

study confirms the formation of OSS from isomalathion in concentrations depending on the carrier used. It can probably modify the potentiating activity of isomalathion also (Verschoyle et al., 1982). OSS being a stable product, monitoring its contents in malathion powders may be desirable.

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Registry No. II, 23060-14-2; III, 1642-46-2; VII, 22608-53-3; isomalathion, 3344-12-5; kaolinite, 1318-74-7; calcium silicate, 1344-95-2; polyethylene, 9002-88-4.

LITERATURE CITED

- Baker, E. L.; Zack, M.; Miles, J. W.; Alderman, L.; Warren, Mc. W.; Dobbin, R. D.; Miller, S.; Teeters, W. R. Epidemic Malathion Poisoning in Pakistan Malaria Workers. *Lancet* 1978, 31-34.
- Damico, J. N. The mass spectra of some organophosphorus pesticide compounds. *J. Assoc. Off. Anal. Chem.* 1966, 57, 165-167.
- Halder, A. K. Effect of Carrier on Isomalathion Formation. Ph.D. Dissertation, Post Graduate School, Indian Agricultural Re-

- search Institute, New Delhi, India, 1982.
- Halder, A. K.; Parmar, B. S. Effect of Carrier on Isomalathion Formation in Malathion Powders. *J. Pestic. Sci.* 1984, 9, 147-150.
- Miles, J. W.; Mount, D. L.; Staiger, M. A.; Teeters, W. R. S-Methyl Isomer Content of Stored Malathion and Fenitrothion Water-Dispersible Powders and Its Relationship to Toxicity. *J. Agric. Food Chem.* 1979, 27, 421-425.
- Miles, J. W.; Mount, D. L.; Churchill, F. C. The Effect of Storage on the Formation of Minor Components in Malathion Powders. Presented at the 24th Annual Meeting of the Collaborative International Pesticide Analytical Council, Granada, Spain, 1980.
- Rengasamy, S. Formation and Transformation of S-Methyl Isomer of Malathion in Technical Malathion and Malathion Products. Ph.D. Dissertation, Post Graduate School, Indian Agricultural Research Institute, New Delhi, India, 1986.
- Rengasamy, S.; Parmar, B. S. Investigation of Some Factors Influencing Isomalathion Formation in Malathion Products. *J. Agric. Food Chem.* 1988, 36, 1025-1030.
- Verschoyle, R. D.; Reiner, E.; Bailey, E.; Aldridge, W. N. Dimethylphosphorothioates: Reaction with Malathion and Effect on Malathion Toxicity. *Arch Toxicol.* 1982, 49, 293-301.

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New Approach To Improve the Gelling and Surface Functional Properties of Dried Egg White by Heating in Dry State

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The controlled heating in dry state was very effective to improve the gelling and surface functional properties of dried egg white. After heating at 80 °C in dry state (7.5% moisture content) for 7 days, the solubility of dried egg white was not affected and the Maillard reaction was not detectable. The surface hydrophobicity increased with heating time. The foaming and emulsifying properties increased in proportion to increases in heating time, correlating with surface hydrophobicity. The gel strength increased about 4 times with heating for 10 days. The electrophoretic patterns of heat-treated dried egg white revealed the formation of hydrophobic and disulfide protein-protein interaction. A mild change was observed in the CD spectrum when dried egg white was heated in dry state. Thus, the heating in dry state seems to be a new approach to improve the functional properties of food proteins.

Egg white proteins are extensively utilized as functional food products in food processing. For application in food processing, the pasteurization of liquid egg white is usually carried out for a few minutes at temperature near 60 °C. However, because of high heat sensitivity of egg white proteins, the functional property such as whipping property begins to be damaged at temperatures as low as 54 °C (Cunningham and Lineweaver, 1965). Dried egg white also generally receives heat treatment at 55-65 °C to reduce microbial numbers. The effect of heating on egg white at pasteurization temperature has been studied by many researchers (Seideman et al., 1963; Garibaldi et al., 1968; Cunningham and Lineweaver, 1965, 1967; Chang et al., 1970; Cunningham, 1974). However, they studied the effect of heating on egg white only in solution. There are

no studies about the effect of heating in dry state on the functional properties of egg white proteins. It is of interest to know the effect of heating in dry state on the functional properties of dried egg white by storing at various temperature in dry state for different times. Thus, we found a significant improvement of functional properties such as the foaming, emulsifying, and gelling properties of dried egg white by heating in dry state at 80 °C for several days. This finding may make possible to expand further applications of dried egg white in food processing. In addition, this phenomenon is interesting in terms of the investigation of conformational changes of proteins with heating in dry state.

