

Drying characteristics of soil in a microwave environment

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Vaitekunas, D., Raghavan, G. S. V. and van de Voort, F. R. 1989. **Drying characteristics of a soil in a microwave environment.** Can. Agric. Eng. **31**: 117-123. A system to monitor mass and temperature of soil samples during drying in a microwave environment is described. The drying characteristics of a clay, sandy loam and sand were observed. Mass-time and temperature-time curves indicated that the evaporation and release of water from a small volume of soil behaves according to a theoretical three-part sequence dictated by thermodynamic principles: (1) constant temperature rise without mass loss; (2) constant mass loss without temperature rise and; (3) rising temperature with exponentially decaying mass loss. Time to transition points and drying efficiency are discussed with respect to microwave power setting, soil type and initial moisture content.

INTRODUCTION

Microwave ovens have been tested as a rapid means of soil moisture determination (Miller et al. 1974; Hankin and Sawhney 1978; Gee and Dodson 1981). From a thermodynamic point of view, the microwave oven drying process exposes the sample to a steady, uniform heat source (Vankoughnett 1973). According to one theory, samples containing free and bound water should exhibit three distinct phases of weight and temperature change. The theoretical mass-time and temperature-time profiles are shown in Fig. 1 (Hall 1980).

The first phase, occurring between time 0 and T_f (time before free water evaporation) is characterized, in a steady heating environment, by a constant temperature rise and no change in mass. In the interval, $0 < t < T_f$, the mass at any given time is described by the equation

$$M(t) = k$$

where k is the initial mass. The second phase,

$$T_f < t < T_b$$

where T_b is the start of bound water evaporation, is characterized by constant mass loss with no temperature change, as described by the equation

$$M(t) = A(t - T_f) + B,$$

where A is the slope and B is the initial mass of soil. The third phase, T_b to T_d (time to complete drying), is characterized by exponentially-decaying mass loss and limited exponential temperature rise. The corresponding equation for mass is

$$M(t) = C_{\text{exp}} (D(t - T_b)) + E$$

where C is the moisture left in the sample at time T_b , D is the drying time constant and E is the final dry mass of soil. The ratio C/E , expressed as a percentage is therefore the water-holding capacity of the soil sample at time T_b . Assuming the

soil is homogeneous, the difference ($T_d - T_b$) should approach a theoretical constant for the same initial sample mass.

Results of previous studies have shown that total drying time may be reduced to the order of 20 min, depending on the mass of the sample, and that moisture content determinations compare to within 0.5% of values obtained by conventional electric oven methods which require in the order of 20 h (Gee and Dodson 1981). The main problem in using microwave ovens for experimental drying of soil is overheating of samples, resulting in splattering of soil when not properly contained (Hankin and Sawhney 1978), and loss of organic matter and nitrates (Miller et al. 1974). These problems were encountered due to lack of a method to determine the time at which the third phase of drying is reached and to control the temperature of the samples during that phase.

The primary aim of this study was therefore to introduce a technique to monitor temperature and mass in order to obtain data for system control. Given that the water content and heat transfer characteristics differ, depending on the soil types, three soils (clay, sandy loam and sand) were chosen for this study in order to determine the importance of soil texture on drying efficiency and time to transition points.

MATERIALS AND METHODS

Microwave oven instrumentation

The microwave oven used in this study was a laboratory model CEM MDS-81 Microwave Drying/Digestive System with a generated wavelength of 2450 MHz, rated at 600 W, variable from 0 to 100% in increments of 1% and programmable in terms of its time/power profile. The cavity dimensions were width 0.40 m, height 0.23 m and depth 0.34 m. The cavity contained a rotary platform and was equipped with an exhaust fan option. Two major modifications were made to the microwave oven: (1) the addition of a balance to monitor weight (mass) changes, and (2) addition of specially designed thermocouples to monitor temperature changes. The microwave oven was mounted on a raised platform to allow a load cell (Mettler PL 1200 electronic balance) to be placed under the unit. By means of a specially designed Teflon pan and stem passed through the hole previously occupied by the rotary platform, direct weighing could be carried out in the microwave cavity (see Fig. 2) with the balance outside the oven. This configuration was carefully tested for microwave leakage using a Holaday HI-1500 microwave leak detector and no measurable leakage was noted. For data acquisition purposes, the analog mV signal from the balance was tapped by by-passing the digital display on the balance. The signal from the load cell was determined to have a sensitivity

