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I am submitting herewith a thesis written by Ellen R. McDonald entitled "Refinement of Loss-on-Ignition Method of Organic Matter Content in Highly Amended Soils." I have examined the final copy of this thesis for form and content and recommend that it be accepted in partial fulfillment of the requirements for the degree of Master of Science, with a major in Ornamental Horticulture and Landscape Design.

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REFINEMENT OF LOSS-ON-IGNITION METHOD FOR ORGANIC MATTER CONTENT IN HIGHLY AMENDED SOILS

A Thesis

Presented for the

Master of Science

Degree

The University of Tennessee, Knoxville

Ellen R. McDonald

December 1984

ACKNOWLEDGMENTS

The author wishes to express her gratitude to Dr. L. M. Callahan for serving as her committee chairman and directing her graduate program.

The author would also like to express her appreciation to Dr. R. J. Lewis for his help and for serving as a member of the author's committee.

The author is also indebted to Dr. G. M. Lessman for his guidance and for serving on the author's committee.

In addition, the author expresses her gratitude to Dr. G. D. Crater for serving as an ex-officio member on the author's committee.

Appreciation is also expressed to all other faculty and staff of the Ornamental Horticulture and Landscape Design Department, especially the secretaries. Also, heartfelt thanks are expressed to all of the author's fellow graduate students for their special friendship and constant support.

Finally, the author wishes to thank her parents for their faith and support during her course of study.

ABSTRACT

The purpose of this study was to refine the loss-on-ignition method of organic matter determination based on the previous findings of David Cochran (1980). An additional objective was to develop a pretreatment of organic matter for the reduction or prevention of error due to sample loss from violent eruption caused by rapid exposure to high ignition temperatures.

Pretreatment tests indicated that precharring samples at a temperature of 300° C for 10 minutes prevented loss of samples due to violent eruption at high ignition temperatures. The three sands and eight organic materials were then heated at different temperatures for different time periods. Two of the sands were washed river sands and the third was a mined silica sand. The eight organic materials used were corn stalk chop, cotton stalk chop, Michigan peat, oak sawdust, pine bark, sewage sludge, soybean stalk chop, and sphagnum peat. Temperatures used ranged from 350° to 450° C and time periods ranged from 2 to 8 hours. In all cases the best temperature and time combination was 400° C for 4 hours.

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CHAPTER I

INTRODUCTION

Organic matter is an essential component of rootzone mixtures in turfgrass culture under field conditions. Organic matter is necessary in supporting microorganism populations which in turn are responsible for molding of other components into suitable structures constituting a "soil." Organic matter provides a source of nutrients, increases water holding capacity of a soil, and aids in resistance of soil to compaction. With increased transition of golf greens and athletic fields to artificial soil mixtures containing high percentages of sand and organic matter, it becomes increasingly important to accurately determine amounts of each soil component present. Artificial rootzone mixtures may contain as much as 30 percent organic matter by volume.

Methods for determination of organic matter content of soils have been extensively reported. Among methods generally used are the Walkley-Black (2, 12), hydrogen peroxide (1), and loss-on-ignition. Based on findings by Cochran (3) it was determined that the loss-on-ignition method is most reliable of the three methods mentioned. However, Cochran reported only a general temperature and time for the loss-on-ignition method requiring additional tests to determine a more precise temperature and time.

Objectives of this study were (1) to refine temperature and time required for the loss-on-ignition method for organic matter

determination and (2) to develop a pretreatment of organic matter for the reduction or prevention of error due to sample loss from violent eruption caused by rapid exposure to high ignition temperatures.

CHAPTER II

REVIEW OF LITERATURE

Loss-on-ignition method was found by Cochran (3) to be more reliable than the commonly used Walkley-Black (2, 12) and hydrogen peroxide (1) methods in determining soil organic matter in artificial media. The method is based on high temperature ignition of soil, or an artificial soil mixture, with the organic fraction being oxidized leaving inorganic components. By subtracting weight of ash remaining from original weight of the soil sample, the difference obtained is assumed to be weight of organic matter lost. According to Ball (2) the usual procedure calls for removing soil moisture by heating the sample at 105° C overnight, then igniting a known quantity of dry soil at 850° C for 30 minutes. Loss-on-ignition values are expressed as percent of weight lost after the 105° C moisture loss (2).

Several temperatures and times have been reported for the loss-on-ignition method (2, 4, 7, 8, 10, 12, 14). There is little agreement as to the best or most reliable temperature or time to use. Hesse (8) recommends drying the sample at 110° C, then placing the dried soil into a muffle furnace at a temperature of 350° to 400° C for 8 hours. Wright (15) proposed air drying a soil sample and then igniting it in an electric furnace at temperatures between 650° and 900° C. Rhodes et al. (12) suggests oven-drying the sample overnight at 105° C, cooling the sample to room temperature in a

desiccator, then recording weight. The sample is then put in a furnace for 7 hours at 350° C, again cooled to room temperature in a desiccator, and weighed again.

Ball (2) supported two methods for loss-on-ignition: a high temperature and a low temperature ignition procedure. Both methods involved overnight drying of the soil sample in an oven at 105° C. The high temperature ignition procedure ignited the sample in a muffle furnace at 850° C for 30 minutes. The low temperature ignition procedure ignited the sample at 375° C for 16 hours. Ball recommended the high temperature ignition when less accuracy is needed and low temperature ignition where greater accuracy is required. Other ignition method recommendations include igniting samples at 430° C for 24 hours (4), 500° to 550° C for 2 hours (7), and 800° C for 2 hours (14). In all procedural recommendations ignition loss was expressed as percent weight lost after the 105° C moisture loss.

Loss-on-ignition method has been dismissed by some investigators as an inaccurate method of determining organic matter content of soil (1, 11, 13). Waksman and Stevens (13) stated that as much as 10 percent error may be inherent in this method due to loss of chemically bound water. Errors also result from destruction of micas and clay minerals, which are destroyed at temperatures ranging from 400° to 900° C (1, 6). Rather (11) pointed out that errors may result from loss of carbon dioxide from carbonates and from oxidation of incompletely oxidized minerals, such as ferrous iron compounds, at temperatures ranging from 500° to 900° C (6).

Several attempts have been made to correct errors associated with the loss-on-ignition methods (1, 2, 11). Ball (2) stated that primary loss of structural water and carbon dioxide occur at temperatures between 450° and 600° C, and that these errors are likely to be avoided at temperatures lower than 450° C. Keeling (9) supported this observation and found that heating clays for 16 hours at 375° C resulted in over 90 percent of carbonaceous materials being destroyed, but with no loss of structural water. Rather (11) recommended that samples, prior to being ashed, be treated repeatedly with dilute hydrochloric and hydroflouric acids to eliminate loss of hydroxyl groups. This process works, although small amounts of organic matter were destroyed. Alexander and Byers (1) modified Rather's procedure by decreasing the number of times samples are treated with acids by increasing concentration of the acids. This reduced loss of organic matter, but they concluded that the procedure was too slow and expensive for routine analysis.

Usefulness of the loss-on-ignition method for organic matter determination is considerable since organic matter determination is a routine laboratory analysis (2, 7, 12, 15). Harrison and Bocock (7) found that loss-on-ignition is a fairly precise method for determining soil bulk densities. Rhodes et al. (12) found a very strong correlation between ignition values and organic carbon content, thus making the method very useful for soils of medium-to-high organic carbon content. Ignition loss as an estimate of organic

matter composition, because of its simplicity and speed, can be useful in a wide variety of studies, even taking into account inherent errors (2).

CHAPTER III

MATERIALS AND METHODS

Studies were conducted to determine a more precise temperature and time for improving accuracy of the loss-on-ignition method. Tests were conducted on eight organic materials and three sands as may be used in high organic matter-sand mixes for golf greens. Experiments were conducted from November 1982 to June 1984 utilizing laboratory facilities of the Ornamental Horticulture and Landscape Design Department at The University of Tennessee, Knoxville.

I. OVEN CHARACTERIZATION

To check accuracy of the muffle furnace being used in these studies, oven characterization tests were performed. The furnace used was a Thermolyne Model No. F-6020 Series No. 60. The controller element was a newly purchased Thermolyne Furnatrol I Model No. CP18210 replacing an older model Thermolyne Dubuque III Model No. CPS-A8720 used in the original investigations by Cochran (3). The Furnatrol I model proved to be a definite improvement over the older Dubuque III model. The older model had a short "delay" built into it to prevent excessive cycling and chattering of the relay points. This permitted temperatures to occur other than the temperature set on the controller. The Furnatrol I model has a fully automatic, completely solid state non-indicating temperature control system. It eliminates variations in temperature due to line voltage

variations and ambient temperature fluctuations. There are no relays and contactors which permit extremely rapid cycling.

A pyrometer was used to characterize accuracy of the furnace. The thermocouple of the pyrometer was placed at several different locations inside of the oven to check temperature variations of the furnace. Temperatures proved to vary no more than 10° C from front to back, or from side to side inside of the oven.

II. PRECHARRING

Low temperature precharring tests were conducted on the eight organic materials. Purpose of precharring was to determine temperature and time for sample pretreatment to prevent violent eruption of samples during the combustion process at higher temperatures. Sample eruption under rapid exposure to high temperatures proved a source of some sample error.