MATERIALS AND METHODS

Egg white (DEW) spray-dried at 60-70 °C after decarboxylate treatment was provided by Q. P. Corp., Tokyo. Heat treatment of DEW was done as follow: 5 g of DEW was put in a test tube tightly sealed and then incubated at 80 °C for various periods of time (day) in dry state (7.5% moisture content). As soon as the sample was

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heated to a given time, the tube was taken out from the incubator and cooled at room temperature and subsequently surface functional properties and gelling property were measured. The measurements were performed after passing the sample solutions through a filter paper to remove any insoluble materials.

Measurement of solubility was carried out by determining the protein concentration in the samples (0.2%) of heat-treated DEW after filtration through a membrane filter (0.45 μm). Protein concentration of filtrate was determined by the modified Lowry method of Miller (1959).

Measurement of surface hydrophobicity of protein in heat-treated DEW were carried out by the method of Kato and Nakai (1980) using a fluorescence probe, *cis*-parinaric acid. Ten microliters of an ethanolic solution of *cis*-parinaric acid was added to 2 mL of 0.12% protein solution in 0.01 M phosphate buffer, pH 7.4. The mixture was excited at 325 nm, and the relative fluorescence intensity was measured at 420 nm in an Aminco-Bowman spectrofluorometer, Model J4-8962. The relative fluorescence intensity reading was adjusted to 1.0 when 10 μL of *cis*-parinaric acid solution was added to 2 mL of 0.01 M phosphate buffer, pH 7.4, in the absence of protein. The initial slope (S_0) was calculated from the fluorescence intensity vs protein concentration plot.

Foaming properties of heat-treated DEW in a dry state were determined by measuring the conductivity of foams produced when air at a constant flow rate of 90 cm^2/min was introduced for 15 s into 5 mL of 0.15% protein concentration in 0.01 M phosphate buffer, pH 7.4, in a vertical glass column (2.4 \times 30 cm) with a glass filter at the bottom (Kato et al., 1983). The conductivity of foams was measured by an electrode that had a cell. The cell was fixed inside the glass column 1 cm apart and 2.4 cm above the filter and was connected to conductivity meter (Kyoto Electric Industry Co., Model CM-07). Foaming power was defined as the maximum conductivity of foams produced after 15 s when air was introduced. Foam stability was calculated from conductivity curves as disappearance time of foam.

Emulsifying properties of heat-treated DEW were determined by the conductivity method (Kato et al., 1985). The emulsions were prepared as follow: 5 mL of corn oil and 15 mL of 0.175% protein solution in 0.05 M phosphate buffer, pH 7.4, were homogenized in Ultra Turax equipment (Hansen and Co.) at 12000 rpm for 1 min at 20 $^\circ\text{C}$. The emulsifying activity of each emulsion was calculated from the difference between the conductivity of protein solution and emulsion. The stability of each emulsion was calculated from the initial slope of the conductivity curve, as described previously (Kato et al., 1985).

Measurements of gelling properties were carried out as follows: 10% heat-treated dry powdered egg white solutions were made to determine its gel strength. Of this solution, 4 mL was put into an aluminum tube and tightly sealed. The gelation was carried out in a water bath at controlled temperature of 80 $^\circ\text{C}$ for 20 min. After a given time, the tubes were taken from the hot bath and cooled at room temperature (20 $^\circ\text{C}$) within 30 min. The gel intensity was immediately measured with a Tensile tester (Tensilon/UTM-II, Toyo Baldwin Co.). The gel intensity was expressed as breaking stress (g).

Polyacrylamide gel electrophoresis was performed as follows: The 7% polyacrylamide gel was set in a slab (50 \times 80 \times 1 mm). Portions of 20 μL of protein samples (0.1%) containing 0.005% bromophenol blue marker were applied to each slot of gel. Electrophoresis was performed

Table I. Solubility of Heat-Treated DEW at 80 $^\circ\text{C}$ in the Dry State for Various Periods of Time

heating time, days	absorbance at 660 nm	solubility, ^a %
0	0.68 \pm 0.02	100 \pm 3
1	0.69 \pm 0.03	102 \pm 4
3	0.68 \pm 0.01	100 \pm 2
5	0.69 \pm 0.03	102 \pm 4
7	0.66 \pm 0.03	99 \pm 5
10	0.64 \pm 0.02	95 \pm 3

^a The values of solubility of heat-treated samples are represented as the ratio to nonheated DEW sample (0 day). Reported values are means \pm standard deviations, $n = 4$.

at a constant current of 10 mA for 4 h with an electrophoresis buffer of Tris-glycine, pH 9.5. The gel sheets were stained with Coomassie blue G-250 for 30 min and destained in methanol/acetic acid/water (20:10:70, v/v/v) overnight.

