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The Effect Of Two Flame Retardants On Particulate And Residue Production

U. S. DEPT. OF AGRICULTURE
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PRODUCTION OF PARTICULATE AND RESIDUE
CURRENT STATUS AND TRENDS

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ABSTRACT

The benefits from fire use — including hazard reduction, silvicultural manipulation, pathogen control, and nutrient recycling — might be forfeited by public reaction to smoke, whether harmful or not. Generally, the public desires alternatives to burning, but might accept fire if direct control of emissions were possible. The effects of diammonium phosphate (DAP) and ammonium sulfate (AS)

on particulate emission and fire intensity were investigated. Particulate emission rate and total quantity were increased by DAP to several times those produced from the control. Inversely, very little increase was observed for AS. Since burning rate for AS-treated fuels was also decreased, this retardant might be useful in smoke control through extension of burning periods.

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FOREWORD

Pressure and criticism currently are mounting against open burning of forest debris in the West because of particulate emissions and their visual impact. This controversy has sparked much speculation and some research on alternatives to burning, including better utilization and mechanical treatment of slash. Because fire might be a requirement for future productivity of the site, it might be important to look at the characteristics of fire itself; specifically, in this study particulate production rates and total amount of particulate.

Emissions from slash fires include water vapor, gases, and particulate. The particulate includes both char particles and tar droplets. Several variables are involved in the descrip-

tion of particulate emissions: Time and rate of production, total quantity, size distribution, tar composition, and color. Emissions during the smoldering phases of the fire pose a problem in areas of nighttime down-canyon winds and early morning inversions. On the other hand, if atmospheric conditions are stable during the initial stages of the fire, production rate might become the critical factor. Because the size of the particle determines its settling rate, this variable is important under some conditions. Moreover, the fraction of tar in the particulate becomes important when considering the ramifications of smoke emissions, because it might be related to health problems.

Effects of Flame Retardants

Generally speaking, flame retardants decrease the maximum rate of combustion and increase the production of char, which is a portion of the airborne particulate from wood fires. Another source of particulate is probably the tar fractions. A retardant that enhances char and tar production will produce more particulate if it does not in some way also enhance the more complete combustion of the char by glowing. Past work has shown that most flame retardants increase CO, CO₂, and H₂O produced from the pyrolysis of wood and cellulosic fuels.¹

The pyrolysis and combustion of cellulosic fuels can be graphically expressed as involving two competitive pathways. The low energy pathway, which occurs at temperatures around 270° C. or less for cellulose, produces char and relatively large amounts of water and carbon dioxide. The char usually sustains glowing combustion. The high energy path-

way, which occurs at 340° C. or higher for cellulose, produces flammable volatiles; these support flaming combustion and produce little char or residue.

The effect of most pyrolytic flame retardants is to emphasize the low energy pathway at the expense of flaming. This is generally characterized by the lowering of the temperature of pyrolysis and an increase in char. The enhancement of the "glowing" mechanism at the expense of "flaming" does not mean there will be a decrease in residue following the fire. There may be a relative increase in energy released by way of the low energy pathway, but the total energy released may be decreased because of the residue produced. Whether the residue sustains glowing or not depends upon the chemical composition of the retardant used and the environmental conditions. This is not readily apparent if one considers only pyrolysis. When combustion is included, the picture becomes complicated by the possibility of a retardant catalyzing the glowing combustion of the residue as compared to another equally effective retardant that might alter some other combustion phenomenon.

¹ F. Shafizadeh. *Pyrolysis and combustion of cellulosic materials*. P. 419-474, in: *Advances in Carbohydrate Chemistry*. R. Stuart Tipson, Ed., Vol. 23. New York and London: Academic Press. 1968.

