

AEROBIC COMPOSTING OF TOBACCO SOLID WASTE

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Abstract

Solid waste generated by tobacco industry contains mostly tobacco particles and flavoring agents. Its main characteristics are high contents of nicotine, which is toxic, and total organic carbon in the aqueous extract. Therefore, tobacco waste must be separated from urban waste. The aim of this work was to stabilize tobacco solid waste by aerobic composting. The experiments were carried out in the closed thermally insulated column reactors (1 L and 25 L) under adiabatic conditions over 16 days. Monitoring covered temperature changes in the reactor, CO₂ production and the numbers of mesophilic and thermophilic population in the mixed microbial culture. During the process the highest temperature was 64 °C, pH varied between 7.85-9.25 and moisture content in composting mass was between 65.8-76.5%. By the end of the experiment total solids in tobacco waste were reduced by approximately 50% and nicotine by 90%. Biodegradation rate of organic fraction in solid waste was simulated using the empirical model. It was found out that the selected model describes aerobic composting fairly well and confirms the hypothesis that the released heat is proportional to the biodegradation process.

Introduction

Solid waste, such as food, yard, agricultural and industrial waste, and waste paper are clogging the landfills.¹ Annual production of household waste in Croatia is around one million tons, and with only a few modern landfills available, majority of waste is still disposed of in dumps. There is no accurate information about total volume of industrial and hazardous waste. In few municipal landfills in Croatia, hazardous and municipal waste used to be disposed of together.² Currently, industrial solid waste with total organic carbon (TOC) in aqueous fraction above 200 mgL⁻¹ cannot be disposed of in sanitary landfills³ and is either incinerated or composted into a humus-like product.

Tobacco industry generates large volume of non-recyclable tobacco powdery waste. Tobacco solid waste is classified as agro-industrial waste. Its TOC level in aqueous fraction being in excess of 200 mgL⁻¹ it cannot be disposed of in sanitary landfill. Moreover, its nicotine content⁴ makes it hazardous. Aerobic composting is one of the ways to reduce accumulation of industrial solid waste and to detoxify it.⁴⁻⁷ Organic

fraction of solid waste can be degraded either in nonreactor system or reactor system. The advantages of the closed vessels or reactor systems over nonreactor systems are in the better control of composting conditions and the emissions of air pollutants and odor.^{8,9} Composting generates a stable product, the source of organic matter for humus that gives soil its proper structure and moisture holding capacity. Compost may contain valuable nutrients, but it depends on quality of the original organic substrate.

This work was aimed at investigating aerobic composting of tobacco solid waste in the laboratory-scale column reactor and proposing the reactor and kinetic model that represents the experimental results.

Experimental

Reactor system and experimental run

Laboratory-scale aerobic composting was performed in the closed thermally insulated column reactors¹⁰ of 1.0 L and 25.0 L effective volume. Tobacco solid waste (Figure 1) contained tobacco powder and particles and 4% of flavoring agents (sugars, humidifiers, organic acids and fruit extracts). Its initial pH value was 7.9, C/N ratio 15.1, moisture content 65.8%, volatile solids (VS) amounted 68.5% of total solids (TS) and nicotine 2000 mgkg⁻¹ of TS.



Figure 1. Tobacco solid waste before composting.

The reactors were filled with 0.5 kg and 12.5 kg of the composting mass respectively and operated at airflow rate of 0.9 L min⁻¹ kg⁻¹VS. Continuous upward aeration was provided by an air blower. Prior to entering into the reactor, air was saturated with moisture by passing through Drechsel bottle. That ensured permanent air

humidity at the reactor's inlet and outlet. Temperature had been monitored by thermocouples during 16 days. The exhaust hot air with containing carbon dioxide was cooled and the condensate was collected in a graduated cylinder. Before being released into the atmosphere, CO₂ was collected in a tube filled with solid KOH as an adsorbent. Blind test was carried out to determine total amount of carbon dioxide released into the reactor during 16 days of aeration. All experiments were doubled.