SDS slab polyacrylamide gel electrophoresis was carried out by the method of Laemmli (1970), using 10% acrylamide separating gel and 3% stacking gel, containing 0.1% SDS. Protein samples (20 μL , 0.1%) were prepared in Tris-glycine buffer, pH 8.8, containing 1% SDS and 1% mercaptoethanol. Electrophoresis was carried out at a constant current of 10 mA for 5 h with an electrophoresis buffer of Tris-glycine containing 0.1% SDS.

SDS slab polyacrylamide gel electrophoresis in the absence of 2-mercaptoethanol was carried out with commercial gradient separating gel from 10 to 20% (Daiichi Pure Chemicals Co., Ltd., Tokyo) in gel size (84 \times 90 \times 1.0 mm) as described by Laemmli (1970). Protein samples were prepared in 0.125 M Tris-HCl buffer, pH 6.8, containing 2% SDS, 20% glycerol, and bromophenol blue marker dye (0.005%). Sample (20 μL) was applied to each slot of gel. Electrophoresis was carried out at constant current of 30 mA for 2 h in electrophoretic buffer of Tris-glycine, pH 9.5, containing 0.1% SDS. Then the gel was stained with Coomassie brilliant blue and destained in methanol/acetic acid/water (20:10:70, v/v/v) overnight.

Measurement of free amino groups was carried out as follows: A 50- μL portion of DEW solution (0.2%) was added to 1 mL of 0.2 N sodium borate buffer, pH 9.6, and then 1 mL of TNBS reagent (equal volumes of 0.1% trinitrobenzenesulfonic acid and 0.126% sodium sulfide) was added. This mixture solution was allowed to stand at 37 $^\circ\text{C}$ for 2 h and subsequently cooled to the room temperature before the absorbance was measured at 420 nm.

Circular dichroism (CD) analysis was performed as follows: 10 mg of DEW samples was dissolved in 7 mL of 0.01 M sodium phosphate buffer, pH 7.2. Each of samples was filtered through a membrane filter (0.22 μm). The protein solution thus obtained was used for CD analysis. CD was measured on a Jasco Model J-500 spectropolarimeter in the far-ultraviolet region (190–260 nm) with a 0.009-cm path length cell at 25 $^\circ\text{C}$. The accuracy of the data was improved by averaging eight scans integrated with the data processor (Model DP-501). CD spectra were expressed in terms of mean residue ellipticity ($\text{deg cm}^2 \text{dmol}^{-1}$).

RESULTS

The optimal heating condition of DEW was preliminarily examined in the range 65–100 $^\circ\text{C}$. The heat treatment at 80 $^\circ\text{C}$ in the dry state (7.5% moisture) yielded the best functional properties. In addition, as the heating at 80 $^\circ\text{C}$ is efficient for the reduction of microbial population in the industrial application, this condition was chosen in this experiment. Table I shows the observed values of solubility of heat-treated DEW at 80 $^\circ\text{C}$ in the

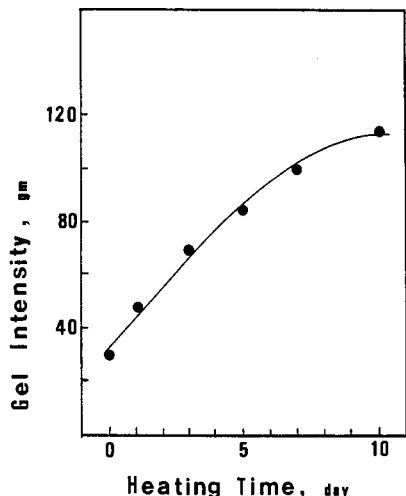


Figure 1. Relationship between gel strength and heating time in the dry state of DEW.