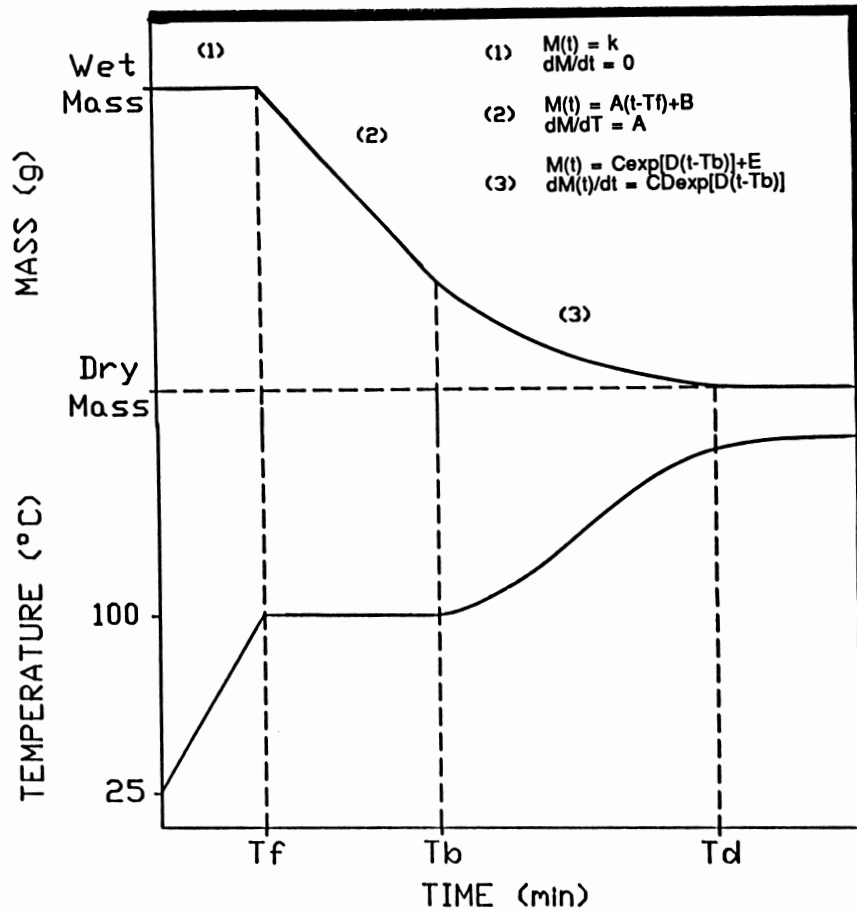


Figure 1. Theoretical mass-loss and temperature curves for the three phases of drying.

of 0.2698 ± 0.0004 mV/g and was converted to gram mass using a relation based on the initial and final weights recorded from the digital readout of the balance.

Defining the offset voltage as:

$$O = [(Fr/C) - Fw] C \quad (1)$$

where:

O = offset (mV),
 Fr = final reading (mV),
 Fw = final mass (g), and
 C = constant/slope (0.2698 mV/g).
the mass is given by:

$$M = (R - O) / C \quad (2)$$

where:

M = mass (g), O = offset (mV),
 R = reading (mV), and
 C = constant/slope (0.2698 mV/g).