Physico-chemical and microbiological analyses

Air temperature at the reactor inlet and outlet and at three levels of the composting mass (bottom, middle and near the surface) had been monitored continuously by the thermocouples connected to the data logger. Airflow rate was measured by air flow meter (0.1-0.5 LPM and 0.2-2.0 LPM, Cole-Parmer). The graduated cylinder measured daily production of condensate. CO₂ content in the exhaust air was measured by gravimetric method¹¹ and the result expressed as g CO₂ day⁻¹. Key parameters of the original waste mixture and the compost were analyzed at the beginning and during composting tests (pH value, moisture content in the percentage of wet weight, total and volatile solids and total Kjeldahl nitrogen). Analytical methods were the standard ones for compost analysis.¹² Colony-forming units (CFU) of mesophilic and thermophilic bacteria and fungi were determined on the general-purpose media (nutrient agar for bacteria and malt agar for fungi) by pour plate method.¹³ For the plate count a dilution series (0.9% NaCl) was made from each sample. The plates were incubated under 80% relative humidity at 28 °C for the growth of mesophilic fungi, at 37 °C for the growth of mesophilic bacteria, and at 50 °C for the growth of thermophilic microorganisms. The results were expressed as CFU of mesophilic and thermophilic bacteria and fungi per gram of the composting mass. Different microorganisms in composting mass were distinguished according to their colony forms on agar plates. The results were verified by optical microscopy after methylene blue staining, Gram staining and Schaeffer-Fulton spore staining.¹³ Microbial species isolated as pure cultures were further identified by API 20 E, API 20 NE, API CHB and API 20 C AUX systems (bioMerieux, Marcy-l'Étoile, France). HPLC method¹⁴ with diode-array detector was used to measure the content of nicotine in initial solid waste and at the end of composting (Merck-Hitachi, L-4500 Column RP-18).

Results and discussion

Composting process

For the efficiency of composting in the controlled reactor system it is necessary to know the reaction rate of biological degradation in the composting mass and the level of degradation achieved. Therefore, composting of tobacco solid waste was monitored during 16 days and experimental results presented in Figures 2-4. Temperature variations in both reactors in double sets were recorded to be similar. The lag period on temperature curve (Figure 2) was not recorded because the original substrate was rich in microorganisms.

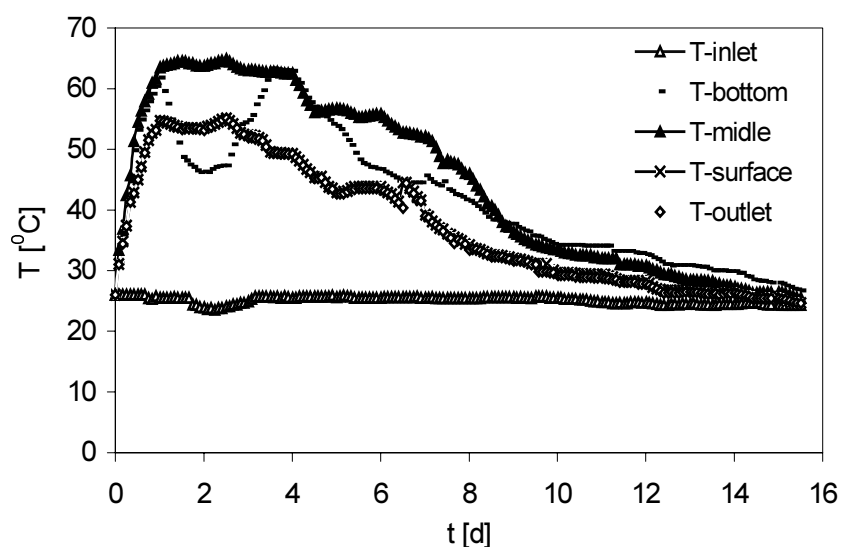


Figure 2. Temperature over time during 16 days of composting.

Thus, after several hours the temperature in the composting mass started to rise due to intense biodegradation. It reached maximum after day one, remained high till day four and then gradually decreased till day nine. After that the decrease became more and more marked, until the compost and ambient temperatures leveled. As shown on the curve for CO_2 (Figure 3), its formation followed the pattern of temperature curve because initially mesophilic microorganisms used most readily degradable organic compounds, which generated carbon dioxide, and caused abrupt temperature increase. It was estimated that at the end of composting 415 g of CO_2 evolved per each kilo of total solids.

Microbial succession during composting is known to be relatively rapid, since it may take 1-2 days to reach 55 °C. At the beginning of the experiment the substrate had the ambient temperature, mesophilic bacteria and fungi (Figure 4) dominated and the prevailing isolates were two bacteria *Bacillus subtilis* and *Pseudomonas aeruginosa* and two yeasts *Candida* sp. and *Saccharomyces cerevisiae*. Simple organic compounds in tobacco solid waste caused multiplication of microorganisms and sharp temperature increase to 64 °C. The increased temperature led to the reduction of mesophilic microorganisms. Further substrate biodegradation was taken over by thermophilic microorganisms.