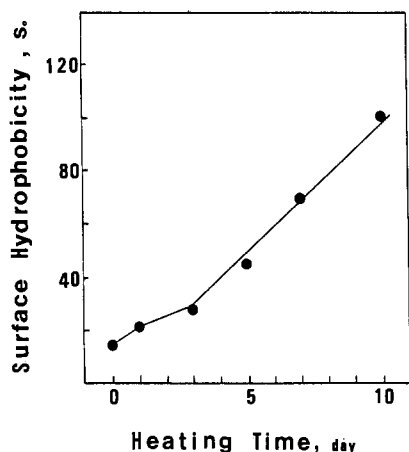


Figure 2. Relationship between surface hydrophobicity and heating time in the dry state of DEW.

dry state for various periods of time. The solubility was expressed as the ratios to value of nonheated sample. It was observed that there was no effect for heating in the dry state on the solubility of DEW within the incubation period of 7 days, while it slightly decreased with heating time up to 10 days. The values of the solubility were $99 \pm 5\%$ and $95 \pm 3\%$ of the samples incubated for 7 and 10 days, respectively. This result indicates that the effect of heating in the dry state on the solubility of DEW proteins was very small. The effect of heating in the dry state on the gelling properties was investigated. Figure 1 shows the relationship between heating time in the dry state at 80°C and the gel strength of DEW. The gel strength of DEW solutions was greatly increased with increased heating time. Thus, heating in the dry state progressively increased the relative gel strength and resulted in excellent firmness gel structure. The relative gel strength indicated about 4 times the value of nonheated DEW after 10 days of heating. Figure 2 shows the relationship between surface hydrophobicity of DEW and heating time in dry state. The surface hydrophobicity greatly increased to give a strong positive correlation with increases of heating time in dry state. The values of surface hydrophobicity (S_0) increased about 7-fold over the value of nonheated DEW, when heated at 80°C for 10 days in the dry state.

As shown in Figure 3, the foaming properties gradually increased with heating time in the dry state. The foaming power and foam stability were enhanced with increases of heating time in the dry state even when DEW samples were heated for 10 days. Similarly the relationship between the

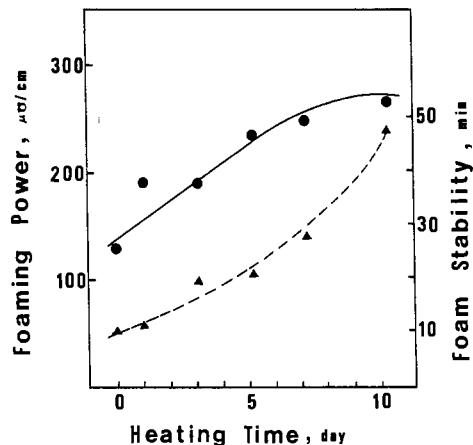


Figure 3. Relationship between foaming properties and heating time in the dry state of DEW: ●—●, foaming power; ▲---▲, foam stability.

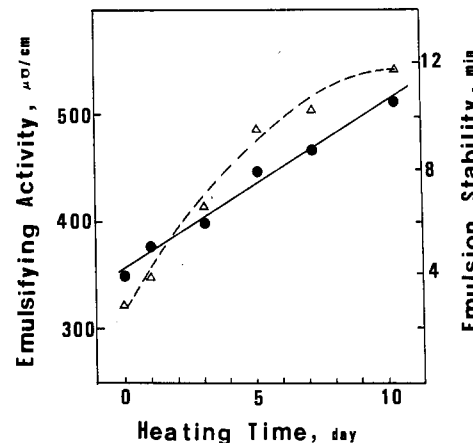


Figure 4. Relationship between emulsifying properties and heating time in the dry state of DEW: ●—●, emulsifying activity; ▲---▲, emulsion stability.

emulsifying properties and heating time in dry state of DEW was investigated (Figure 4). Good emulsifying activity was given by the increases of heating time, and a linear correlation has been observed between emulsifying activity and heating time in the dry state. Emulsion stability also greatly increased by heating in the dry state. From this result it seems likely that heating in the dry state for a limited period of time is desirable to improve the foaming and emulsifying properties and to obtain optimal functionality of dried egg white proteins. This result suggests that heat treatment of proteins in the dry state tends to improve the emulsifying and foaming properties by enhancing structural flexibility and surface hydrophobicity.