Two precharring methods were tested: (1) graduated precharring and (2) specific temperature and time precharring. Graduated precharring consisted of placing samples in a cool oven and allowing gradual charring to occur up to the desired ignition temperature. When ignition temperature was reached, after approximately 45 to 60 minutes, sample ashing time began. Three replications were tested for each organic material. The end of the precharring period occurred when the furnace controller indicator showed that the pretest ignition temperature had been reached. Timing of a minimum ignition period could be determined at this point. A minimum ignition exposure

time was the important consideration. Extra ignition time exposure posed no significant errors.

The second precharring method involved exposing the eight organic materials to several different low precharring temperatures and times to determine which gave complete charring. Temperatures tested were 100°, 200°, and 300° C and times tested were 5, 10, 15, 30, and 60 minutes.

III. TEST MATERIAL PREPARATION

Eight different organic materials were selected for this study based on Cochran's (3) investigations. The organic materials used were the same eight used by Cochran. Organic materials tested were: corn stalk chop, cotton stalk chop, Michigan peat, oak sawdust, pine bark, sewage sludge, soybean stalk chop, and sphagnum peat. Michigan peat, sphagnum peat, and pine bark were obtained from local garden stores. Corn, cotton, and soybean stalks were obtained from crops grown on The University of Tennessee Plant Sciences Farm in Knoxville, TN. Oak sawdust was obtained from a local sawmill and sewage sludge from the Knoxville, TN, Third Creek Waste Disposal Plant.

All organic materials were oven-dried at 105° C then ground in a Wily mill to pass a 1 mm sieve in order to achieve uniform size. Test samples were stored in large paper bags in a dry location but were placed in smaller bags and again oven dried for at least 24 hours at 105° C just prior to actual use.

The three different types of sand used were the same three tested by Cochran (3). Two sands were washed sands from the Tennessee River and obtained from American Limestone Company in Knoxville, TN. The third sand was a mined, pure silica sand from White Silica Sand Company in Caryville, TN. One of the washed river sands was a yellow color and contained small amounts of mica and will hereafter be called "yellow sand." The other washed sand was brown and contained larger amounts of mica and other particulate matter and will hereafter be called "brown sand." The white mined sand will hereafter be referred to as "white sand." Each sand was screened through a 2 mm sieve prior to testing in order to remove all gravel and course debris. Particle size distributions were determined by the dry sieve method described by Day (5) using 100 g samples. All sands were dried at 105° C for at least 24 hours prior to use.

IV. SAMPLE SIZE

Sample size was determined by dimensions of the crucible used. Crucibles were high sided glazed Coors porcelain with 30 ml capacity and measuring 43 mm high and 37 mm diameter. The muffle furnace was large enough to hold 20 crucibles in tight arrangement. This easily permitted 11 crucibles to be loosely spaced in the muffle furnace to comprise the eight organic materials and three sands thus making up one replication for one test run.

Sample size was determined by filling crucibles one-half full with each organic material and the three sands. The half full mark was selected as a further safeguard against organic matter loss due to possible violent eruption even after precharring. Weight of one-half full crucibles of the less dense organic materials was approximately 1.0 g, thus this weight was used for all eight organic materials. The sands approximated 5.0 g per half-full crucible thus 5.0 g sample weights were used.

V. LOSS-ON-IGNITION METHOD

The loss-on-ignition method being refined is the procedure developed by Cochran (3) which was based on the method described by Ball (2). Process of refining the method began with oven-drying samples at 105° C for at least 24 hours. Samples were weighed into cool crucibles which had been previously weighed after being heated in a drying oven at 200° C for at least 10 minutes. The muffle furnace was preheated to the precharring temperature just prior to placing crucibles inside. Crucibles containing samples were then placed inside of the furnace and precharred. At the end of the precharring time, samples were removed from the furnace, the ignition temperature set on the controller, and time allowed for the furnace to reach the ignition temperature. When ignition temperature was indicated on the controller, precharred samples were returned to the furnace. However, ashing time actually began when the ignition temperature in the furnace was indicated on the

controller. Because of some heat loss due to opening of the furnace door, a short delay occurred between the time samples were returned to the furnace and ignition temperature was indicated on the controller.

Difference between sample oven-dry weight and weight of ash remaining in crucibles was termed loss-on-ignition. Loss-on-ignition therefore was considered to be the organic matter actually destroyed.

Tests were conducted with four replications of each organic material and three sands. Samples were ignited at temperatures of 350°, 400°, and 450° C for 4, 6, and 8 hours. To complete refinement of the method, temperature range was narrowed even further by adding two more temperatures and two shorter time exposures. Thus, final temperatures and times tested were 375° C and 425° C for 2, 3, and 4 hours and 400° C for 2 and 3 hours.

CHAPTER IV

RESULTS AND DISCUSSION

Studies were conducted to refine the loss-on-ignition method by identifying a more accurate temperature and time for sand and organic matter determination. An additional objective was to develop a pretreatment to reduce or prevent error due to sample loss from violent eruption caused by rapid exposure to high ignition temperatures. Results of these investigations were used to characterize each of three sands and eight organic materials studied in their separated form. It was considered much more important to retain individual peculiarities of each test material rather than risk loss of distinguishing characteristics as would occur in mixtures of the sands and organic materials.

I. CHARACTERIZATION OF SANDS

Particle Size Distribution

Cochran (3) performed fractional analysis of the three sands used in this study (Table 1). He found brown sand to be the coarsest with majority of particles falling in the coarse to medium ranges, while yellow and white sand had the majority of their particles in the medium to fine ranges.

Table 1. Particle size distribution of the three sands.

Size Fraction	mm	White Sand %	Brown Sand %	Yellow Sand %
Very Coarse	(1.000-2.000)	0.92	4.45	6.22
Coarse	(0.500-1.000)	3.04	54.49	15.01
Medium	(0.250-0.500)	44.69	39.30	27.52
Fine	(0.100-0.250)	41.72	1.57	34.24
Very Fine	(0.050-0.100)	4.57	0.05	13.69
Silt	(0.002-0.050)	2.3	4.0	1.4
Clay	(< 0.002)	3.0	2.5	2.4

Ignition

Brown, white, and yellow sands showed similar responses to changes in ignition temperatures and times used. All three sands lost small amounts of materials at each temperature and time combination. Brown and yellow sands showed greater weight losses than white sand (Figures 1, 2, 3). All three sands showed highest weight loss at 425° C for 4 hours. Brown sand exhibited highest weight loss of 0.68 percent followed by 0.66 percent for yellow sand and 0.40 percent by white sand (Table 2). The higher weight loss values reported for brown and yellow sands could be due to presence of higher quantities of alterable crystalline minerals known to be present in these sands.

According to Cochran (3), sharp increases in percent loss-on-ignition occurred as temperature was increased from 400° to 600° C. However, the temperature range used in this study was only from 350° to 450° C. There were no significant differences in weight loss for temperature and time ignition combinations for any one sand as shown in Table 2.

II. PRECHARRING OF ORGANIC MATERIALS

Two low temperature precharring methods were tested. They were graduated precharring and specific temperature and time precharring. Both methods were found acceptable. Graduated precharring prevented violent ash eruption of the eight organic materials tested. Results of the specific temperature and time method showed that