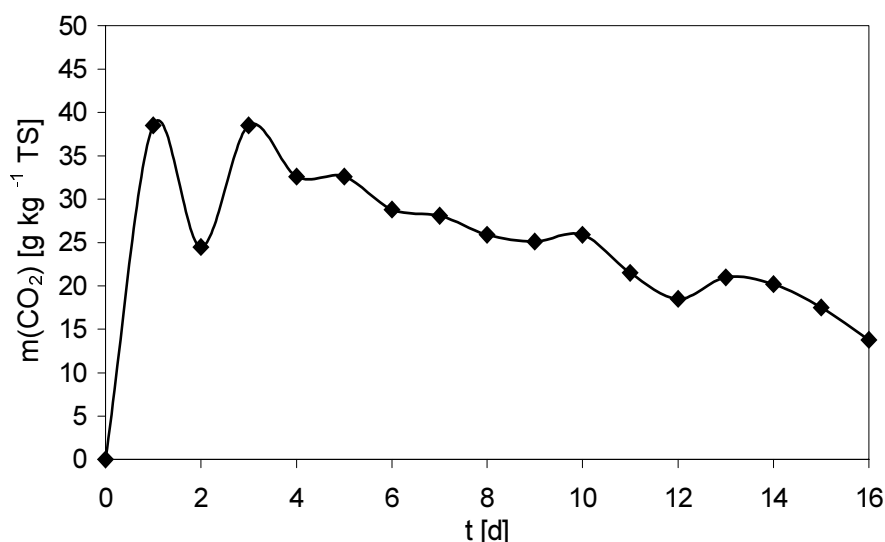


Figure 3. Mass of produced CO₂ due to substrate biodegradation.

Presumably, at this phase thermophilic microorganisms overtook a role in biodegradation of cellulose and other organic macromolecules in the substrate.¹³ This process was relatively slow, and decreased the rate of heat generation, and, thus, the temperature. At that stage *Bacillus subtilis* and aerobic gram-positive thermophilic actinomycetes were dominant strains in the composting mass. The latter strain had pale yellow colonies, grew at 30 °C and 55 °C, and had the spores that were killed at 80 °C after 5 min treatment in aqueous suspension.¹³ Simultaneously, the microbial community contained the cells of *P. aeruginosa*, *S. cerevisiae* and *Candida* sp. though in a small number.

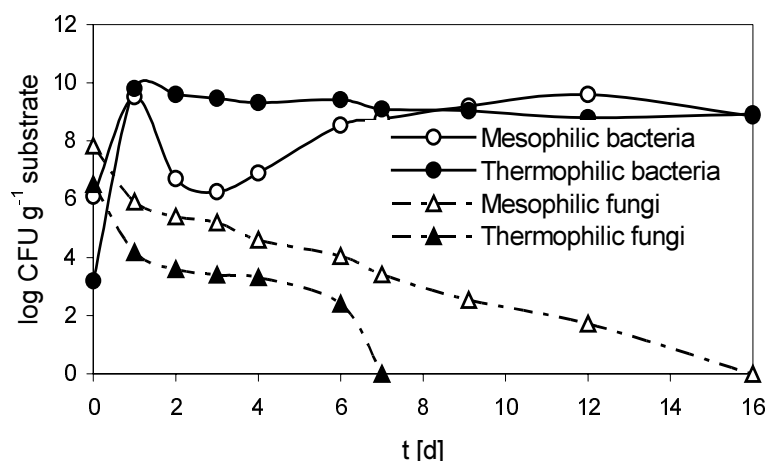


Figure 4. Bacterial and fungal growth during composting.

After seven days at about 47 °C, mesophilic bacteria were re-activated and grew again. Bacterial population remained high throughout further 9 days, during which the temperature gradually declined until the compost reached the ambient temperature. In contrast to that, yeast cells declined gradually and by the end of the process they entirely diminished from the compost.