Retardants and Smoke Production

Laboratory studies have shown that some retardants, such as diammonium phosphate (DAP), apparently polymerize the tars.² These polymerized tars might lead to the increase in smoke. For example, Eickner and Schaffer³ found that the most effective fire retardant chemicals, DAP and ZnCl_2 , greatly increased smoke production in tests on

Douglas-fir plywood. Other chemicals tested showed a decrease in smoke density. They also found that chemicals previously suspected of promoting postglowing reactions, such as sodium dichromate and ammonium sulfate, also reduced smoke index values. Similar results using particle board were reported by Syska.⁴

Diammonium Phosphate and Ammonium Sulfate

The flame retarding effects of DAP and ammonium sulfate (AS) on rate of spread and intensity for wood and fine fuel fires have been tested under controlled conditions.⁵ Findings show DAP to be most effective in slowing spread and reducing intensity. Two other differences between DAP and AS are apparent. The residue remaining after the tests is always much less from the AS treat-

ment. For example, the residue from AS treated excelsior fuel usually is close to non-existent (fig. 1). The other difference is the tremendous amount of black smoke produced by DAP treatment as compared to the much smaller amount of whitish smoke produced by the AS treatment.

² Thermogravimetric data on file at the Northern Forest Fire Laboratory, Missoula, Montana.

³ H. W. Eickner and E. L. Schaffer. Fire-retardant effects of individual chemicals on Douglas-fir plywood. *Fire Technol.* 3: 90-104, illus. 1967.

⁴ Arthur D. Syska. *Exploratory investigation of fire-retardant treatments for particle board.* USDA Forest Serv. Res. Note FPL-0201, 32 p. 1969.

⁵ Charles W. George and Aylmer D. Blakely. *Effects of ammonium sulfate and ammonium phosphate on flammability.* USDA Forest Serv., Intermountain Forest and Range Exp. Station, Ogden, Utah (in preparation).

Figure 1. — The relationship between residue from spreading excelsior fires and treatment level.

EXPERIMENTAL DESIGN

The Fuel Cribs

This study was conducted under controlled laboratory conditions using a standard fuel crib, which meant we could control the various environmental and fuel factors and their influence on particulate production and energy-release rate. The cribs were conditioned to a known moisture content and chemical treatment was accurately computed. The flow and volume characteristics of the effluent gases were measured and the total convection column accurately subsampled isokinetically. This type of control is difficult to achieve under field conditions.

The cribs were constructed using about 20 pounds (9 kg.) of 3/4- by 3/4-inch (1.9-cm.) ponderosa pine (*Pinus ponderosa*) sticks, 24-3/4 inches (62.9 cm.) long. These were nailed together with 1-1/4-inch (3.2-cm.) wire brads in a configuration that would insure good combustion and allow insertion of five 24-inch (61-cm.) Douglas-fir (*Pseudotsuga menziesii*) 4 by 4's (10.2 cm. by 10.2 cm.). The average dry weight of a crib and 4 by 4's,

excluding nails, was 44.0 lbs. (20.0 kg.) (tables 1A and 1B). Inasmuch as each crib had a basal area of 4.25 ft.² (0.39m²) and a loading of 10.4 lb. ft.⁻² (50.8 kg. m⁻²), they were equivalent to 227 tons acre⁻¹ (508,000 kg. hectare⁻¹). The approximate heat content of each crib was 8,500 B.t.u. lb.⁻¹ (4,722 cal. g.⁻¹) or 3.74 by 10⁵ B.t.u. (9.42 by 10⁷ cal.).

The 4 by 4's were conditioned in a sealed cabinet maintained at 90° F. (32.2° C.) and a relative humidity of 91 percent. The humidity was held constant by the use of saturated potassium nitrate solutions. Three sample 4 by 4's from different areas of the conditioning cabinet were weighed daily and sampled for actual moisture content.

The cribs were conditioned in a room maintained at 78° F. (25.6° C.) and approximately 30 percent relative humidity; the latter was maintained using a mechanical humidifier. The cribs were weighed daily and moisture contents were determined from sticks identical to those used to build the cribs.