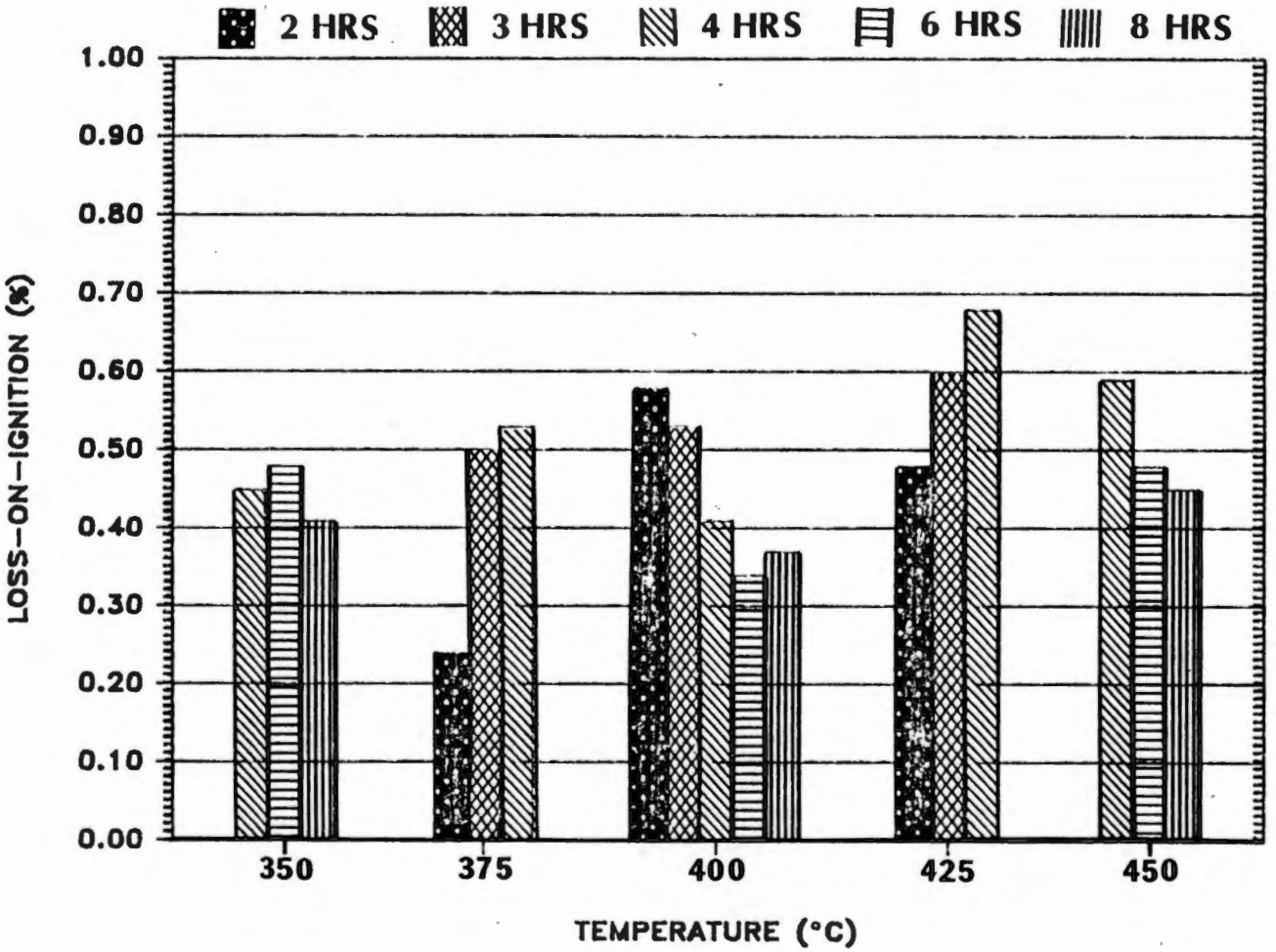

Figure 1. Loss-on-ignition of brown sand when ignited at different temperatures and times.

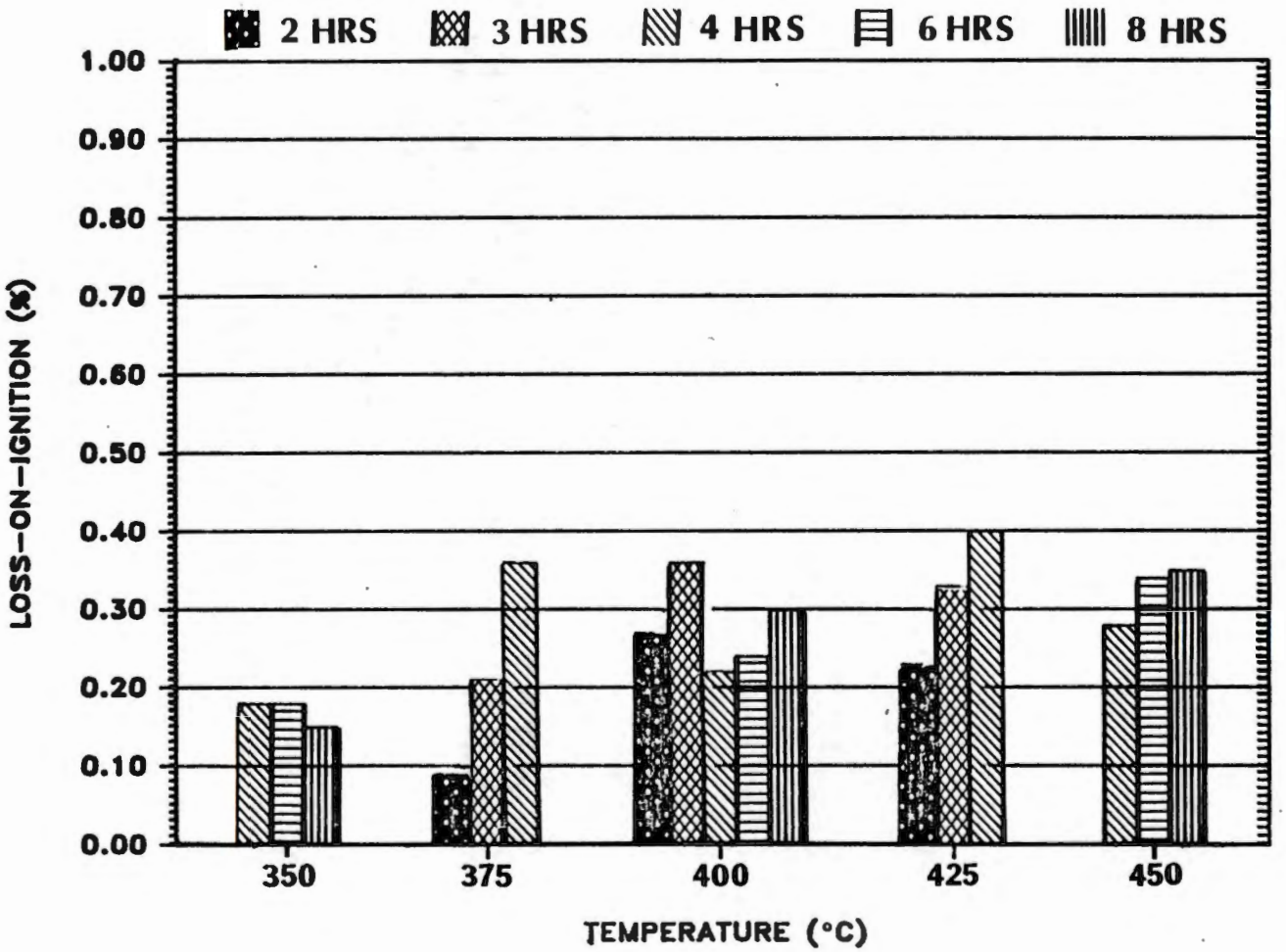

Figure 2. Loss-on-ignition of white sand when ignited at different temperatures and times.

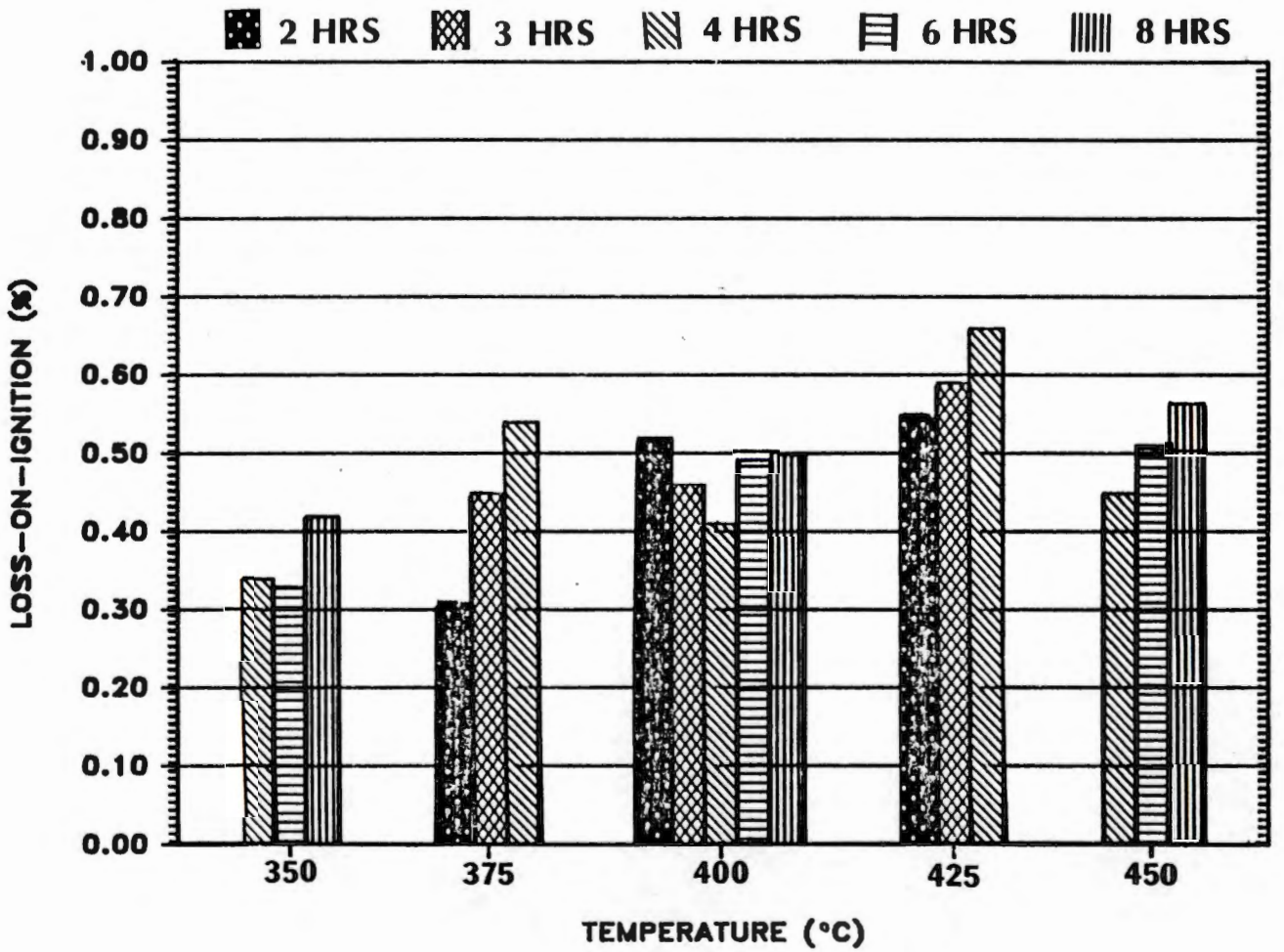

Figure 3. Loss-on-ignition of yellow sand when ignited at different temperatures and times.