Moisture content and volatile solids in tobacco solid waste at the beginning of the experiment were 65.8% and 68.5% respectively. At the end of composting they came to 76.5% and 39.3% respectively. Total condensate yield after 16 days was 80.2 mL and 800 mL, depending on the reactor volume and as the result of biodegradation of volatile solids and water production. Figure 5 shows raw compost. Analytical results showed that C/N ratio dropped from 15.1 to 9.8 and nicotine content from 2000 mg kg⁻¹ to 211 mg kg⁻¹ of total solids. It was supposed that *Pseudomonas aeruginosa* was responsible for nicotine biodegradation, because of its permanent presence in the mixed microbial culture of the composting mass throughout composting.⁴

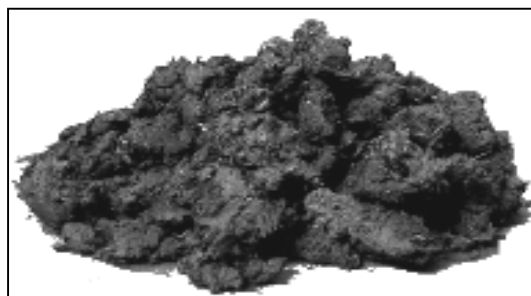


Figure 5. Raw compost after 16 days of composting.

It was estimated that 51% of the total solids biologically degraded after 16 days, what was in agreement with literature data.⁸ Similar results were obtained in all experiments. Maturation of raw compost proceeded in the open box for another 30 days, during which period moisture dropped to 38.4%.

Modeling of the composting process

Laboratory scale bioreactor was one of the unsteady state column reactors. Substrate biodegradation was slow compared to the airflow rate and oxygen transfer through the boundary gas layer. It was assumed, therefore, that the change between inlet and outlet air oxygen concentration was very small. Hence, oxygen level inside the column was practically equal at all points. Accordingly, several assumptions were taken into account in developing the model: the process was carried out under adiabatic conditions; air flow rate during composting was constant; with constant humidity, and composting rate was formally expressed as degradation rate of the dry substrate r_S ,



The following expression was used, Eq. [1]

$$r_S = f(w_S, T) \quad [1]$$

where w_S denotes mass fraction of substrate and T temperature in reactor during composting.

As biodegradation rate is known to be a slow process, it was assumed to be uniform all over the bed of the composting mass. Consequently, the reactor could be modeled as continuous stirred reactor (CSTR). Two balances are needed – one for the composting mass and one for overall heat balance. The material balance for dry composting mass (m_{S0}) in the reactor was expressed by Eq. [2]

$$r_S = -\frac{dw_S}{dt} = -\frac{dm_S}{dt} \frac{1}{m_{S0}} \quad [2]$$

The heat balance equation, Eq. [3], took into account that the process was carried out under adiabatic conditions

$$m_o c_{pS} \frac{dT}{dt} = (-\Delta H_r) m_{S0} r_S - \rho_z Q_v c_{pz} (T - T_u) \quad [3]$$

Real reaction rate dependence as a function of all intrinsic variables was unknown. Based on the temperature - time curve shape, a rather complicated empirical kinetic model is proposed,

$$r_S = k(T)[w_S e^{-nt} + w_S^m] \quad [4]$$

where n and m are the adjustable parameters.

Constant k in Eq. [4] was generally temperature-dependent. Eqs. [2-4] acted as the reactor's basic model in the composting process. For application of the model and its experimental verification, several parameters and process variables were required. Table 1 shows necessary data for application of the proposed model.

Table 1. Dimensions and characteristics of process.

Mass of wet substrate, m_o	11.5 kg
Mass of dry substrate (TS), m_{So}	3.9 kg
Air flow rate, Q_v	0.9 L min ⁻¹ kg ⁻¹ VS
Heat capacity of air, c_{pz}	1.01 kJ kg ⁻¹ K ⁻¹
Heat capacity of mass in the reactor, c_{pS}	3.58 kJ kg ⁻¹ K ⁻¹
Air density, ρ_z	1.3 kg m ⁻³
Inlet air temperature, °C	24-26 °C