The effects of heating in the dry state on the structure of dried egg white proteins were studied by CD analysis. Figure 5 shows typical CD spectra of nonheated and heat-treated for 5 days at 80°C in dry-state samples of DEW. As shown in the patterns, the changes in CD spectrum with heating for 5 days were very mild. It is of interest that the heat treatment in the dry state of DEW for 5 days hardly affects the CD spectrum.

The polyacrylamide gel electrophoresis patterns of heat-treated DEW samples in the dry state are presented in Figure 6A. The polyacrylamide gel patterns revealed the formation of soluble aggregates that could migrate slowly into the gel when the DEW samples were heated for 3 and 5 days, and the intensities of the bands of aggregates were increased with increased heating time. The result suggests that polymerization occurred between egg

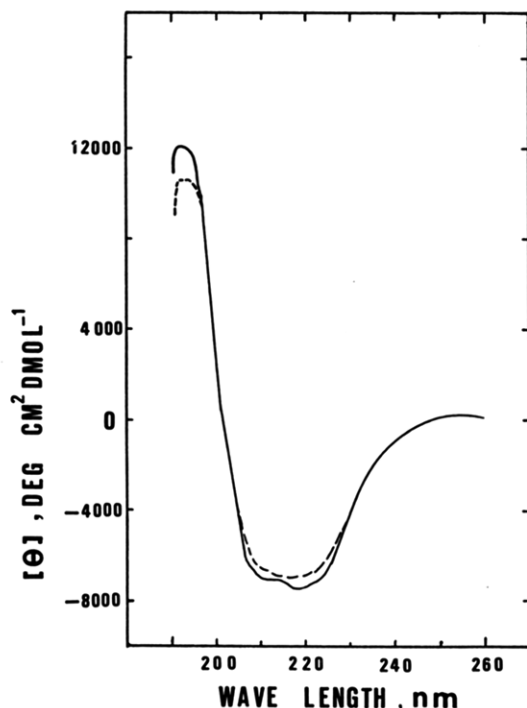


Figure 5. CD spectrum of nonheated and heated DEW in the dry state: —, nonheated; ---, heated for 5 days.

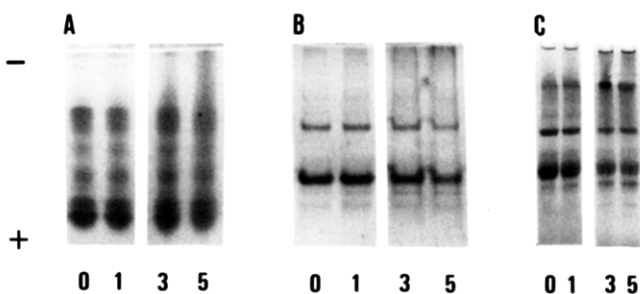


Figure 6. Electrophoretic patterns of heat-treated DEW at 80 °C in the dry state for various periods of time: (A) 7% polyacrylamide gel electrophoresis patterns; (B) SDS-polyacrylamide gel electrophoresis patterns in the presence of 2-mercaptoethanol; (C) SDS-polyacrylamide gel electrophoresis patterns in the absence of 2-mercaptoethanol. Key: 0, nonheated sample; 1, heated sample for 1 day; 3, heated sample for 3 days; 5, heated sample for 5 days.

white proteins by heating in dry state for 5 days. The SDS-polyacrylamide gel electrophoresis was performed in order to assess the binding type of this aggregate of heat-treated DEW. From the SDS-polyacrylamide gel electrophoresis patterns in the presence of 2-mercaptoethanol (Figure 6B), there are no changes in the bands at any heating time, indicating that the soluble aggregate was already dissociated by SDS and 2-mercaptoethanol. But when SDS-polyacrylamide gel electrophoresis was performed in the absence of 2-mercaptoethanol (Figure 6C), the bands with the highest staining intensity in the heated DEW considerably decreased. However, some of the aggregates remained undissociated. In light of this fact, the type of binding is likely to be hydrophobic interaction and it might partially result from disulfide bonds between egg white proteins. This result suggests that sulfhydryl-disulfide interchange reactions occurred during heating in the dry state.

Table II shows the changes in the free amino groups on the heat-treated DEW in the dry state for various periods of time. No significant changes in the free amino groups were observed at any heating time. This suggests that

Table II. Changes in Free Amino Groups of Heat-Treated DEW at 80 °C for Various Periods of Time

heating time, days	absorbance at 420 nm ^a	heating time, days	absorbance at 420 nm ^a
0	0.39 ± 0.03	5	0.39 ± 0.04
1	0.38 ± 0.03	7	0.37 ± 0.03
3	0.37 ± 0.04	10	0.37 ± 0.04

^aThe values of absorbance indicate the color development with TNBS reagent. Reported values are means ± standard deviations, *n* = 4.

controlled heating in the dry state used in this experiment apparently avoid the occurrence of browning reactions.