Table 2. Mean percent weight loss of brown, white, and yellow sands ignited at different temperatures for different time periods.

Temp (°C)	Time (Hrs)	Percent Weight Loss[a] Brown Sand	White Sand	Yellow Sand
350	4	0.45 a	0.18 a	0.34 a
350	6	0.48 a	0.18 a	0.33 a
350	8	0.41 a	0.15 a	0.42 a
375	2	0.24 a	0.09 a	0.31 a
375	3	0.50 a	0.21 a	0.45 a
375	4	0.53 a	0.36 a	0.54 a
400	2	0.58 a	0.27 a	0.52 a
400	3	0.53 a	0.36 a	0.46 a
400	4	0.41 a	0.22 a	0.41 a
400	6	0.34 a	0.24 a	0.49 a
400	8	0.37 a	0.30 a	0.50 a
425	2	0.48 a	0.23 a	0.55 a
425	3	0.60 a	0.33 a	0.59 a
425	4	0.68 a	0.40 a	0.66 a
450	4	0.59 a	0.28 a	0.45 a
450	6	0.48 a	0.34 a	0.51 a
450	8	0.45 a	0.35 a	0.56 a

[a]Means within columns followed by the same letter are not significantly different at the 0.05 level of probability according to Duncan's Multiple Range Test.

300° C for 10 minutes was the best combination because it gave complete precharring. Temperatures below 300° C permitted variable amounts of uncharred organic matter which could be lost by violent eruption when samples were exposed to ignition temperature. The second method of a specific temperature and time appeared best for the purpose of this study as it required much less time.

III. CHARACTERIZATION OF ORGANIC MATERIALS

Five of the eight organic materials studied displayed similar loss-on-ignition responses. These organic materials were corn stalk, cotton stalk, oak sawdust, soybean stalk, and sphagnum peat (Tables 3, 4, 5 and Figures 4, 5, 6, 7, 8). Weight loss for organic materials in this group ranged from 94.37 percent for cotton stalk at 375° C for 2 hours to 99.02 percent for sphagnum peat at 400° C for 6 hours. Maximum percent weight loss was 99.02 for sphagnum peat, 98.66 for oak sawdust, 98.17 for soybean stalks, 97.55 for cotton stalks, and 97.50 for corn stalks. However, none of these five organic materials had maximum weight losses which were significantly higher than their respective weight losses at 400° C and 4 hours.

Ignition time was not as strong an influence in affecting weight loss as temperature. Cochran (3) found that time period of heating became less important as temperature was increased above 200° C. However, results of these investigations tend to suggest that 4 hours appears to be the shortest time needed to yield acceptable results (Tables 3, 4, 5).

Table 3. Mean percent weight loss of corn and cotton stalk chop ignited at different temperatures for different time periods.

Temp (°C)	Time (Hrs.)	Percent Weight Loss[a] Corn Stalk	Cotton Stalk
350	4	96.68 abcd	94.84 hij
350	6	96.15 cde	95.45 fghij
350	8	95.88 de	94.99 ghij
375	2	95.91 cde	94.37 j
375	3	94.89 f	94.64 ij
375	4	96.15 cde	95.69 efghi
400	2	96.20 bcde	96.05 bcdefg
400	3	96.21 bcde	95.86 cdefgh
400	4	97.29 a	97.49 a
400	6	97.03 ab	97.13 ab
400	8	96.79 abc	96.79 abcd
425	2	95.95 cde	96.23 bcdef
425	3	95.57 ef	95.61 efghi
425	4	96.33 bcde	96.10 bcdefg
450	4	97.50 a	97.55 a
450	6	97.08 ab	96.73 abcde
450	8	97.06 ab	96.89 abc

[a]Means within columns followed by the same letter are not significantly different at the 0.05 level of probability according to Duncan's Multiple Range Test.

Table 4. Mean percent weight loss of soybean stalk chop and sphagnum peat ignited at different temperatures for different time periods.

Temp (°C)	Time (Hrs)	Percent Weight Loss[a] Soybean Stalk	Sphagnum Peat
350	4	95.99 cd	95.87 g
350	6	95.93 cd	96.07 g
350	8	96.66 bc	98.15 abcd
375	2	96.37 bc	96.55 fg
375	3	94.97 d	95.92 g
375	4	96.65 bc	96.86 efg
400	2	96.64 bc	97.50 cdef
400	3	97.17 ab	97.57 bcde
400	4	97.99 a	98.69 a
400	6	97.80 a	99.02 a
400	8	97.80 a	98.37 abc
425	2	97.06 abc	97.35 def
425	3	96.08 bc	97.24 def
425	4	97.45 ab	97.97 abcd
450	4	98.17 a	98.85 a
450	6	98.00 a	98.58 ab
450	8	97.79 a	98.44 abc

[a]Means within columns followed by the same letter are not significantly different at the 0.05 level of probability according to Duncan's Multiple Range Test.

Table 5. Mean percent weight loss of Michigan peat and oak sawdust ignited at different temperatures for different time periods.

Temp (°C)	Time (Hrs)	Percent Weight Loss[a] Michigan Peat	Oak Sawdust
350	4	85.54 abc	96.90 f
350	6	85.17 abcd	96.96 ef
350	8	83.74 abcd	98.07 abcd
375	2	81.18 d	97.43 cdef
375	3	81.19 d	97.13 def
375	4	82.36 cd	97.52 bcdef
400	2	82.78 bcd	98.41 a
400	3	82.80 bcd	97.57 bcdef
400	4	86.25 abc	98.02 abcde
400	6	84.25 abcd	97.90 abcdef
400	8	84.68 abcd	98.40 abc
425	2	82.34 cd	97.36 cdef
425	3	83.60 abcd	97.21 def
425	4	86.91 abc	97.64 abcdef
450	4	87.48 a	98.31 abc
450	6	86.33 abc	98.20 abcd
450	8	85.27 abc	98.54 ab

[a]Means within columns followed by the same letter are not significantly different at the 0.05 level of probability according to Duncan's Multiple Range Test.

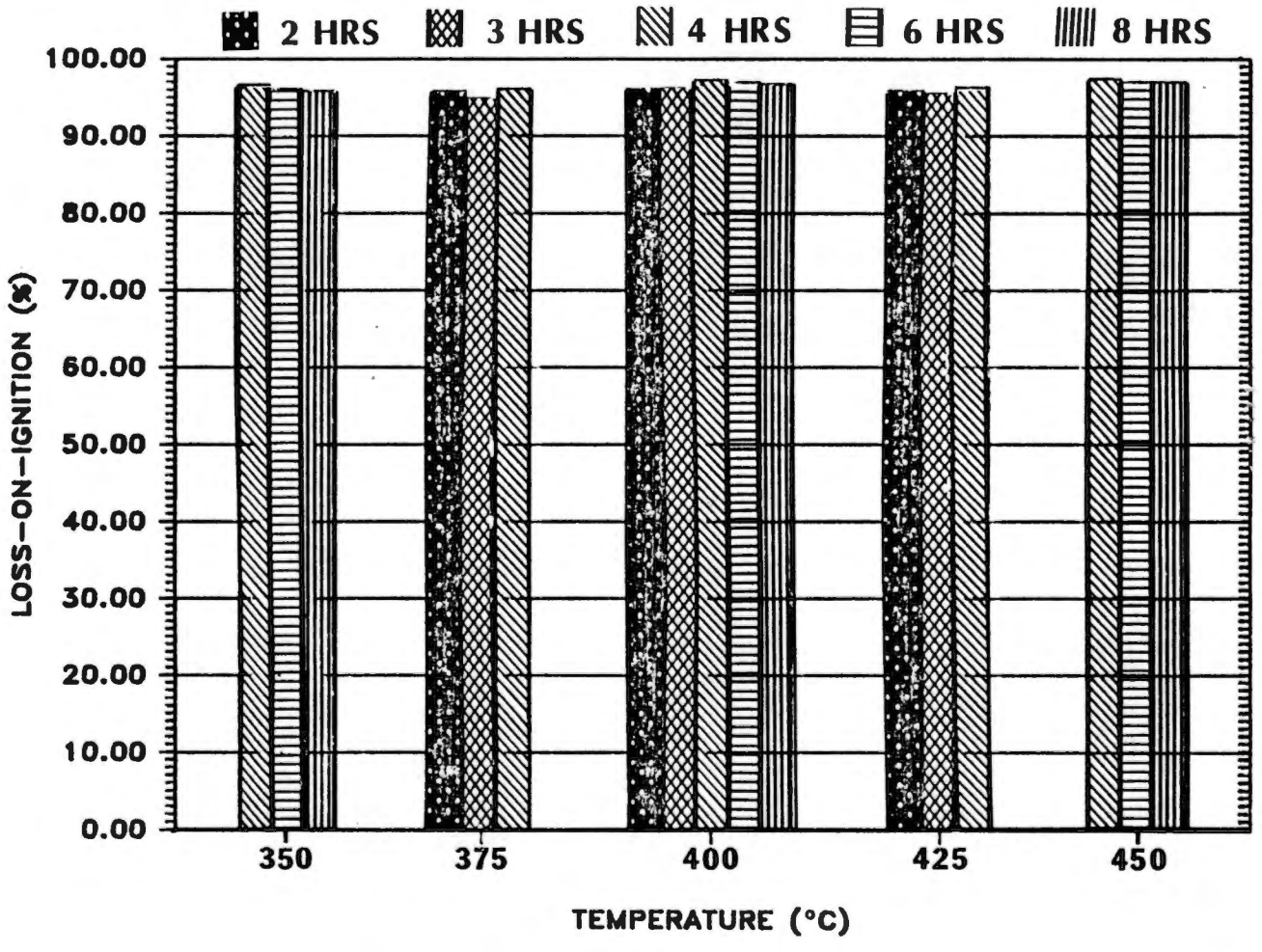

Figure 4. Loss-on-ignition of corn stalk chop when ignited at different temperatures and times.