In addition to these given variables, it was necessary to experimentally calculate the reaction enthalpy and select the kinetic model. The reaction enthalpy was calculated from experimental results, by measuring the composting mass temperature during the reaction. It was assumed that the process had been carried out under adiabatic conditions and that the released heat was proportional to the progress of biodegradation. Eq. [2] was combined with Eq. [3] to yield

$$m_o c_{pS} \frac{dT}{dt} = (-\Delta H_r) m_{So} \left(-\frac{dw_S}{dt}\right) - \rho_z Q_v c_{pz} (T - T_u) \quad [5]$$

and

$$c_{pS} dT = \frac{m_{So}}{m_o} (-\Delta H_r) dw_S - \frac{\rho_z Q_v c_{pz}}{m_o} (T - T_u) dt \quad [6]$$

and at boundary conditions $t = 0$: $T = T_o$, $w_S = 0$ and $t = t_k$: $T = T_k$, $w_S = 1$

$$\int_{T_o}^{T_k} c_{pS} dT = -\frac{m_{So}}{m_o} (-\Delta H_r) \int_0^1 dw_S - \frac{\rho_z Q_v c_{pz}}{m_o} \int_0^{t_k} (T - T_u) dt \quad [7]$$

after integration and re-arrangement we obtain:

$$(-\Delta H_r) = \frac{\rho_z Q_v c_{pz}}{m_{So}} \int_0^{t_k} (T - T_u) dt + c_{pS} \frac{m_o}{m_{So}} (T_K - T_0) \quad [8]$$

At the end of reaction, temperature of compost became equal to ambient temperature,

$$(-\Delta H_r) = \frac{\rho_z Q_v c_{pz}}{m_{So}} \int_0^{t_k} (T - T_u) dt \quad [9]$$

If the reaction had finished before the complete substrate degradation, Eq.[9] would have been slightly different,

$$(-\Delta H_r) = \frac{\rho_z Q_v c_{pz}}{m_o w_{Sk}} \int_0^{t_k} (T - T_u) dt \quad [10]$$

where w_{sk} is mass fraction of dry substrate obtained at the end of composting.

Calculated reaction enthalpy is given in Table 2. The obtained value for the selected substrate was in the range of the literature^{5,15} and took into account the percentage of biologically degradable volatile solids. Considering that the rates of released heat and the generated CO₂ were proportional to biodegradation rate, the conversions could be calculated from the proposed model. To estimate the dependence of the reaction rate on temperature, the expression taken from the literature^{5,16} was applied

$$k = k_0 [1,066^{T-20} - 1,21^{T-60}] \quad [11]$$

Kinetic parameters were estimated in Eq. [4] using known input variables from Table 1, calculated reaction enthalpy and the proposed rate-temperature dependence (Eq. [11]). Direct nonlinear regression analysis based on the Nelder-Mead simplex method was performed to determine k_0 , m and n in the kinetic model (Table 2). The model was fitted taking into account all data, i.e. experimental temperature, generated CO₂ during composting and substrate conversion. The experimental substrate conversion was obtained by measuring total solids reduction in the composting mass at given time times: at the start and after days 1, 5, 7, 10 and 16. After that, the difference in TS masses (g) at the start and at the specified time was divided by the starting TS mass (g). The result represented the substrate conversion (dimensionless) at the given time point. Mass fraction of CO₂ was calculated similarly, by dividing the masses of generated CO₂ at the given time by the total mass obtained in the composting process taking into account the

fact that the mass of CO₂ obtained in the process is proportional to the final conversion of substrate. When optimal values of kinetic parameters were determined, the set of differential equations (Eqs. [2-4]) describing the system, were numerically solved using Runge-Kutta method. The theoretical curve was drawn together with the actual data plot in Fig. 6. It shows the agreement between the measured and calculated temperatures throughout composting. The graphical presentation also shows that theoretical substrate conversion is in agreement with experimentally obtained conversion and mass fraction of generated CO₂.

Table 2. Results of estimated parameters

Reaction enthalpy, ΔH_r	-7513.2 kJ kg ⁻¹ TS
Parameter, m	1.9
Parameter, n	0.034
Constant k_0	0.024 h ⁻¹
Mean square deviation, SD	0.2035

The values of estimated parameters suggested good selection of the proposed composting model, although only three kinetic parameters (k_0 , m and n) were estimated.^{17,18}