Specially modified copper-constantan thermocouples shielded with nickel-plated copper braid (van de Voort et al. 1987) were also installed in the oven. The analog signals from the thermocouples and balance were digitized by a Campbell Scientific CR-7 datalogger (Logan, Utah 84321), which was programmed to convert voltages (mV) to temperature ($^{\circ}$ C) and mass (g). The resultant data were stored on a magnetic tape for later transfer to an IBM PC.

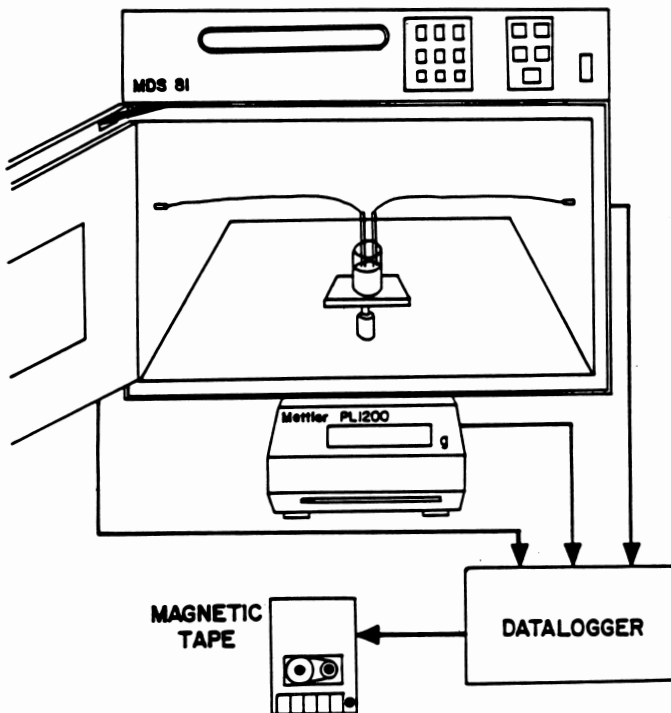


Figure 2. Experimental setup for measuring soil mass and temperature during microwave drying.

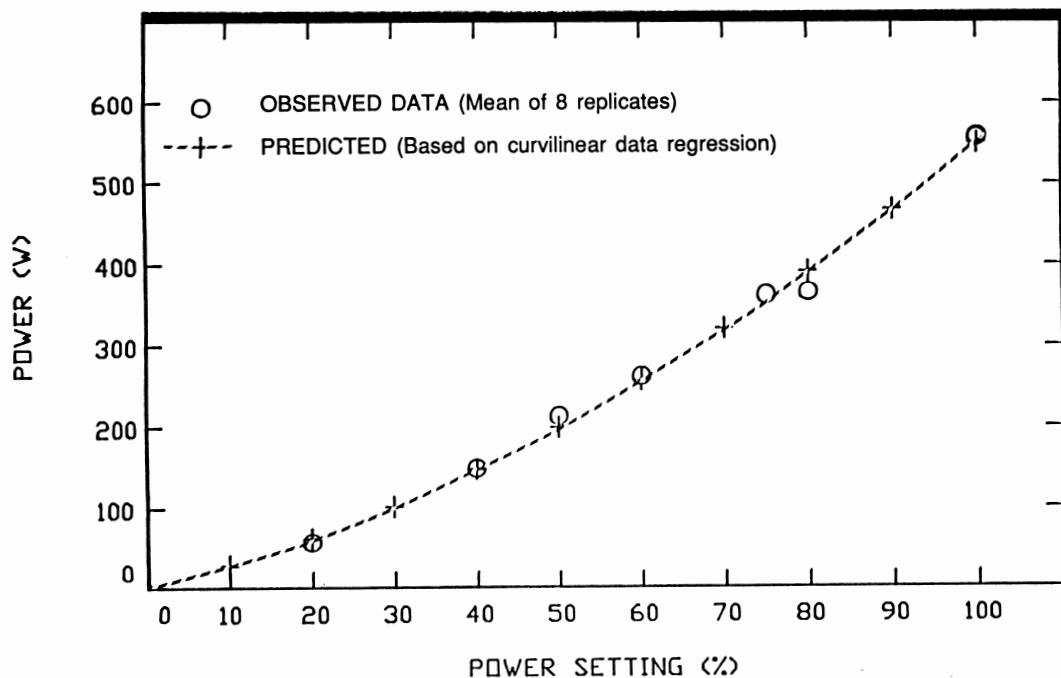


Figure 3. Conversion of microwave power setting in percent to power in Watts (Model CEM MDS-81 Microwave Drying/Digestive System).