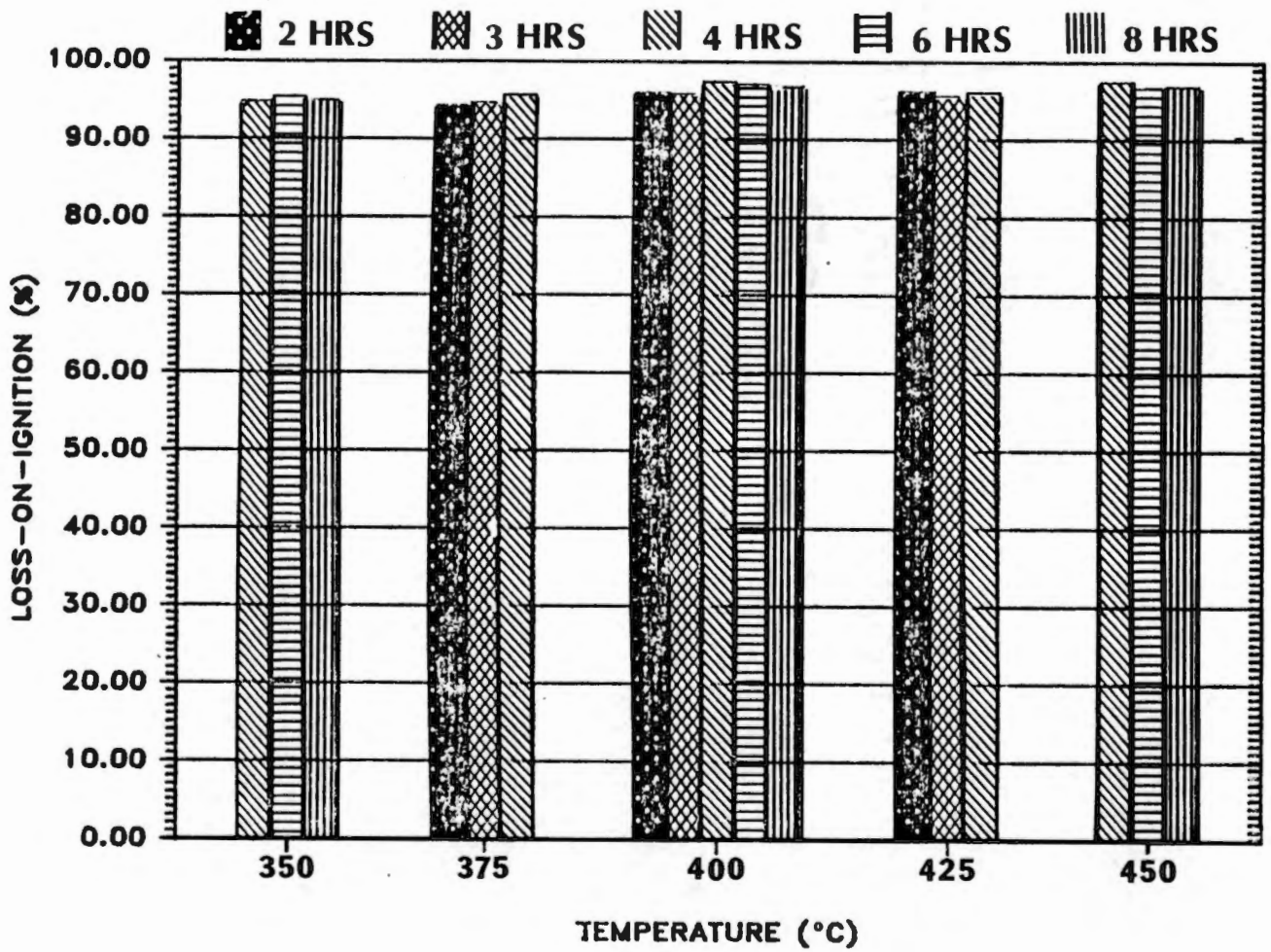

Figure 5. Loss-on-ignition of cotton stalk chop when ignited at different temperatures and times.

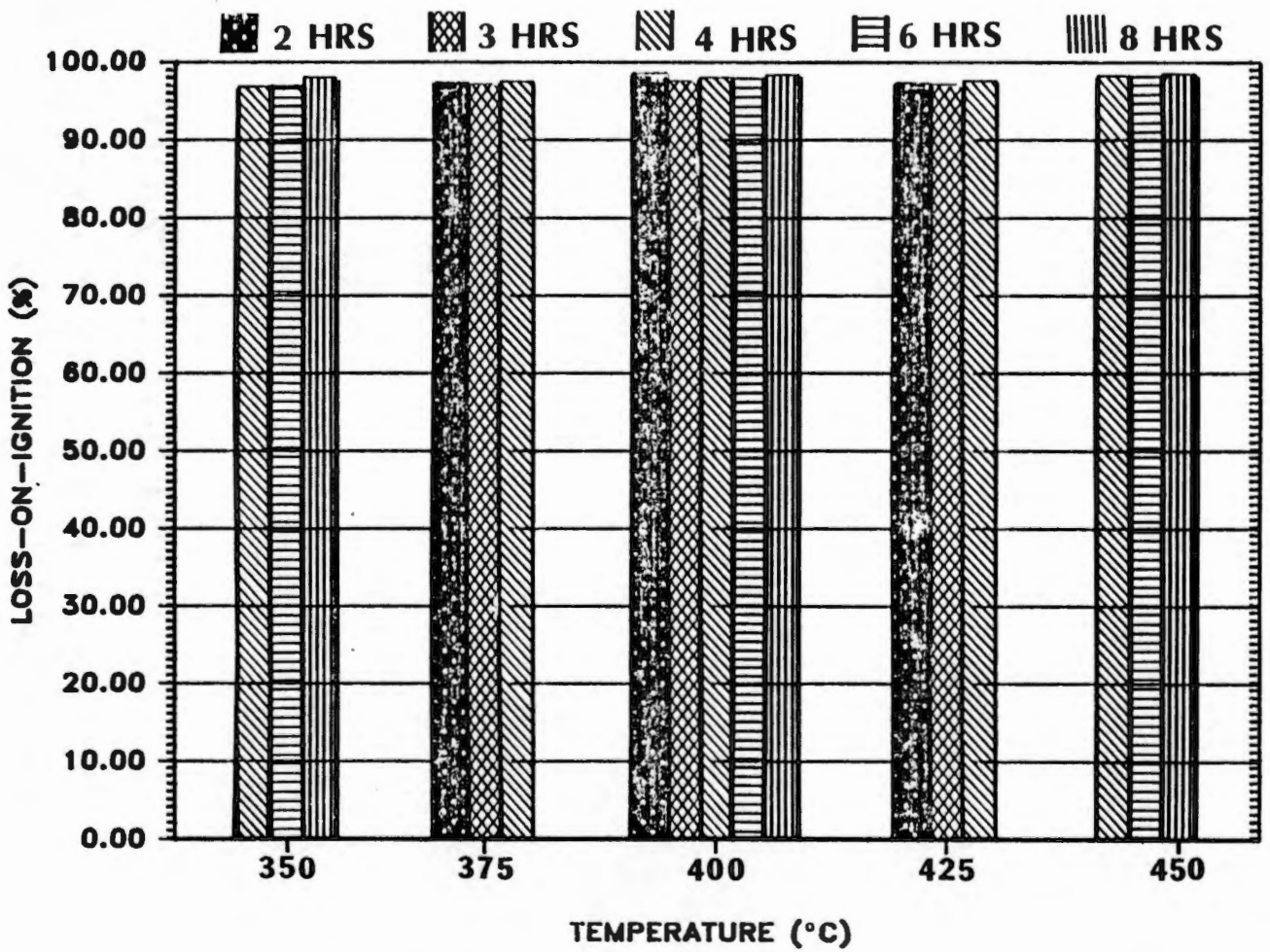

Figure 6. Loss-on-ignition of oak sawdust when ignited at different temperatures and times.