DISCUSSION

It was found that controlled heating in the dry state was an effective method to improve the functional properties of DEW without loss of solubility. The optimal functional properties of DEW were obtained by heating at 80 °C in 7.5% moisture content for 10 days. No detrimental effects on the emulsifying, foaming, and gel properties were observed within this heating time, while the solubility began to decrease slightly upon heating after 7 days. Hence, heating for 10 days in the dry state is proposed to gain the maximum functional properties of DEW. What kind of changes occurred in structure of DEW proteins by heating in dry state is an interesting problem. A slightly change in the CD spectrum and a significant increase in surface hydrophobicity suggest mild conformational changes in DEW proteins. The "molten" structure that is partially unfolded and more flexible than the native form may be formed by a controlled heating in the dry state. In addition, protein-protein interactions due to sulfhydryl-disulfide interchange were also involved in DEW proteins by heating in the dry state, as shown in electrophoresis patterns. The surprising result is that water solubility of DEW was not affected with increased heating time, while surface hydrophobicity was significantly increased, suggesting that the hydrophile and lipophile were balanced by this heat treatment. Thus, heating of DEW proteins in the dry state seems to enhance the hydrophobic residues to occupy a large area at the surface of protein molecules. Therefore, the formation of hydrophobic and disulfide protein-protein interaction was revealed in the electrophoresis data of heated DEW in the dry state. We reported a positive correlation is observed between the surface hydrophobicity and emulsifying properties (Kato and Nakai, 1980). Thus, the improvement of emulsifying properties is elucidated by the increase in surface hydrophobicity. The important structural factors for foaming properties are reported to be protein flexibility (Kato et al., 1985) and protein-protein interaction (Kato, 1984), in addition to surface hydrophobicity. Since these structural changes occurred in DEW with heating in the dry state, the foaming properties may be improved. However, the reason of the improvement of gelling properties is hard to elucidate, although it is the most interesting phenomenon. It is well-known that the gelling properties can be affected by the cross-linkages of the unfolded molecules containing hydrogen bond, ionic, and hydrophobic interactions. Therefore, it suggests that the "molten" structure may strengthen these cross-linkings. In addition, the increase of cross-links with disulfide bonds may be critical for gel formation. A possible elucidation is that the exposed and reactive residues of the molten molecules, in addition to disulfide bonds, are situated in the appropriate positions to form a strong and stable gel matrix by the interaction between proteins during heating for gelation, while the reactive residues of the native molecules may remained

buried in the interior of protein molecules.

Further studies are needed to elucidate more closely the factors responsible for the improvement of gelling properties of DEW proteins when heated in the dry state.