Treatment

A calibrated spray apparatus was used to apply 10, 20, 30, or 40 percent solutions of each chemical to the cribs and the 4 by 4's. The preweighed fuel was placed on a dolly and run back and forth under the sprayer until completely coated with retardant. The

highest treatment levels were achieved by alternating several hours of drying with spraying. The exact amount of chemical added was calculated after each crib and 4 by 4 reached a constant weight in their respective conditioning area (table 1).

Table 1. — The weight, moisture content, and chemical treatments of the cribs

Crib weights (grams)			Percent chemical		Percent moisture		
Stick size		Total	Stick size		Stick size		Total
4 in.	¾ in.		4 in.	¾ in.	4 in.	¾ in.	
Untreated							
10,737	9,098	19,835	16.3	5.8	*	*	*
10,509	8,908	19,417	20.2	6.8	*	*	*
9,938	8,585	18,523	20.2	6.8	*	*	*
10,480	8,699	19,179	15.3	5.5	*	*	*
Diammonium Phosphate Treated							
10,092	9,102	19,194	19.4	6.8	0.0	0.7	0.4
10,645	9,069	19,723	13.9	6.2	.2	.7	.4
11,964	8,853	20,822	15.3	5.5	.1	1.0	.5
9,996	9,011	19,007	19.3	6.3	.5	1.3	.9
10,762	9,101	19,953	15.7	5.7	.8	2.3	1.4
11,215	9,526	20,741	16.0	5.9	2.4	3.1	2.7
10,268	8,856	19,142	20.0	6.6	1.7	5.6	3.5
10,345	8,861	19,206	16.3	5.6	1.3	5.6	3.3
11,464	9,677	21,141	16.1	6.2	2.3	7.6	4.7
11,629	9,007	20,706	16.0	5.9	.7	8.4	4.0
10,925	9,112	20,037	15.8	6.0	1.5	9.9	5.3
11,026	8,528	19,554	17.0	5.3	2.4	14.8	7.8
Ammonium Sulfate Treated							
11,303	8,915	21,218	15.5	5.3	.1	.5	.2
11,020	8,692	19,712	13.6	5.9	.2	1.0	.5
11,388	8,668	20,056	13.7	6.1	.3	1.1	.7
11,238	9,376	20,614	17.0	4.7	1.7	1.2	1.5
10,290	9,093	19,179	16.9	5.9	.4	1.9	1.1
10,845	9,284	20,129	15.7	5.3	.4	2.6	1.4
10,005	9,360	19,365	15.6	5.8	.7	3.4	2.2
10,576	9,270	20,026	16.3	6.3	1.8	4.9	3.0
10,783	9,302	20,085	16.9	5.7	.6	5.6	2.9
12,224	9,289	21,513	16.8	6.0	.6	6.8	3.3
11,646	8,823	20,469	15.8	5.9	1.7	8.3	4.5
10,877	10,077	20,954	16.8	6.4	2.2	8.9	5.4
11,037	8,847	19,884	18.1	6.6	2.1	11.3	6.2
11,136	9,129	20,265	16.7	4.3	1.8	11.9	6.6

Burning Procedures

The cribs were burned on a table placed on a weighing system similar to that described by George and Blakely⁶ (fig. 2). Two methods were used to control temperature buildup around the load cells. A layer of Zonolite insulation 3 inches (7.6 cm.) deep was placed between the fuel and the weighing system. This material was found to be thermally stable up to 1,500°C. (2,700°F.); it is not hygroscopic. Streams of compressed air were continuously directed around the load cells during the tests as a further safeguard against temperature changes that could cause electronic drift.

The procedure for burning began by conditioning the combustion chamber to a temperature of 90° F. (32.2° C.) and a relative humidity of 20 percent. A slight positive pressure was maintained to insure removal of all of the effluent through the stack. The induced convection had no measurable effect on the burning characteristics of the fires. The test cribs, including the 4 by 4's, were placed on the weighing system and ignited at time zero using 100 ml. of a 50:50 mixture of ethanol and acetone contained in two trays. During the following 60 minutes, weight loss and radiant flux from the flame zone were continuously recorded and flame heights were photographed at 1-minute intervals. A Gier and Dunkle directional radiometer with a

15°-view angle placed 20 feet (6.1 m.) from the fire was used to measure radiant flux.