Power calibration operation

Although the oven is rated at 600 W and the power is controlled within 1% of rated power, the useful heating energy is not linearly related to percent power. This is due to the nonlinearity of the controlling circuitry and variations in the microwave oven efficiency at different power settings. It was therefore necessary to calibrate to an accurate value. The method chosen to determine the amount of microwave power transmitted was to absorb the microwave energy in 250 mL of distilled water, measure the change in water temperature over time and then calculate the power required to achieve the measured change in temperature according to the following equation;

$$P = MCp(dT/dt) \quad (3)$$

where:

P = power (W),

M = mass (kg),

$C_p = 4180 \text{ J kg}^{-1} \text{ K}^{-1}$ = water heat capacity at 296 through 298 K,

T = temperature (K), and

t = time (s).

A constant heat capacity of water was assumed since it only varies by about 2% in the measured range of temperatures (298–373 K). Eight replicate runs at three power settings (50, 75 and 100%) were performed and the resulting power output calculated and averaged. Additional measurements were taken at power settings of 20, 40, 60 and 80% and a zero intercept was included as a data point for regression analysis. The following second-degree equation was found to fit the data with an R^2 of 0.99:

$$P = 2.5 (Pp) + 0.03 (Pp)^2 \quad (4)$$

where:

P = power (W),

Pp = microwave power expressed in % according to setting.

The calibration curve and observed points are shown in Fig. 3. It was also noted that the absorbed energy at 100% power was 557 W, representing 93% of the rated maximum.

Soil samples/experimental design

Soil samples (clay, sand and sandy loam) were obtained from the Champlain Valley region of Quebec. These soils were categorized in terms of their respective clay, silt and sand contents according to standards of the American Association for Testing and Materials (1970) using the dry-sieve method. The particle-size analyses are shown in Table I. Soil samples were prepared at four moisture levels, (a) saturated, (d) air dried at room temperature for 2 d, and (b) and (c), mixing a saturated and a dry sample in known proportions to give two intermediate moisture contents. The latter were allowed to equilibrate in sealed containers for 2 d. For the study of soil-drying profiles in the microwave oven, about 100 g of soil was placed in a 150 mL pyrex beaker on the weighing platform in the geometrical center of the oven. Samples were subjected to various microwave energies according to the unit's power setting; weights as a function of time were monitored. Separate experiments were carried out using thermocouples only, as both temperature and weight data could not be recorded simultaneously without the thermocouples causing irregularities in the weight profile due to their mass and vibration.

The experiment was a 3^2 factorial with soil types and power settings as the two factors. Four replicates of each treatment were performed for both mass and temperature measurements

Table I. Particle size analyses for selected soils

Soil	% clay ($<0.002 \text{ mm}$)	% silt ($>0.002, <0.05 \text{ mm}$)	% sand ($>0.05 \text{ mm}$)
Clay	62	20	18
Sandy loam	12	26	62
Sand	4	2	94

with the average results used for data analysis. Measurements were made every 5 s for either mass or temperature to provide a smooth profile of the changes taking place. The moisture determination for the individual soil samples was computed by:

$$\%MC = (WW - DW) / DW \quad (100) \quad (4)$$

where:

$\%MC$ = % moisture content on a dry basis,

WW = wet mass (g), and

DW = dry mass (g).

The dry mass value was determined to be the point at which the weight did not change more than 0.1 g min^{-1} , i.e., $dM/dt = 0$.

