IN THE LABORATORY

High-Frequency Dielectric Heating in Heterogeneous Catalysis

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High-frequency dielectric heating of materials is rapidly becoming a common practice in the plastics, wood, cellulose, and other industries (*1*). There are many advantages in this method of heating materials to a desired temperature. Since the dielectric heating arises within the material, due to rotational motion of molecules or translational oscillation of ions, the material is heated uniformly from the inside, and the factors of heat transfer are not involved. High-frequency dielectric heating is characterized by (1) speed of attaining a desired temperature, (2) uniformity of heating, and (3) absence of heat transfer from without.

These features make it very attractive for application in heterogeneous catalytic reactions. The uniformity of heating

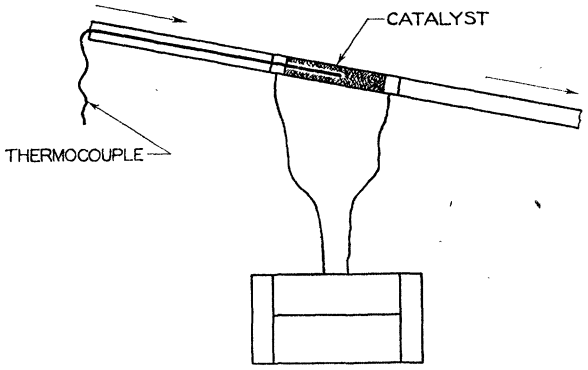

FIG. 1.

will eliminate the local overheating in spots of the catalyst and consequently minimize undesirable side reactions, increase the life of the catalyst, and produce a more uniform product. The rapidity of attaining the desired temperatures might decrease the over-all processing time, increase the space velocity, and decrease the size of the catalytic chamber.

These last features could have particular advantage for catalytic processes using a so-called "fluidizing" technique such as fluid catalytic cracking of petroleum. Naturally, only such solid catalysts as are dielectric by nature can be heated by high-frequency electric field.

In the following experiments, conducted in our laboratory, two reactions were carried out using dielectric catalysts: (1) dehydration of ethyl alcohol over alumina, and (2) dehydrocyclization of heptane to toluene over chromia-alumina.

The apparatus[1] consisted of a pyrex glass tube, with an inside diameter of 20 mm., filled with catalyst. The catalyst bed was 20 cm. long. Two brass electrodes were placed on both ends of the catalytic bed, outside the glass tube and connected to a Westinghouse radio frequency generator (1-KW unit).

After establishing a desired temperature (measured by a thermocouple placed in the center of the catalyst bed), which took from 3 to 5 minutes, the materials were introduced and the products recovered and analyzed in the usual way. The arrangement is shown in Fig. 1. The reactions proceeded with excellent results, giving a 90 per cent yield in the case of dehydration (at 350° and space velocity of 0.5) and 75 per cent yield for dehydrocyclization (at 500° and space velocity of 0.5).

Reference

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Method for Differential Staining of Mycorrhizal Roots

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In connection with an investigation of the cause of a foliar decline disease of southern pines (*2*), a study was made of the anatomical features of mycorrhizal roots formed on shortleaf pine (*Pinus echinata* Mill.). Several techniques have been used for the differential staining of hyphae of the Hymenomycetes, which form the mantle and Hartig net of ectotrophic mycorrhizae. Cotton blue in lactophenol is fairly specific for the hyphae provided counterstaining is not desired. Young (*6*) also obtained satisfactory results with cotton blue. McArdle (*3*) used the following differential methods: (1) hematoxylin with safranine, and (2) Pianeze III. In a study of the mycorrhizae of Colorado flora, Thomas (*5*) used a combination of safranine and fast green. A modification of the orseillein BB procedure described by Strasburger (*4*) has been used for staining mycorrhizae. (It should be mentioned that orseillein is not the same as orcein.) The procedure is as follows: Stain for 10 hours in a saturated solution of orseillein BB in 3 per cent acetic acid and counterstain with crystal violet in clove oil. All of the above techniques failed to contrast clearly the mantle and Hartig net with the cortical elements of the short roots.

Experimental tests demonstrated that a modification of Cartwright's (*1*) picroaniline blue procedure is superior to the above techniques for the differential staining of mycorrhizal roots. In the procedure which has been adopted for

[1] The author wishes to thank Dr. W. F. Winget for valuable help in operating the high-frequency unit.

histologic studies of all types of such roots, the roots are fixed for at least 48 hours in a solution of 1.5 ml. of acetic acid, 8.5 ml. of formaldehyde, and 90 ml. of 50 per cent ethyl alcohol. Zirkle's (7) N-butyl alcohol procedure is used for imbedding in paraffin, and Land's gum arabic-potassium dichromate procedure, for attaching ribbons to the slides. The slides are (1) stained for approximately 30 minutes with dilute safranine prepared by adding 3 ml. of a 0.5 per cent solution of aqueous safranine to 70 ml. of water in a Coplin jar, (2) rinsed in water to remove excess stain, (3) stained for 5–10 minutes in a solution prepared by adding 3 ml. of Cartwright's picroaniline blue to 70 ml. of water in a Coplin jar, (4) rinsed again in water to remove excess stain, (5) dehydrated by carrying the slides through a series of alcohol dilutions to 95 per cent alcohol, (6) cleared in Diaphane solvent, and finally (7) mounted in Diaphane.