After the fire went out, the residues from the 4 by 4's and the cribs were collected and the dry weight calculated.

Figure 2. — A schematic representation of the combustion facilities. (Note: not drawn to scale.)

Particulate Sampling

The particulate sampling system consisted of a Neucleonic Corporation of America Model HAS 69 high volume sampler and Gelmen type A glass filter paper. The sampler was connected to a piece of pipe 4 inches (10 cm.) in diameter; this was placed (as shown at top of figure 2) in the center of the effluent stack approximately 50 feet above the fire. The end of the pipe was sealed and four holes

were drilled on the underside. The holes decreased in size from the center of the stack outward so that the effluent cross section could be sampled equally.

The flow rate through the sampler was found to be temperature dependent. Therefore, gas temperature was measured and recorded at the sampler during a run. Calibration curves were used to obtain actual flow rate. The flow rate up the stack was determined using a United Sensor and Control Corporation Model PAE-36-M-W pitostatic probe

⁶ Charles W. George and Aylmer D. Blakely. Energy release rates in fire retardant evaluation. *Fire Technol.* 6: 203-210, illus. 1970.

with an electrical readout. The temperature of the stack gases was measured using a chromel alumel thermopile. Data from the pitostatic probe and the thermopile were used to give stack flow rate as a function of time. This information, along with the sampling rate, allowed us to determine what percentage of the total effluent from the fire was being subsampled at any given time.

Filter papers were dried and preweighed before a test fire. A filter paper was placed in the particulate sampler; upon ignition, the sampler was turned on and flow rate and time were recorded. These readings were recorded at intervals corresponding to a drop in flow rate of $1 \text{ ft.}^3 \text{ min.}^{-1}$ or 5 minutes, whichever occurred first. A given filter paper was changed when the indicated flow rate was reduced to $5 \text{ ft.}^3 \text{ min.}^{-1}$ ($0.142 \text{ m.}^3 \text{ min.}^{-1}$). The changes involved a standard 1-minute shutdown time. This procedure was continued for 60 minutes.

Contaminated filters were dried and weighed to obtain particulate deposited during the time the filter was in place. Sample flow rate was corrected to standard conditions and then corrected to experimental conditions. The average flow rate and the total flow between readings were then computed.

The sums of the flows for each filter were divided into the particulate accumulated on the filter to give particulate density (mg.m.^{-3}) as a function of time. The densities between filters were interpolated from the adjacent filter data. The rate of particulate emitted and the total particulate produced by the fire were obtained by relating the data from the sampler back to stack conditions.

The approximate tar content of the particulate was determined by extraction of filter paper subsamples using tetrahydrofuran. The elemental phosphorus content of the particulate from the DAP treated samples was determined using acid digestion and colorimetry. Water soluble sulfate present in the particulate was determined turbidimetrically using Sulfaver, a product of Hach Chemical Company, Ames, Iowa.⁷

The data were statistically analyzed using Program SCRAP (6.0.003) on an IBM 1620 computer.

⁷Norman A. Huey. *Determination of sulfate in atmospheric particulate: turbidimetric barium sulfate method*. P. 11-14, in: *Selected Methods for the Measurement of Air Pollutants*. R. A. Raft, Ed. USDHEW Public Health Serv., Cincinnati, Ohio. 1965.

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Treatment Effect on Thermal Variables

The effect of the retardants on the burning characteristics of the crib fires is quite apparent. Both AS and DAP lowered the maximum weight loss rate, flame height, and radiant energy output. The effect of DAP treatment level on weight loss rate is shown in figure 3 and that for AS level is shown in figure 4. As can be seen, the increase in treatment level lowers the weight loss rate and increases the time to reach maximum intensity. Treatment level refers to the percent of chemical on the cribs, excluding the 4 by 4's. The graphs of the equations for treatment against maximum

radiation are shown in figures 5 and 6. The DAP treatments are most effective in both cases. The difference between types of chemicals is significant at the 99 percent level as determined by the F test (table 2).