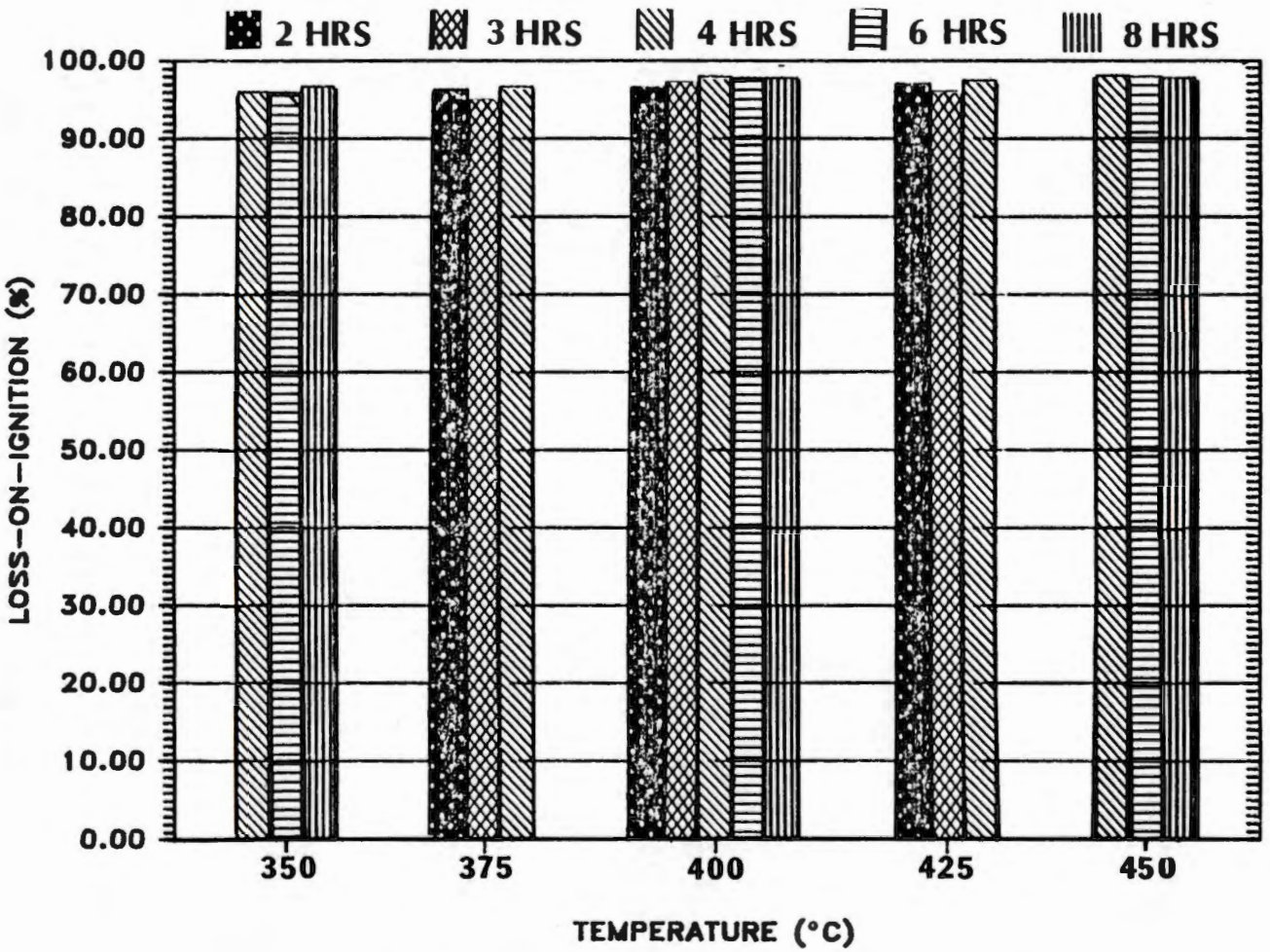

Figure 7. Loss-on-ignition of soybean stalk chop when ignited at different temperatures and times.

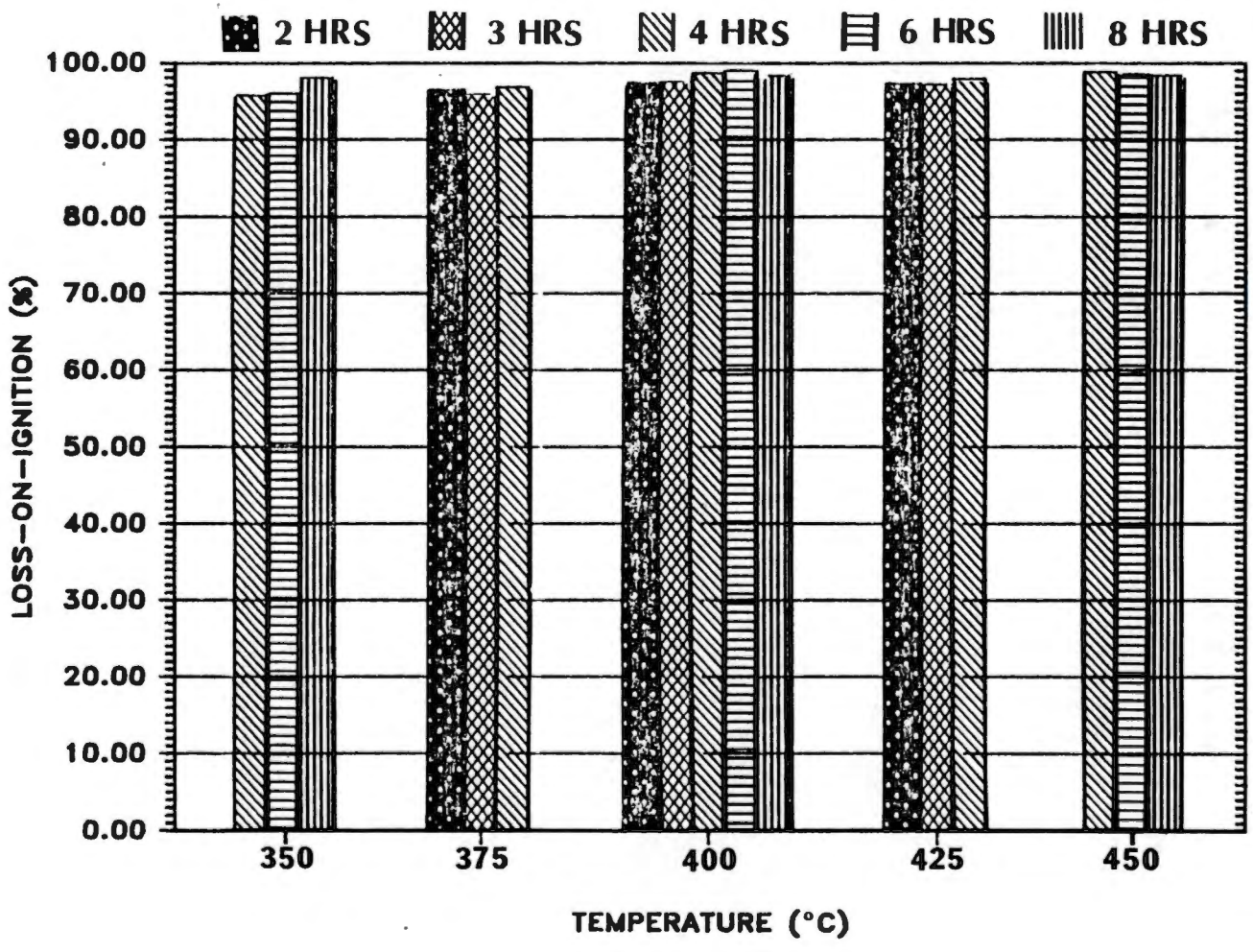

Figure 8. Loss-on-ignition of sphagnum peat when ignited at different temperatures and times.

Michigan peat exhibited similar temperature and time response patterns as the previous five organic materials except ignition loss values were much lower overall (Table 5 and Figure 9). Maximum ignition loss was 87.48 percent at 450° C for 4 hours. Like the previous five organic materials, maximum ignition loss values for Michigan peat was not significantly higher than ignition loss values of 86.25 percent at 400° C for 4 hours. Lower percent ignition losses for Michigan peat was expected since Cochran (3) showed this organic material to contain only 86 percent organic matter. An ignition exposure time of 4 hours appeared to be the minimum time needed to give acceptable results.

Pine bark ignition loss values were even lower than values for Michigan peat (Figure 10). Cochran (3) reported that considerable non-combustible contaminants were present in pine bark with a maximum ignition loss of 69.38 percent at 800° C for 12 hours. In these investigations, maximum weight loss of 65.55 percent occurred at 450° C for 4 hours, but a non-significantly different loss of 65.52 percent occurred at 400° C for 4 hours (Table 6). Thus a minimum ignition exposure time of 4 hours was sufficient.

Sewage sludge gave the lowest ignition loss values of all eight organic materials studied (Figure 11). Highest weight loss of 33.89 percent resulted at 450° C for 4 hours (Table 6). Weight loss for the lowest temperature and shortest ignition time which was not significantly different from the highest weight loss value was 33.07 percent at 400° C for 4 hours. Thus, the lower temperature