By this procedure the blue-staining hyphae of the Hymenomycetes associated with ectotrophic mycorrhizae are clearly contrasted with the red-staining elements of the short roots. The dark-colored hyphae of the pseudomycorrhizal fungi are not stained. Differentiation of the intracellular hyphae, which grow out from the Hartig net, was obtained by using Wratten filter B (No. 58) in the microscope lamp.

The results obtained by the use of the above technique suggest the possibility that the foliar decline diseases of pine may be related to a reversal of symbiosis, which causes the mycorrhizal fungi to become parasitic on the short roots when soil conditions become unfavorable.

References

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5. Thomas, W. D., Jr. *Phytopathology*, 1943, **33**, 144–149.
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A Blood Test for Estimating the Week of Pregnancy[1]

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In a recent report (*1*) it was shown that the ability of human pregnancy plasma to inactivate pitocin increased over a thousandfold from the time of conception until term, and it was suggested that such a measurement might be useful in determining the week of pregnancy. The enzyme to which we refer as pitocinase is probably a peptidase and has not yet been fully characterized. In the present investigation it is shown that the concentration of this enzyme in the blood increases in a linear fashion for the first 16 weeks after conception, and that it is only during this period that the quantitative determination is reasonably accurate. Fortunately, it is during the first half of pregnancy that such information is usually desired, for beyond that time the activity, size, and osseous development of the baby are reasonably good criteria. The present report describes in detail the methods employed and the interpretation of the results.

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Method

Plasma from fresh, nonhemolyzed, oxalated blood, commercial pitocin, and saline are incubated at 37° C., and samples are removed at various times for assay of the residual pitocin. The plasma concentration and incubation times vary, of course, with the approximate duration of pregnancy. These are shown in Table 1. If the approximate duration is not known, more than one determination must be made. Immediately after mixing, one-quarter of the sample is withdrawn and diluted to 12.5 ml. with 0.85 per cent saline. One drop of 2 N acetic acid is added to bring the pH to about 5.5 (with chlorophenol red as an indicator), and the tube is placed in a boiling-water bath for 5 minutes. The sample is then filtered and neutralized (using bromthymol blue) with 1 drop of 2 N sodium hydroxide. This is the "zero time 100 per cent pitocin sample" with which the others are compared. The remaining portions, exactly equal in volume to the first, are withdrawn at the indicated times and treated in an identical manner. When incubations are prolonged for more than two hours, it is essential to incubate *in vacuo* in modified Thunberg tubes containing a few drops of toluene in order to prevent loss of enzyme activity through oxidation or bacterial action. Incubations of 24 hours are likely to give lower values because of shifting temperature optima.

TABLE 1

Wks. after conception	Range of units/ml. plasma	Incubation mixture (ml.)			Incubation times*	k′
		Pitocin	Plasma	Saline		
3–4	0.06–0.1	0.5	5.0	0	0, 18, 24 hrs.*	76
5–6	0.16–0.2	"	"	9.5	0, 18, 24 hrs.*	208
7–9	0.33–0.66	"	"	0	0, 1, 3, 5 hrs.*	76
10–12	1.0 –2.3	"	"	0	0, 20, 45, 90 min.	76
13–15	3.3 –6.0	"	"	4.5	0, 15, 30, 50 min.	139
16–18	10– 18	"	"	19.5	0, 15, 30, 45 min.	346
19–21	20– 30	"	"	Dilute to 50 ml.	"	693
22–24	35– 45	"	3.33		"	1,039
25–27	50– 65	"	2.0		"	1,732
28–30	60– 85	"	1.67		"	2,079
31–38	80–110	"	1.25		"	2,772

* When time exceeds 90 minutes, incubate anaerobically with toluene.

The relative amount of pitocin in each filtrate is then estimated by a standard U. S. P. oxytocic assay, using the isolated uterus of a rat or guinea pig. While details of the bio-assay cannot be presented in this brief communication, the technics and computations are well standardized (*2*). Obviously, the accuracy of the test hinges upon the care with which the bio-assay is made.

Using a sheet of semilog paper, the percentage of pitocin remaining in each sample is plotted (on the log scale) against time (on the arithmetic scale). Starting with 0 time and 100 per cent pitocin, a straight line is best fitted through the remaining points in order to obtain the average velocity of the reaction. The time in minutes where this line crosses the 50 per cent mark is called $t_{\frac{1}{2}}$, or the time for half destruction.