The sequential photographs in figure 7 clearly show the effects of the two chemicals on intensity: buildup time is increased and the intensity is decreased (indicated by flame size); DAP was the most effective. Figure 8 shows the three fires at the time of maximum intensity and the residue left following active combustion.

Effect on Residue

The residue remaining following the fire was related to the type of treatment (fig. 9). The total residue and DAP treatment level had a correlation coefficient R^2 of 0.87; the R^2 for AS was 0.21. However, the effects of

the two types of treatment were statistically different at the 99 percent level. The residue from the 4 by 4's alone was found not to be significant, both within and between types of treatments.

Figure 3. — Weight loss rates relative to time for several levels of DAP treatment.

Figure 4. — Weight loss rates relative to time for several levels of AS treatment.

Figure 5. — The relationship between maximum weight loss rate and treatment level.

Figure 6. — The relationship between maximum radiant flux and treatment level.

Table 2. — Regression analysis and statistical significance of various test fire parameters

Parameters	Treatment	N	Equation	R ²	F	Significance
						Percent
Treatment level versus maximum weight loss rate	DAP	16	$Y = 1.833 - 183.4x + 8.27x^2$	0.94		
	AS	18	$Y = 1.840 - 123.1x + 6.39x^2$	.76		
	Pooled	34	$Y = 1.828 - 139.8x + 6.19x^2$	.76	10.01 (F=3,28)	99
Treatment level versus maximum flame height	DAP	16	$Y = 10.55 - .9143x + .0391x^2$	.84		
	AS	18	$Y = 10.62 - .4601x + .0089x^2$	.79		
	Pooled	34	$Y = 10.60 - .6778x + .0240x^2$	.75	4.10 (F=3,28)	95
Treatment level versus maximum radiation	DAP	16	$Y = 1.382 - .1148x + .0041x^2$	.95		
	AS	18	$Y = 1.402 - .0304x - .0304x^2$	.69		
	Pooled	34	$Y = 1.388 - .0638x + .0013x^2$	.65	23.50 (F=3,28)	99
Treatment level versus crib residue	DAP	17	$Y = 2.495 - .576x$	.87		
	AS	19	$Y = 2.179 + .105x$	.21		
	Pooled	36	$Y = 2.258 + .345x$	.42	40.42 (F=2,32; improvement)	99
Treatment level versus 4 by 4 crib	DAP	17	$Y = 11.42 - 5.392x$	<.01		
	AS	19	$Y = 12.55 - .110x$	.01		
	Pooled	36	$Y = 12.00 - 8.071x$	.01	3.46 (F=2,32; improvement)	N.S.
Treatment level versus total particulate	DAP	11	$Y = 53.36 + 59.55x - .715x^2$	.98		
	AS	12	$Y = 41.83 + 3.83x - .216x^2$	.36		
	Pooled	23	$Y = 83.08 + .706x + 1.97x^2$	.32	231.00 (F=3, 17)	99
Treatment level versus maximum particulate rate	DAP	11	$Y = 6.148 + 4.953x - .123x^2$	.88		
	AS	12	$Y = 2.611 + .487x - .035x^2$	.36		
	Pooled	23	$Y = 7.077 + .451x + .095x^2$	.22	63.00 (F=3,17)	99

Figure 7. — Sequential photographs showing the burning of: A, An untreated crib; B, a crib treated with 4.55 percent AS; and C, a crib treated with 4.05 percent DAP.

MAXIMUM FLAME HEIGHT

UNTREATED

A S-4.55%

D A P-4.05%

7

16

27

TIME SINCE IGNITION

(minutes)

RESIDUE

43

60

86

Figure 8. — Photographs of: A, Untreated crib; B, a crib treated with 4.55 percent AS; and C, a crib treated with 4.05 percent DAP, showing maximum flame height and time of occurrence and residue following active combustion.