Data analysis

The transition times T_f and T_b were obtained from the mass time histories using an iterative regression technique based on

the theoretical models for the first two phases. T_f was determined to be the point at which there was a significant departure from constant mass. T_b was determined to be the point at which the slope of the constant mass loss line decreased significantly. T_d was identified as the mid-point of the first interval during which the mass-loss rate was less than 0.1 g min^{-1} . In this way, coefficients for the theoretical models were estimated for data in the respective intervals. Statistical analyses were performed using soil type and power as the two factors. Moisture content was considered to be a covariable in order that differences due to soil type not be confounded with differences in initial moisture content.

Drying efficiency during free water evaporation

The efficiency of microwave energy in removing water from a soil sample during the free water evaporation phase can be evaluated by computing the ratio of useful energy (energy for water evaporation) over the total energy emitted:

Table II. Values of coefficients A and B for the first phase linear equation, and of C, D and E for the second-phase exponential decay equation

Soil type	TMT†	Power (W)	A (g/min)	B (g)	C (g)	D	E (g)
Clay	a	550	-11.4	99.8	10.1	-1.12	61.6
			-12.0	99.7	12.3	-0.94	71.8
			-9.7	99.6	7.8	-1.16	83.7
			-‡	-	7.8	-1.56	91.5
	b	356	-7.3	99.4	6.3	-1.00	61.1
			-8.6	99.5	10.1	-0.84	72.3
			-7.3	99.4	8.8	-0.79	83.7
			-	-	8.4	-0.98	91.8
	c	200	-4.3	99.8	7.4	-0.51	62.2
			-5.2	100.0	8.9	-0.48	72.2
			-4.3	100.4	7.6	-0.51	83.7
			-	-	7.8	-0.53	91.9
Sandy loam	a	550	-9.2	99.5	7.9	-1.68	73.3
			-9.1	99.5	6.7	-1.00	83.6
			-8.8	99.4	4.9	-0.90	89.3
			-	-	2.3	-0.69	97.7
	b	356	-6.0	99.7	8.7	-0.89	73.6
			-7.5	100.0	9.2	-0.84	83.4
			-5.0	99.8	5.9	-0.71	89.4
			-	-	2.6	-0.33	97.5
	c	200	-3.7	100.2	4.4	-0.72	73.9
			-4.2	100.2	7.3	-0.47	83.4
			-3.5	99.9	6.4	-0.42	88.7
			-	-	1.8	-0.40	98.1
Sand	a	550	-7.7	99.1	5.5	-1.37	75.1
			-8.0	99.8	5.4	-1.17	82.1
			-5.8	99.7	3.9	-1.24	90.9
			-	-	0.9	-0.34	98.7
	b	356	-6.1	99.7	7.5	-1.14	76.9
			-5.4	99.6	6.3	-0.67	82.3
			-3.7	99.5	3.8	-0.89	90.6
			-	-	0.8	-0.04	95.7
	c	200	-3.9	100.1	4.8	-0.46	75.9
			-2.9	99.7	5.7	-0.49	83.2
			-4.7	99.7	3.6	-0.70	90.4
			-	-	5.1	-0.01	94.8

†TMT=treatments: a=saturated; b,c=mixed air-dried and saturated -, at known proportions; d=air-dried.

‡No free water left in sample after air-drying.

Note: minimum value of R^2 is 0.99.

$$N = \frac{(A)(Hfg)(100)}{60(P)} = \frac{3762(A)}{P} \quad (5)$$

where:

N = water removal efficiency (%),

Hfg = latent heat of vaporization (kJ kg^{-1}),

$A = dM/dt$ = rate of mass loss during free water loss (g min^{-1}),
and

p = power setting (W).

RESULTS AND DISCUSSION

The estimates of the model coefficients A through E obtained from these experiments are presented in Table II. The minimum coefficient of determination (R^2) obtained for the second- and third-phase equations was 0.99, showing that the theoretically-based equations describe the observed data very well. Typical mass and temperature profiles at 50% power (Figs. 4 and 5) clearly exhibit the theoretically predicted patterns.