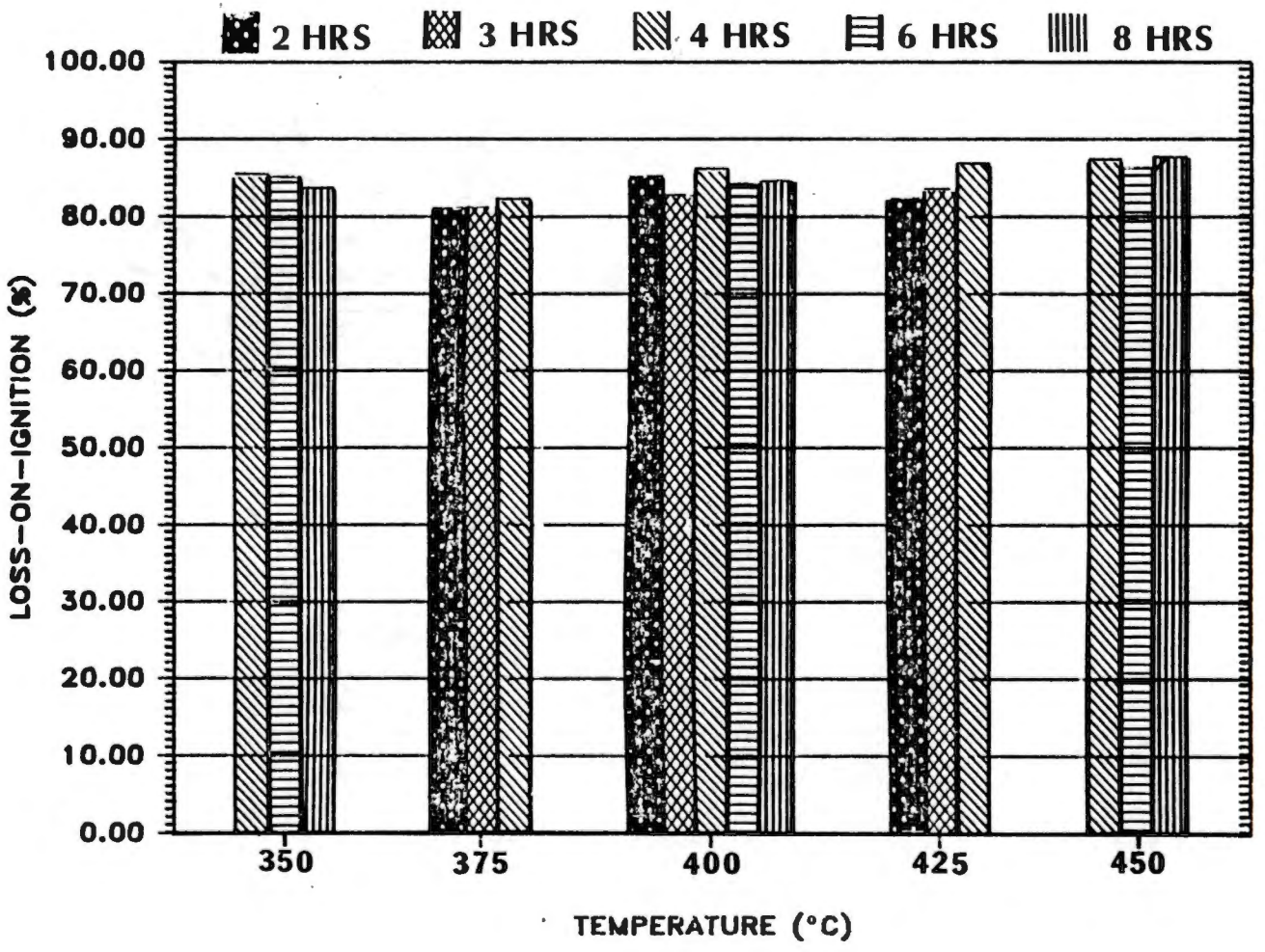

Figure 9. Loss-on-ignition of Michigan peat when ignited at different temperatures and times.

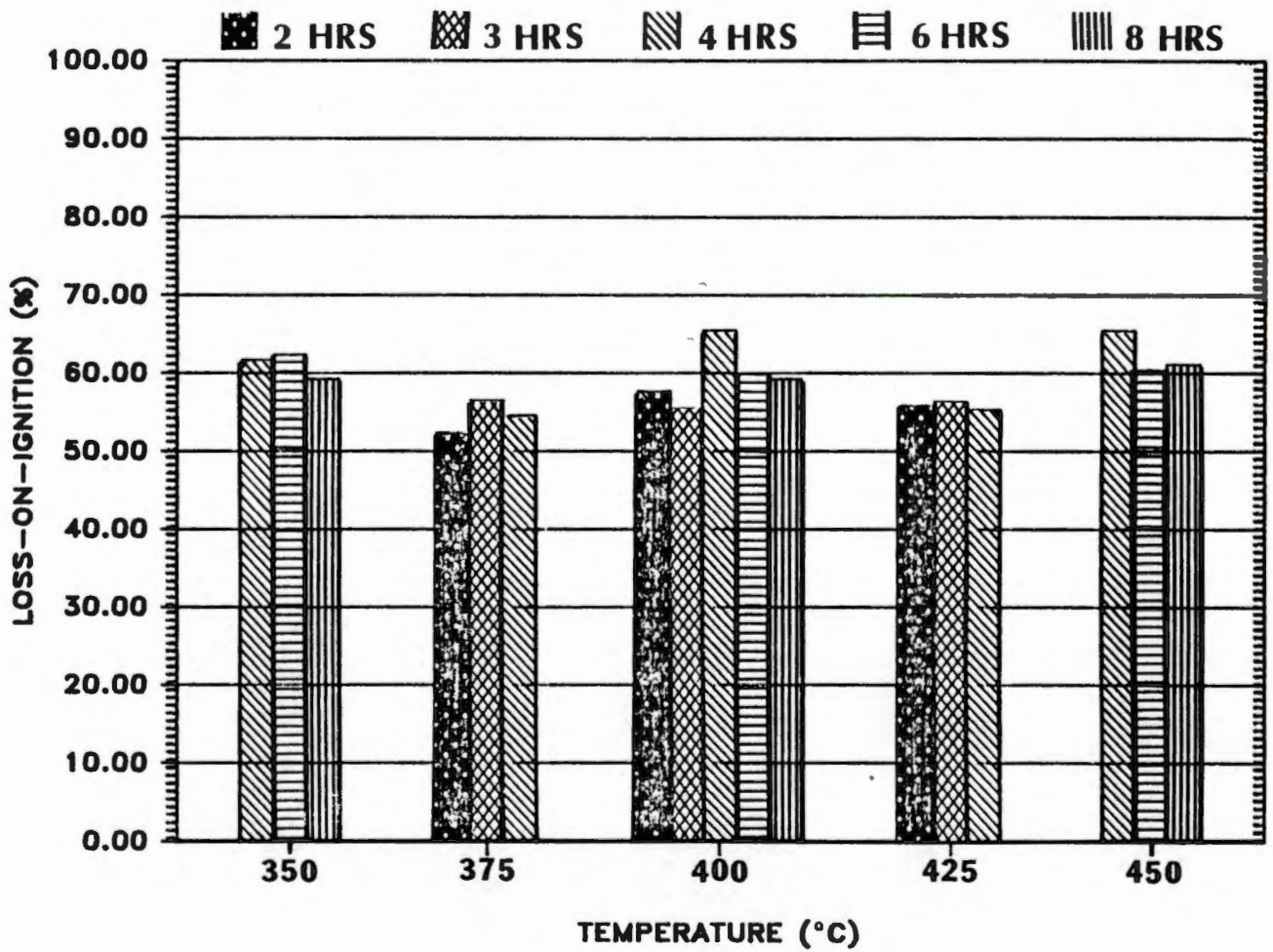

Figure 10. Loss-on-ignition of pine bark when ignited at different temperatures and times.

Table 6. Mean percent weight loss of pine bark and sewage sludge ignited at different temperatures for different time periods.

Temp (°C)	Time (Hrs)	Percent Weight Loss[a] Pine Bark	Sewage Sludge
350	4	61.73 bc	31.94 ab
350	6	62.39 b	31.18 ab
350	8	59.29 cde	30.24 b
375	2	52.44 h	28.20 b
375	3	56.49 efg	27.57 b
375	4	54.62 gh	28.77 b
400	2	57.77 def	28.19 b
400	3	55.43 fg	28.45 b
400	4	65.52 a	33.07 a
400	6	59.90 bcd	31.64 ab
400	8	59.26 cde	30.96 ab
425	2	55.91 fg	28.90 b
425	3	56.34 fg	27.60 b
425	4	55.33 fg	29.44 b
450	4	65.55 a	33.89 a
450	6	60.46 bcd	32.18 ab
450	8	61.16 bc	32.17 ab

[a]Means within columns followed by the same letter are not significantly different at the 0.05 level of probability according to Duncan's Multiple Range Test.