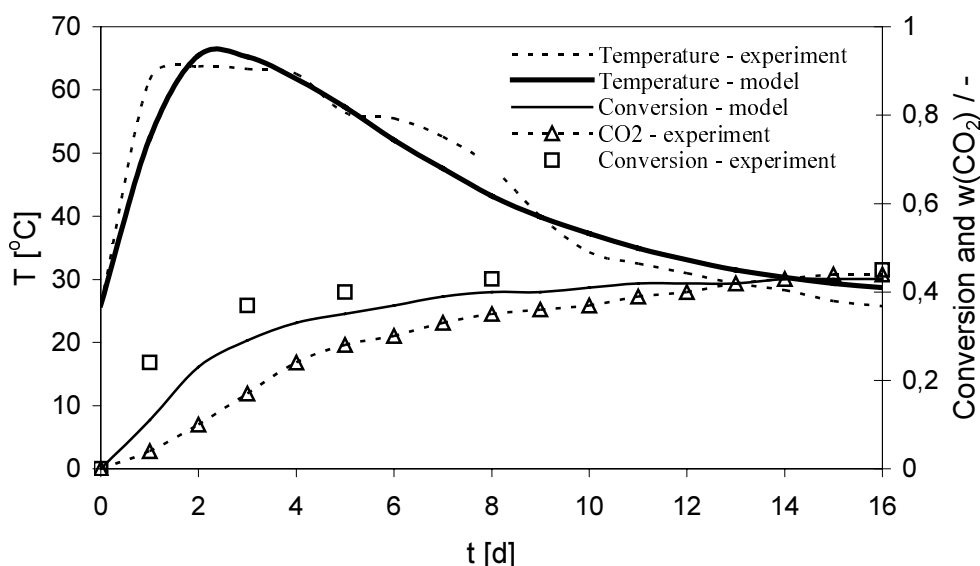


Figure 6. Comparison of temperature curve, mass fraction of CO₂ and substrate conversion obtained in the experiment and by modeling the process

Overall behavior of composting is very interesting, taking into account all factors influenced by the complexity of the process, i.e. either by the concurrent growth of different species of microorganisms (bacteria and fungi) or the volume and kind of a biodegradable substrate. The kinetic model is presented by empirical equation bearing in mind that most processes follow the exponential rate law. The reaction enthalpy is calculated from the experimentally obtained dependency of temperature vs. time, but the procedure cannot show possible changes in its value during composting.

Therefore, the analysis of the reaction enthalpy, calculated at different composting periods can be valuable for identifying the reaction sequences and possible mechanisms. The kinetic model is proposed for one reaction only. It is valid throughout the entire composting and its parameters are unchanged (k , k_o , m and n). By splitting duration of the process into time intervals and taking into account just the time between two adjacent conversion measurements (based on the loss of dry organic mass) we got five distinct time intervals, after day 1, 5, 7, 10 and 16. The reaction enthalpy at every interval was calculated using Eq. [12],

$$(-\Delta H_r)_i = \frac{1}{m_{So}(w_{S,i} - w_{S,i-1})} \left[m_o c_{ps} (T_i - T_{i-1}) + \rho_z Q_v c_{pz} \int_{t_{i-1}}^{t_i} (T - T_u) dt \right] \quad [12]$$

The calculated enthalpies are shown in Table 3. Obviously, they were not the same in all periods, but the mean was very close to the overall reaction enthalpy, obtained from all experimental data for the entire composting period.

Table 3. Enthalpies based on the time intervals during composting.

Time interval / day	X_A	$X_{A,i} - X_{A,i-1}$	$(-\Delta H_r)_i / \text{kJ kg}^{-1}$	Temperature interval / °C	$T_i - T_{i-1}$
0 – 1	0.21	0.21	- 1902.78	26.8 – 61.6	35.8
1 – 5	0.37	0.16	- 6749.18	61.6 – 56.4	5.2
5 – 7	0.42	0.05	- 5947.25	56.4 – 52.6	3.8
7 – 10	0.43	0.01	- 9130.54	52.6 – 34.3	18.3
10 – 16	0.45	0.02	- 11373.22	34.3 – 25.7	8.6
$(-\Delta H_r)$ obtained over the entire interval:			- 7513.2 kJ kg ⁻¹		

Clearly, during substrate biodegradation, there was a series of consecutive and parallel reactions of individual rate and mechanism each, depending on many aforementioned factors. It was very complicated to perform a single analysis and

verification (chemical and/or biochemical) with respect to given all relevant factors that changed the number of potential reactions and their mechanism.

