract (1:10)	-	-	0.60	4.57	10.56	15.00	-	-	0.82	1.90	2.35	5.28	-	-	0.50	5.80	8.89	12.84	-	-	0.58	1.25	3.50	4.94
FPF water extract (1:10)	-	-	0.50	4.00	9.98	14.39	-	-	0.78	1.00	1.56	2.03	-	-	0.50	5.90	9.00	13.94	-	-	0.75	2.00	4.50	5.80

Table.4 Effect of different treatments on carbon dioxide evolution at different intervals of incubation

Treatments	Carbon dioxide evolved (mg CO ₂ 100 gc ⁻¹ day ⁻¹)						
	Days after incubation						
	5	10	15	20	30	40	50
T ₁	20.36	19.58	14.19	10.29	9.51	9.46	6.10
T ₂	10.23	8.03	7.82	7.21	6.28	5.71	5.00
T ₃	9.35	8.34	6.93	5.93	5.05	4.50	3.33
T ₄	6.05	5.72	5.04	4.88	4.39	3.60	2.60

The higher rate of evolution of carbon-dioxide could be due to the increased decomposition in these systems. Higher carbon-di-oxide releases may be observed due to the microbial attack on easily degradable organic fractions still present in the mixture, in case the samples of the composting mixture are poorly transformed through the biostabilisation process (Garcia-Gomez *et al.*, 2003). This also could be indicative of the more mature nature of urban solid waste alone than the other systems of combinations of urban solid waste and industrial wastes. The mature samples record lower values of C mineralization and compost much below the desired advanced degree of maturation result in more C mineralization than 25 per cent of total organic carbon (Bernal *et al.*, 1998).

In general an increasing trend in cumulative carbon di oxide evolution was observed in all the treatments. During the last week of decomposition period, the cumulative CO₂ evolved ranged from 32.27 mg CO₂ 100 gc⁻¹ in treatment T₄ (urban solid waste alone) to 89.48 mg CO₂ 100 gc⁻¹ in treatment T₁ (urban solid waste+ multiple effect evaporator salts). This higher cumulative carbon-di-oxide evolution in all the other treatments than urban solid waste alone could be attributed to the combination of industrial waste and urban solid waste and their rate of decomposition. Tester *et al.*, (1977) studied the rate and extent of decomposition of sewage sludge compost mixed with soils and reported that the cumulative carbon dioxide evolution was linearly related to the rate of sludge compost applied.

Release and availability of plant nutrients is an index of decomposition of the added organic substrate. Microbial respiration and rate of release of nutrients are directly related. Rate of microbial respiration may be reflected in terms of rate of carbon dioxide evolved. Depending on the factors influencing the rate

of decomposition of organic material, the pattern of evolution of carbon dioxide changes as time lapses. Bangar and Patil (1980) also opined that incubation period had a significant role on carbon dioxide evolution.

The rate of CO₂ evolution is usually employed to measure the decomposition of organic materials. Though several techniques are available to measure the rate of decomposition, the method of Pramer and Schmidt (1964) was used. It is a closed system and may not be simulating the decomposition rate taking place in bigger heaps of organic materials under field conditions.

In conclusion, this study evaluated the toxic effects of enzyme industrial wastes on plants under laboratory conditions. It was found that the enzyme industrial wastes possess phytotoxic effects which were noticed in the inhibition on seed germination as well as the reduction in per cent and rate of seed germination, radicle and plumule elongation indicating reduced vigour of seedlings. It was also studied to understand the rate of decomposition on composting these waste materials along with urban solid waste as the C source. It was found that the rate of decomposition was higher in industrial waste-urban solid waste combination exhibiting the less mature and unstable nature of the studied system. The time taken for decomposition under laboratory conditions may vary in the field conditions as several factors act upon the oxidation of C to carbon-di-oxide. The decomposed and final composts must be checked for phytotoxic effects before its recommendation to agricultural use as mature composts usually are free from causing phytotoxicity. However, further studies are required to understand the effects of composts on seed germination as well as seedling growth.

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