During the first phase, there is no perceptible change in mass as microwave energy raises the water temperature linearly to 100°C , reflecting the constant heat capacity of the soil. However, since the sample is composed of both soil and water, the sensed temperature is that of water and not actual mass-average temperature of the sample. This is due to the dissipation of heat by conduction from the water to the soil matrix. T_f designates the end of the first phase and the start of free-water evaporation.

During the second phase, free moisture is evaporated at a near constant temperature of 100°C , the major sink for microwave energy being the latent heat of evaporation of water. Sample mass decreases linearly (constant rate, A) indicating that the rate of absorption of microwave energy by the remaining free water stays constant. As shown in Fig. 5, the temperature does not stay constant at 100°C for the whole free water loss region. This is due to the localized sensing of temperatures. These variations are due to dielectric phenomena which are more pronounced in the clay and sandy loam samples than in sand. It was found that at a given power level, the drying rate (A) in this phase is highest in clay and lowest in sand. This could be attributed to the additional absorption of energy by colloidal particles which is then conducted as heat to the water. Towards the end of free water evaporation, the temperature of colloidal soil types (clay and sandy loam) rapidly rises to temperatures above the boiling point of water, indicating the effect of dielectric properties of soil. The estimates of B obtained from regressions on the data were all within 0.6 g of the measured initial sample masses.

After T_b minutes, the free water has evaporated and the more difficult to remove bound water is being released. Mass decreases and temperature increases asymptotically in an exponential fashion to T_d . The drying time constant, D , was found to depend on power and moisture content at T_b . C , the mass of bound water left at time T_b , was fairly constant and highest for the clay soil, but tended to be lower in the sandy loam and the sand especially for the air-dried samples, indicating that bound water is less tightly held by the latter two soils and evaporates more readily in air than does the bound water in a clay. Because the water is scarce in the sample by the time T_b is reached, the microwaves are absorbed less efficiently and it is expected that the rate of evaporation would decrease exponentially. Furthermore, the temperature profiles indicate that much more energy is dissipated as sensible heat, with temperatures reaching as high as 300°C (power setting at 100%) during the final stage of the drying process. The temperature profiles of

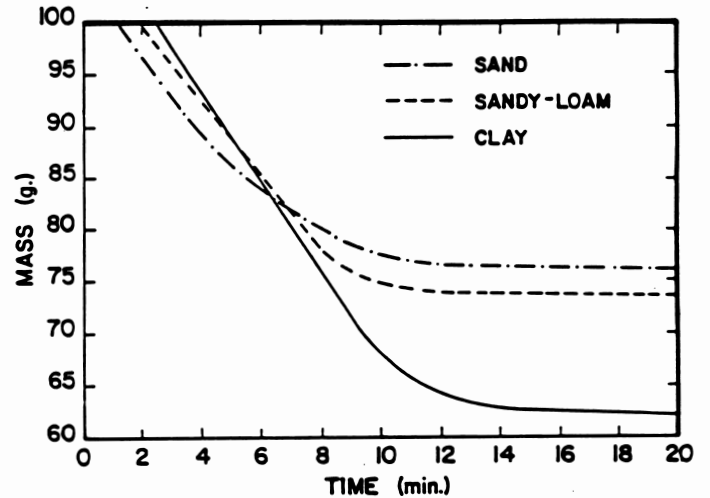


Figure 4. Typical mass loss of three soils while drying in a microwave oven (50% power, saturated soil samples).