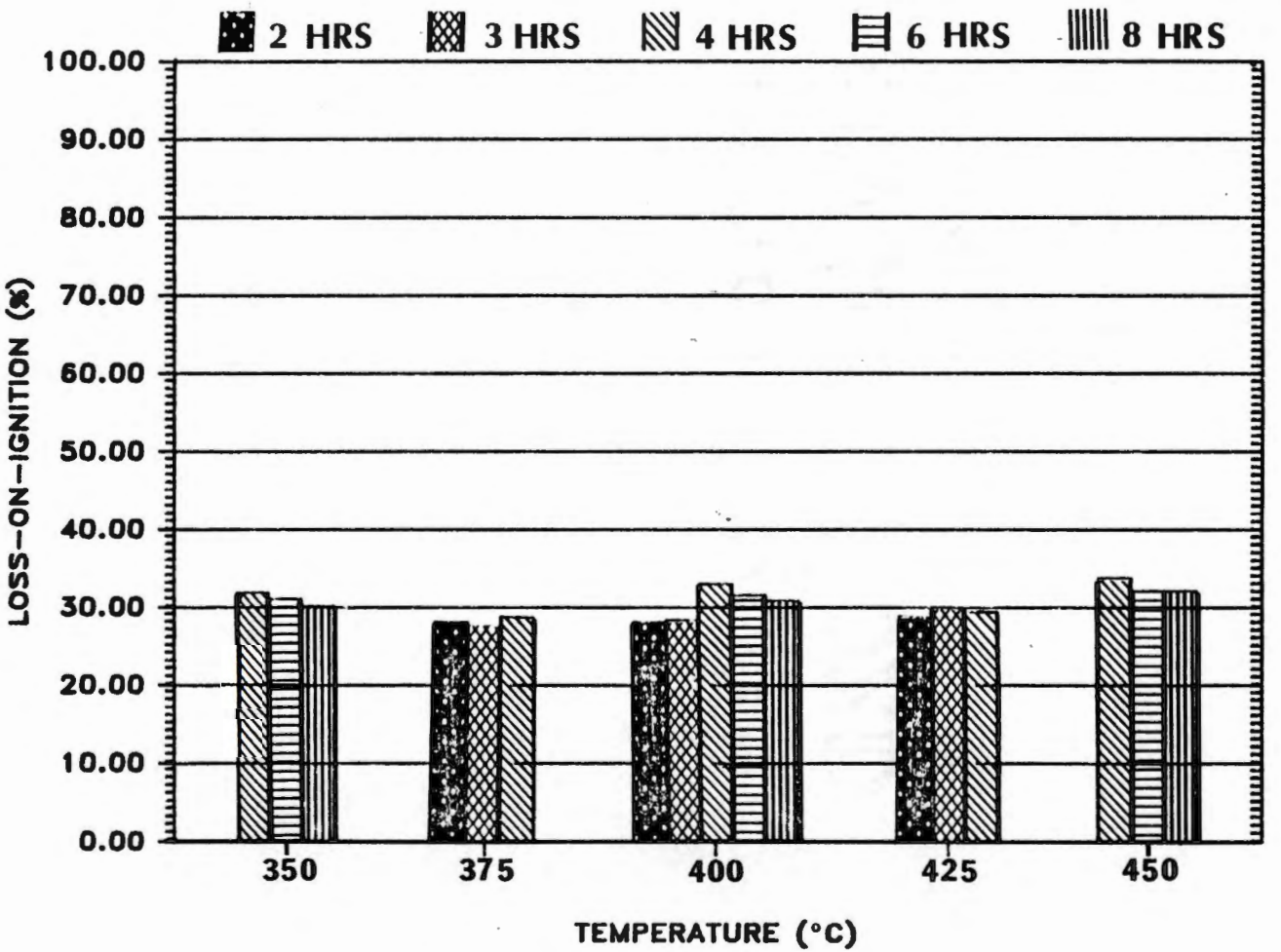

Figure 11. Loss-on-ignition of sewage sludge when ignited at different temperatures and times.

of 400° C and the shorter time exposure of 4 hours was more practical and economical. Cochran (3) found sewage sludge to contain 33.40 percent organic material at 400° C for 24 hours. The large percentage remaining is composed of generally unknown combustion resistant materials.

Cochran (3) determined densities of each organic material used. Sphagnum peat, corn stalk, soybean stalk, cotton stalk, oak sawdust, Michigan peat, pine bark, and sewage sludge had bulk densities of 0.132, 0.157, 0.248, 0.250, 0.250, 0.427, 0.515, and 0.687 g/cc, respectively. Mixing of organic materials and sands under field conditions is done on a volume, not weight basis. Due to variability in bulk densities for each of the materials used here, or any other organic material which could be used, weight per unit volume will vary. Thus percent of organic matter that each material contains would have much more practical use.

Tests utilizing volume standards under laboratory conditions have little meaning. Laboratory tests utilize the weight basis standard because it is much more accurate and can easily be measured for ultra small samples.

In order to develop accuracy and identify unknowns, laboratory technique investigations need to be conducted on separated organic materials and sands. Mixtures of the two different entities could easily mask unknowns.

CHAPTER V

SUMMARY

Purpose of this study was to determine a more accurate temperature and time for the loss-on-ignition method of organic matter determination. Loss-on-ignition is a method found by Cochran (3) to be the most accurate in comparison with the hydrogen peroxide and Walkley-Black methods. It is also the simplest and easiest to perform, and by far the fastest and most inexpensive. Results of these investigations are based on determinations conducted individually on three sands and eight organic materials.

The lowest temperature that did not give significantly different results was selected as a safeguard against errors which may occur at temperatures above 400° C due to loss of structural water (2, 6, 11, 13), loss of carbon dioxide and oxidation of incompletely oxidized minerals (11, 6), and from destruction of micas and clay minerals (1, 6). The shortest ignition time that did not give significantly different results was selected for economy of time as long as accuracy was not compromised, which is particularly advantageous where large numbers of samples are involved. Precharring tests conducted indicated that pretreating samples at a temperature of 300° C for 10 minutes prevented possible loss of samples resulting from rapid combustion and subsequent eruption of the organic materials at high ignition temperatures. Precharring

prevented a potential source of error which would be impossible to determine.

Weight losses of eight organic materials and three sands were measured at various temperatures and time periods. All sands showed a general increase in weight loss with increasing temperature. Within limits, time was not as important a factor in increasing ignition loss. Results indicate that temperature is the most important parameter in the loss-on-ignition method. This is due to lower temperatures causing less destruction of inorganic fractions present in sand and therefore less error. Temperatures in excess of 400° C are commonly reported by researchers to result in destruction of unpredictable levels of clays and other crystalline minerals (1, 2, 6, 10).

Loss-on-ignition percentages were similar for five of the eight organic materials: corn stalk, cotton stalk, oak sawdust, soybean stalk, and sphagnum peat. None had maximum ignition losses which were significantly higher than their respective ignition losses at 400° C and 4 hours. Shortest ignition time exposure giving best overall ignition loss was 4 hours. This is a time savings of 2 hours over the 6 hours recommended by Cochran (3).

Michigan peat had low ignition loss values due to presence of considerable amounts of non-combustible materials. Ignition losses for Michigan peat at 400° C for 4 hours was not significantly different from weight loss values at 450° C. Thus, 400° C for 4 hours gave acceptable ignition loss for Michigan peat with

less time and with less chance for error due to loss of crystalline fractions.

Pine bark contains approximately 30 percent by weight of non-combustible fractions, thus exhibiting very low ignition values (3). Difference in ignition loss at 450° C for 4 hours and 400° C for 4 hours were non-significant, thus the latter temperature and time were more acceptable.

Sewage sludge had the lowest ignition loss due to exceptionally high amounts of variable non-combustible contaminants. The highest loss was at 450° C for 4 hours which was not significantly different from ignition loss at 400° C and 4 hours. Again the latter temperature and time combination was more acceptable.

CHAPTER VI

CONCLUSIONS

Loss-on-ignition provides an accurate, reliable, rapid, and simple method for organic matter determination. A temperature of 400° C for 4 hours gave optimum results for all organic materials tested. These parameters proved acceptable also for the three sands tested. This study supported the temperature recommended by Cochran (3) but provided a time savings of 2 hours.

Results of pretreatment tests indicated that precharring samples at 300° C for 10 minutes prevented loss of samples from violent eruption when exposed to the higher ignition temperature.

LITERATURE CITED

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1. Alexander, L. T. and H. G. Byers. 1932. A critical laboratory review of methods of determining organic matter and carbonates in soil. USDA Tech. Bull. 317.

2. Ball, D. F. 1964. Loss-on-ignition as an estimate of organic matter and organic carbon in non-calcareous soils. Soil Sci. 15:84-92.

3. Cochran, D. S. 1980. Organic matter determination of components of high organic matter-sand-soil mixes. M.S. Thesis. The University of Tennessee, Knoxville.

4. Davies, B. E. 1974. Loss-on-ignition as an estimate of soil organic matter. Soil Sci. Soc. Amer. Proc. 38:150-151.

5. Day, P. P. 1956. Particle fractionation and particle size analysis. In C. A. Black (Ed.). Methods of soil analysis. Part 1. Amer. Soc. Agron., Inc., Madison, Wis. pp. 555-556.

6. Grim, R. E. 1962. Applied Clay Mineralogy. McGraw-Hill Book Co., Inc., NY, pp. 86-112.

7. Harrison, A. F. and K. L. Bocock. 1981. Estimation of soil-bulk-density from loss-on-ignition values. Jour. Appl. Ecol. 18:919-927.

8. Hesse, P. R. 1971. A Textbook of Soil Chemical Analysis. Chemical Publishing Co., Inc., NY, pp. 209-211.

9. Keeling, P. S. 1962. Some experiments on the low-temperature removal of carbonaceous material from clays. Clay Mineral Bull. 28:155-158.

10. Mitchell, J. 1932. The origin, nature, and importance of soil organic constituents having base exchange properties. J. Amer. Soc. Agron. 24:256-275.

11. Rather, J. B. 1917. An accurate loss-on-ignition method for the determination of organic matter in soils. Ark. Agr. Exp. Sta. Bull. 140.

12. Rhodes, E. R., P. Y. Kamara, and P. M. Sutton. Walkley-Black digestion efficiency and relationship to loss-on-ignition for selected Sierra Leone soils. Soil Sci. 45:1132-1135.

13. Waksman, S. A. and K. R. Stevens. 1930. A critical study of the nature and abundance of soil organic matter. Soil Sci. 30:97-116.

14. Williams, R. J. B. 1971. Relationships between the composition of soils and physical measurements made on them. Roth. Exp. Sta. Rep., 1970, Part 2, pp. 5-35.

15. Wright, C. H. 1939. Soil Analysis-Physical and Chemical Methods. D. Van Nostrand Co., Inc., NY, pp. 12-13, 160-178.

VITA

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While working toward her degree, she was employed by The University of Tennessee Agricultural Experiment Station as a Graduate Research Assistant in the Ornamental Horticultural & Landscape Design Department studying in the specialty field of Turfgrass Management.