LITERATURE CITED

- Caribaldi, J. A.; Donovan, J. W.; Davis, J. G.; Cimino, S. L. Heat Denaturation of the Ovomucin-Lysozyme Electrostatic Complex—A Source of Damage to the Whipping Properties of Pasteurized Egg White. *J. Food Sci.* 1968, 33, 514-524.
- Chang, P.; Powrie, W. D.; Fennema, O. Disc Gel Electrophoresis of Proteins in Native and Heat-Treated Albumin, Yolk, and Centrifuged Whole Egg. *J. Food Sci.* 1970, 35, 774-778.
- Cunningham, F. E. Changes in the Egg White during Incubation of the Fertile Egg. *Poultry Sci.* 1974, 53, 1561-1565.
- Cunningham, F. E.; Lineweaver, H. Stabilization of Egg-White Proteins to Pasteurizing Temperatures above 60°C. *Food Technol.* 1965, 19, 1442-1447.
- Cunningham, F. E.; Lineweaver, H. Inactivation of Lysozyme by Native Ovalbumin. *Poultry Sci.* 1967, 46, 1471-1477.
- Kato, A. Functionality of Heat-Denatured Ovalbumin. *New Food Ind.* 1984, 26, 69-82.
- Kato, A.; Nakai, S. Hydrophobicity Determined by A Fluorescence Probe Method and Its Correlation with Surface Properties of Proteins. *Biochem. Biophys. Acta* 1980, 624, 13-20.
- Kato, A.; Takahashi, A.; Matsudomi, N.; Kobayashi, K. Determination of Foaming Properties of Proteins by Conductivity Measurements. *J. Food Sci.* 1983, 48, 62-65.
- Kato, A.; Fujishige, T.; Matsudomi, N.; Kobayashi, K. Determination of Emulsifying Properties of Some Proteins by Conductivity Measurements. *J. Food Sci.* 1985a, 50, 56-58, 62.
- Kato, A.; Komatsu, K.; Fujimoto, K.; Kobayashi, K. Relationship between Surface Functional Properties and Flexibility of Proteins Detected by the Protease Susceptibility. *J. Agric. Food Chem.* 1985b, 33, 931-934.
- Laemmli, U. K. Cleavage of Structural Proteins during the Assembly of the Head of Bacteriophage T4. *Nature* 1970, 227, 680-685.
- Miller, G. L. Protein Determination for Large Numbers of Samples. *Anal. Chem.* 1959, 31, 964.
- Nakai, S.; Ho, L.; Helbig, N.; Kato, A.; Tung, M. A. Relationship between hydrophobicity and Emulsifying Properties of Some Plant Proteins. *J. Inst. Can. Sci. Technol.* 1980, 13, 23-27.
- Seideman, W. E.; Cotterill, O. J.; Funk, E. M. Factors Affecting Heat Coagulation of Egg White. *Poultry Sci.* 1963, 43, 406-417.

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Persistence, Movement, and Degradation of Glyphosate in Selected Canadian Boreal Forest Soils

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Persistence, mobility, and degradation studies of glyphosate, *N*-(phosphonomethyl)glycine, under actual field conditions of boreal forest soils of Ontario, were undertaken after spraying Roundup at the rate of 2 kg of active ingredient (AI)/ha. Soils at three depths were collected and analyzed for residues of glyphosate and its metabolite (aminomethyl)phosphonic acid. Glyphosate was found to remain consistently to a level below 50% of the highest residue values observed beyond 24 days. More than 95% of the total herbicide residue was found in the upper organic layer at any time. There was no evidence of lateral movement of the glyphosate either in runoff water or through subsurface flow. In general, concentrations of the metabolite (aminomethyl)phosphonic acid were very low.

The herbicide glyphosate (GLYPH), *N*-(phosphonomethyl)glycine (Monsanto's Roundup), is an environmentally safe broad-spectrum herbicide having a potential for use in silvicultural programs such as site preparation, conifer release, and nursery stock production. This herbicide has been recommended for use in agricultural as well as forestry substrates in Ontario. Studies on the behavior of glyphosate in or on soil have been reported (Torstensson, 1982; Stark, 1982; Salazar and Appleby, 1982; Torstensson and Stark, 1979, 1981; Rueppel et al., 1977; Torstensson and Aamisepp, 1977; Sprankle et al., 1975a,b). However, inadequate data exist as to its behavior under boreal forest conditions in Ontario, and hence this study was undertaken.

EXPERIMENTAL SECTION

Reagents. Glyphosate (98%) and (aminomethyl)phosphonic acid (AMPA) (94%) were supplied by Mon-

santo Chemical Co. Trifluoroacetic anhydride and trifluoroethanol were purchased from Aldrich Chemical Co. Anhydrous sodium sulfate was heated at 140 °C overnight prior to use. All organic solvents used were pesticide grade (Caledon Laboratories, Georgetown, Ontario, Canada).

Location and Experimental Design. One sand site for persistence and leaching studies and one clay site for mobility study were selected. The sand site was part of a recently planted jack pine plantation that also contained the occasional blueberry plants. The clay site was in an open cutover covered by weeds and the occasional remnant of the original forest, namely white birch, black spruce, and poplar. The sand and clay sites were located in Harker (48°30' N, 79° W) and Lamplugh (48°35' N, 79° W) townships, respectively, about 40 km east of Matheson in the district of Cochrane, Ontario. Each site (20 m × 20 m) was divided into five replicate strips separated by buffer zones (1 m × 20 m). Each strip (2 m × 20 m) was further subdivided into 10 squares (2 m × 2 m) as sampling plots.

Site Preparation. All dead wood, live brush, and as much vegetation as possible were manually removed from the site with minimal disturbance of the duff layer (5-10 cm in depth). For the mobility study, dead wood and other

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