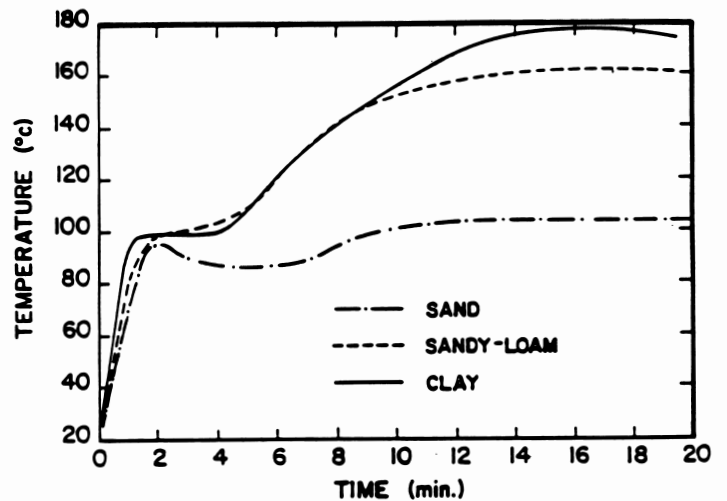


Figure 5. Typical temperature profile inside three different soils drying in a microwave oven (50% power, saturated soil sample).

the three different soils clearly show differing plateaus (Fig. 5), most likely due to the different dielectric properties. T_d was found to be affected by both power setting and initial moisture content; however, the difference ($T_d - T_b$), the time to evaporate bound water, was relatively constant at a given power level (Table III), as expected. Variations in ($T_d - T_b$) may be due to nonhomogeneity of samples with respect to clay content and/or physical changes caused by the drying process (e.g., changing pore structure).

Careful scrutiny of the mass loss data from the soil samples shows that a relatively constant asymptotic plateau is reached; however, given more time there is a continuing loss of mass, especially in the clay and sandy loam samples which reach very high temperatures. This signifies that the soil never completely stops losing mass as the microwave energy continues to be applied. This may be attributed, among other factors, to the continuing decomposition of organic matter and/or oxidation of inorganic matter at elevated temperatures. This problem makes it difficult to determine the actual endpoint or T_d . The use of several drying cycles or the programming of the microwave oven to reduce power under standardized conditions may be a way of overcoming this overheating problem which leads to continuing mass loss.

Table III. Average values of T_f , time to evaporate free water, time to evaporate bound water, and sample moisture contents

Soil type	Power (W)	TMT†	Moisture % (dry basis)	T_f	$(T_b - T_f)$ (min)	$(T_d - T_b)$
Clay	200	a	59.85	2.50	7.17	5.33
		b	36.89	1.75	3.75	4.80
		c	18.86	1.25	2.17	5.58
		d	7.96	-‡	0.00	5.17
	356	a	62.76	1.42	4.41	2.92
		b	37.29	1.17	2.08	3.75
		c	18.71	0.92	1.00	3.83
		d	8.54	-	0.00	3.92
	550	a	60.96	1.00	2.58	2.92
		b	38.48	0.83	1.34	3.83
		c	18.89	0.67	0.83	2.75
		d	8.89	-	0.00	2.50
Sandy loam	200	a	35.24	1.92	5.83	5.05
		b	19.45	1.17	2.33	6.50
		c	11.86	1.08	1.42	5.42
		d	1.50	-	0.00	4.00
	356	a	35.48	1.58	3.00	5.34
		b	19.49	0.83	1.09	5.08
		c	11.77	0.67	0.91	4.50
		d	1.63	-	0.00	3.50
	550	a	36.05	0.92	2.16	2.17
		b	19.33	0.50	1.08	3.09
		c	11.87	0.50	0.50	3.83
		d	2.05	-	0.00	3.42
Sand	200	a	31.08	1.17	6.41	4.42
		b	20.00	0.67	3.83	6.20
		c	10.38	0.75	1.25	4.08
		d	0.26	-	0.00	2.42
	356	a	30.11	0.67	2.83	3.50
		b	21.05	0.70	2.03	3.42
		c	11.43	0.83	1.50	3.25
		d	0.73	-	0.00	3.50
	550	a	32.05	0.25	2.50	2.25
		b	21.43	0.42	1.58	2.50
		c	9.77	0.33	0.84	2.83
		d	0.75	-	0.00	2.25

†TMT=treatments: a=saturated; b,c=mixed air-dried and saturated -, at known proportions; d=air-dried.