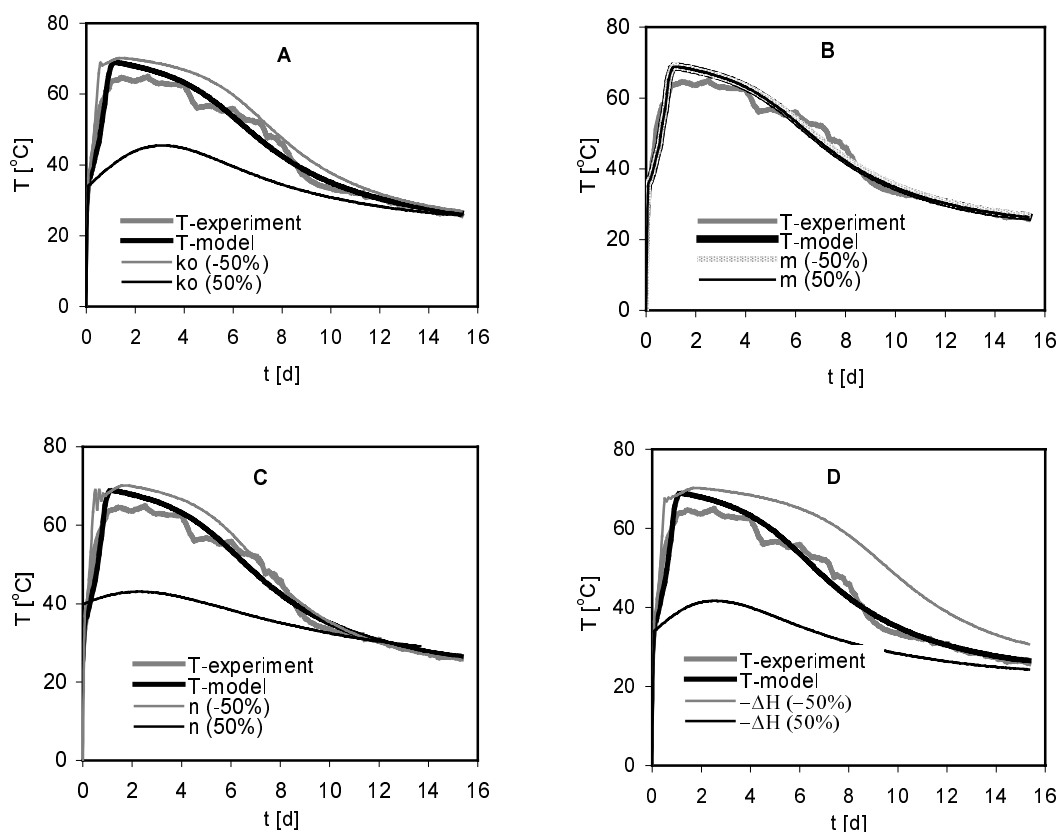


Figure 7. Sensitivity analysis by changing the parameters k_o (A), m (B), n (C) and $(-\Delta H_r)$ (D).

On the basis of such analysis it can be concluded that at the beginning of composting, we achieved rapid increase of conversion, evidenced by the high amount of evolved carbon dioxide, and the calculated reaction enthalpies were below the overall value. Conversely, over next six days the enthalpy sharply increased, whereas the conversion was low. From day seven to day sixteen the enthalpy reached its peak although the conversion remained practically unchanged (from day five to sixteen the difference was as low as 3%). For that reason, the apparent reaction enthalpy in the final stages of composting exceeded mean value. It was evident that the oxidation then was not the main exothermic reaction, and that other types of biochemical reactions occurred, not leading to complete degradation of the organic substrate to water and carbon dioxide. Further investigations should be conducted to confirm our assumptions, such as

composition of the exit gases or final organic matter. It must be noted that during composting, various biochemical reactions took place, all of which were time dependent as concerned development of individual classes of microorganisms. Finally, the model of composting process derived from the simple assumption of a single kinetic model over the entire composting time was valuable in the studies of the reactors design for real life application.

Sensitivity of the model parameter was checked by changing the values of k_0 , m , n , and $(-\Delta H_r)$. Fig. 7(A-D) shows the effect of the parameter change by $\pm 50\%$ of its estimated optimal value. The parameter $(-\Delta H_r)$ exhibited the most pronounced effect due to its strongest influence on the reaction rate.

In Table 4 results of such analysis are presented. As can be seen, temperature-time history is influenced by the reaction enthalpy and by kinetic parameters k_0 and n .

Table 4. Results of parameter sensitivity test.