Figure 9. — The relationship between crib residue and treatment level.

Effect on Particulate

The relationship between weight loss rate strikingly different between the two chemicals. Figure 10 presents the total accumulated particulate produced from several fires. Obviously, the AS treatment had little effect on particulate production, while DAP produced substantial increases in particulate; about 20 times more than that produced by the untreated at the 14.8 percent treatment level. The rate of particulate is increased and the peak rate occurs further from the point of ignition as the amount of DAP increases (fig. 11). The range of particulate produced per unit of weight consumed was 8 to 94 lb.ton⁻¹ for DAP, 3.8 to 7.2 lb.ton⁻¹ for AS, and about 5.5 lb.ton⁻¹ for the controls, as shown in table 1.

The relationship between weight loss rate and particulate production rate is clearly shown in figures 12 and 13. As can be seen, intensity is decreased by both chemicals.

However, there is little change in particulate production by AS.

The range in tetrahydrofuran soluble tar was 41 to 56 percent (dry weight of total particulate) for DAP treatment, 15 to 58 percent for AS treatment, and 40 percent for controls. The amount of tar was apparently directly correlated with DAP treatment and inversely correlated with AS treatment level. The phosphorus content of the DAP particulate was 11 to 17 percent, depending on treatment level. Preliminary estimates show that about 30 percent of the phosphorus added by treatment is emitted by the fire and can be collected with the particulate.

The particulate matter from the AS treated fires was found to contain 33 to 57 percent sulfate, depending on treatment level. However, this did not amount to over 10 percent of the sulfate added by treatment at the highest level.

Figure 10. — The total particulate produced from fires at several treatment levels.

Figure 11. — The production rate of particulate relative to time for various levels of DAP treatment.

Figure 12. — Weight loss rate and particulate production rate relative to time for three DAP treatment levels and the control.

Figure 13. — Weight loss rate and particulate production rate relative to time for three AS treatment levels and the control.

DISCUSSION AND CONCLUSIONS

Two flame retarding chemicals, DAP and AS, reduced the intensity of large wood crib fires. The DAP treatments were somewhat more effective. However, DAP greatly increased particulate production. The AS treatments had much less effect on particulate formation. Total organic residue was increased by DAP treatment; it amounted to as much as 14 percent original organic weight.

As conditions for slash burning are presently dictated from a control standpoint, it is being done at low intensities and at times when weather conditions are not conducive to minimum air pollution. This burning results in large amounts of smoke, poor fuel consumption, and public displeasure. It might be possible to control intensity during the drier months, keep smoke production down, and insure more complete combustion by chemically treating the slash. Obviously, DAP would not do the job.

This study supports the possibility that

DAP does polymerize the tars and make them more thermally stable. If these tars become less available to combustion, they will add to the particulate in the effluent. Apparently, a large amount of the phosphate ends up as some form of phosphorus in the particulate. The question of why AS and DAP act differently in particulate formation might partially be answered by continued study of the effects of phosphate on the tars.

Future research starting with determination of the difference in retarding mechanisms (and therefore in particulate and residue production) between AS and DAP could lead to the development of chemicals that catalyze glowing to the point of practically no flaming or effluent.

The importance of fire in the forest is becoming more evident every day. Perhaps future research and land management personnel should be directed more toward fire controls rather than to "alternatives to fire."

Headquarters for the Intermountain Forest and Range Experiment Station are in Ogden, Utah. Field Research Work Units are maintained in:

Boise, Idaho

Bozeman, Montana (in cooperation with Montana State University)

Logan, Utah (in cooperation with Utah State University)

Missoula, Montana (in cooperation with University of Montana)

Moscow, Idaho (in cooperation with the University of Idaho)

Provo, Utah (in cooperation with Brigham Young University)