‡No free water in sample after air-drying for 2 d.

Average values of T_f time to evaporate free water ($T_b - T_f$), time to evaporate bound water ($T_d - T_b$) and measured moisture content are presented in Table III. ($T_b - T_f$) and ($T_d - T_b$) were both found to depend on power setting and soil type. With the data adjusted for the covariable, initial moisture content, significant differences for the two times were still detected for both power and soil type (0.05 level). This is interpreted as indicating that soil properties other than water-holding capacity are involved in the drying process, as also concluded for the drying rate, *A*.

Free-bound water limit

In all the test treatments, a separating point between linear mass loss and exponential mass decay with time was observed (T_b). The moisture content after this time can be defined as the bound water of the soil where a greater amount of energy is required to evaporate the water at the same rate as in the free-water region. The general tendency is that T_b decreases as the mean soil particle size increases (clay < sandy loam < sand). The ratio

C/E, expressed as a percentage, represents the water-holding capacity of the soil at T_b on a dry basis. Average values of this parameter (based on samples with some free water, i.e., treatments *a*, *b*, and *c*) were 12.3, 8.5 and 6.3% for clay, sandy loam and sand, respectively. These are substantially higher than the average moisture contents of the air-dried samples, (8.47, 1.73 and 0.58%) thus providing evidence for the loss of bound water during the 2-d period.

Drying efficiency

The computed efficiencies (Table IV) ranged between 39 and 95 with variation for a given soil sample being minimal regardless of the power setting and the initial moisture content (Table III). The computed efficiencies were higher for clay and sandy loam samples than for sand. The remainder of the energy was mainly dissipated by conduction heat transfer through the soil samples and the sample surroundings. In the bound water region, the water removal efficiency decreases exponentially with time until the drying process is completed.

Table IV. Computed water removal efficiency (N)†at different power settings for three soil types at different initial moisture levels

Soil	Initial moisture	Power setting		
		100%	75%	50%
Clay	a‡	77.56	77.55	80.25
	b	81.63	91.29	95.87
	c	66.02	78.01	79.88
Sand	a	51.93	64.72	72.36
	b	53.94	57.66	58.33
	c	39.43	39.33	53.47
Sandy loam	a	62.54	63.63	68.51
	b	61.79	79.52	77.95
	c	59.81	53.14	64.88

†See Eq. 5.

‡a=saturated; b,c=mixed air-dried and saturated at known proportions.

Proposed power setting for soil drying

Based on the power settings used in this study, the sample temperature reached values well over the suggested upper limit of 105°C. This could affect the overall soil composition and further studies would be necessary to determine standardized safe power settings. A setting of 50% power (approximately 280 W) appears to work well, having the advantage of reducing noise on the weight load caused by the boiling and escape of water from the sample.

CONCLUSION

The microwave oven and its use for studying the drying characteristics of soil was investigated. Drying characteristics for three soils were determined under variable moisture conditions and using three microwave power settings. The drying profiles of the clay, sand and sandy loam soils are similar to those encountered in conventional drying, however, on a substantially compressed time scale. In addition, much higher temperatures were attained than in the conventional processes. Programming of the microwave power setting could control this and would prevent decomposition of organic and inorganic compounds which

might affect results of chemical analyses performed after drying. For a saturated soil and 550 W absorbable microwave energy, the drying times were of the order of 6.5, 5.3 and 5.0 min for clay, sand and sandy loam; however, based on this work, 50% power settings would be better to work with which would increase the time to about 10–15 min per sample. The use of the microwave oven does not ensure 100% efficiency in terms of water removal; however, it does shorten the time involved in drying. Used in conjunction with a suitable software package, the instrumentation described should provide a useful tool for obtaining mass and moisture data from soil samples.

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