Parameter	SD _p	SD _p – SD _{opt}
$m + 50\%$	0.1937	$1.39 \cdot 10^{-3}$
$m - 50\%$	0.1906	$6.05 \cdot 10^{-4}$
$n + 50\%$	0.5179	0.3279
$n - 50\%$	0.9555	0.7655
$k_0 + 50\%$	0.3419	0.1520
$k_0 - 50\%$	0.9155	0.7255
$(-\Delta H_r) + 50\%$	0.7283	0.5384
$(-\Delta H_r) - 50\%$	1.1095	0.9196

The results obtained in this research showed that the empirical model described aerobic composting fairly well and confirmed the hypothesis that the released heat and mass of generated carbon dioxide were proportional to the biodegradation progress.

Conclusion

The results obtained in this contributory work to aerobic composting in a column reactor showed good reproducibility. The temperature above 50 °C was reached after day one and maintained between 64 °C and 50 °C for seven days, favoring microbial activity in the composting mass. Changes in the number of mesophilic and thermophilic population of the mixed microbial culture had been monitored at different stages of composting.

After 16 days, the volume and the mass of total solids in tobacco waste were reduced by about 50%. It was also recorded that nicotine content in solid waste was successfully reduced and nicotine was biodegraded by approximately 90%. This suggested that composting successfully detoxifies tobacco solid waste. According to C/N ratio (9.8:1) in stabilized product, this type of compost may be used as fertilizer.

The advantage of closed reactor system over the open one is in that it more rapidly stabilizes the organic waste. Furthermore, generation of air polluting CO₂ and of the unpleasant odors during composting is much more easily controlled.

Degradation percentage of organic solids can be reliably predicted from the proposed model and presents basis for future studies of the reactor design. Evaluation of the experiment suggested good selection of the proposed composting model, although only three kinetic parameters (k_o , m and n) were estimated.

List of symbols

c_{ps}	specific heat capacity of the composting material, $\text{kJ kg}^{-1} \text{K}^{-1}$
c_{pz}	specific heat capacity of air, $\text{kJ kg}^{-1} \text{K}^{-1}$
i	time interval (1-5) in Eq. [12]
k	specific rate, Eqs[4] and [11], h^{-1}
k_o	constant in Eq. [11], h^{-1}
m_o	mass of wet substrate, kg
m_{So}	mass of initial dry substrate, kg
$m \text{ CO}_2$	mass of CO ₂ , kg
$m \text{ CO}_{2k}$	mass of CO ₂ at the end of composting, kg
m and n	parameters in Eq. [4]
Q_v	airflow volume, $\text{m}^3 \text{h}^{-1}$
r_s	degradation rate, $\text{kg kg}^{-1} \text{h}^{-1}$
SD	mean square deviation
SD _{opt}	mean square deviation (with estimated parameters)
SD _p	mean square deviation (with changed parameter)
t	time, h
T	temperature in reactor, °C
T_o	temperature of substrate at the beginning of reaction, °C
T_K	temperature of compost at the end of reaction, °C
T_u	temperature of air at the reactor inlet
X_A	conversion of dry substrate
w_s	mass fraction of substrate, $\text{m}_s \text{m}_o^{-1}$, kg kg^{-1}
w_{Sk}	mass fraction of substrate at the end of composting, $\text{m}_s \text{m}_o^{-1}$, kg kg^{-1}
w_{CO_2}	mass fraction of CO ₂ , $m_{CO_2} m_{CO_2k}^{-1}$, kg kg^{-1}
ΔH_r	reaction enthalpy, kJ kg^{-1} of dry substrate
ρ_z	air density, kg m^{-3}
τ	space time, day

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Povzetek

Trdni odpadki iz tobačne industrije vsebujejo toksični nikotin, v vodnem ekstraktu pa organski ogljik, zato se morajo ločevati od komunalnih odpadkov. Namen raziskav je bila stabilizacija teh odpadkov s kompostiranjem. Med poskusi, ki so bili izvajani 16 dni v laboratorijskih adiabatnih reaktorjih, so bili zasledovani temperatura, nastajanje CO₂, ter število mezofilnih in termofilnih mikroorganizmov. Dosežena je bila najvišja temperatura 64 °C, pH območje 7,85-9,25 in vlažnost 65,8-76,5. Ob koncu poskusa sta se vsebnosti trdne snovi v odpadku zmanjšali za 50%, nikotina pa za 90%. Biodegradacija organskih razgradljivih snovi je bila simulirana z matematičnim modelom, ki je zadovoljivo sovpadal z eksperimentalnimi rezultati in potrdil hipotezo, da je sproščena toplota proporcionalna procesu biodegradacije.