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Development of Emission Factors for Ethylene-Vinyl Acetate and Ethylene-Methyl Acrylate Copolymer Processing

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ABSTRACT

Emission factors for selected volatile organic compounds (VOCs) and particulate emissions were developed over a range of temperatures during extrusion of three mixtures of ethylene-vinyl acetate (EVA) copolymers and two mixtures of ethylene-methyl acrylate (EMA) copolymers. A mixture of low-density polyethylene (LDPE) resins was used as a control. EVAs with 9, 18, and 28% vinyl acetate (VA) were used. The EMA mixtures were both 20% methyl acrylate. A small commercial extruder was used. Polymer melt temperatures were run at 340 °F for LDPE and both 18 and 28% EVAs. The 9% EVA mixture was extruded at 435 °F melt temperature. The EMA mixtures were extruded at 350 and 565 °F melt temperatures.

An emission rate for each substance was calculated, measured, and reported as pounds released to the atmosphere per million pounds of polymer processed [ppm (wt/wt)]. Based on production volumes, these emission factors

can be used by processors to estimate emission quantities from EVA and EMA extrusion operations that are similar to the resins and the conditions used in this study.

INTRODUCTION

Industry is faced with a new challenge. Pursuant to the Clean Air Act Amendments (CAAA) of 1990, which mandated the reduction of various pollutants released to the atmosphere, companies are being faced with the daunting task of establishing "emission inventories" for the chemicals used in their processes. The chemicals targeted are those that produce either volatile organic compounds (VOCs) or compounds that are on the list of 189 hazardous air pollutants (HAPs). Title V of the amended Clean Air Act established a permit program for emission sources to ensure an eventual reduction in emissions. When applying for a state operating permit, processing companies are first required to establish a baseline of their potential emissions.¹

In response to the needs of the plastics industry, the Society of the Plastics Industry, Inc. (SPI) organized a study to determine the emission factors for ethylene-vinyl acetate (EVA) and ethylene-methyl acrylate (EMA) extrusion. Sponsored by four major resin producers, the study was performed at Battelle, an independent research laboratory. This work follows two previous SPI-Battelle studies on the emissions of polyethylene² and polypropylene.³

IMPLICATIONS

This study provides quantitative emissions data collected during extrusion of ethylene-vinyl acetate (EVA) and ethylene-methyl acrylate (EMA) copolymers under specific operating conditions. These data can be used by processors as a point of reference to estimate emissions from similar EVA/EMA extrusion equipment based on production volumes.

A review of the literature shows that, while there are some qualitative and quantitative data available on polyethylene thermal emissions, there are fewer studies that mention EVA and EMA. The primary concern about previous polyethylene emissions data is that they were generated using static, small-scale,⁴ or otherwise unspecified procedures.^{5,6}

In the design stages of this and previous SPI-Battelle studies, considerable attention was given to whether the model used accurately reflected real processing conditions. The major contributing factors to the rate of emissions in an extrusion process were considered to be temperature, exposure to oxygen, and residence time. The goal was to reflect the actual on-line processing conditions rather than a static situation. In most extruders, the polymer melt continuously flows through the system, effectively limiting the residence time in any particular heated zone. If a static set-up were studied, the polymer may be exposed to the equivalent temperatures but for a longer period of time. This would effectively exaggerate the thermal exposure of the polymer. In a similar way, the concern over oxygen in the industrial extrusion process is minimized as the extruder screw design forces entrapped air back along the barrel during the initial compression and melting process. The air then exits the system through the hopper. Therefore, the hot polymer is exposed to air only when it is actually extruded through the die. In some of the static testing that has been reported, the hot polymer may have been exposed to air for extended periods of time.

The ideal would seem to be to measure the emissions directly from each individual process. In extrusion, for example, the type and quantity of emissions are known to be influenced by a number of operational parameters, including extruder size and type, extrusion temperature and rate, the air-exposed surface-to-volume ratio of the extrudate, the cooling rate of the extrudate, and the shear effect from the extruder screw. All of these would have to be specified and controlled.

Table 1. Average additive concentration (ppm) in polymer mixtures.

	SLIP	ANTI-BLOCK	ANTIOXIDANT
EVA			
18% VA	0	0	138
28% VA	0	0	263
9% VA	300	1500	145
EMA			
20% MA/3 MI	0	0	250
20% MA/6 MI	0	0	250
LDPE			
	156	300	340

The objective of the SPI-Battelle study was to take representative EVA/EMA resins from a number of suppliers and, using the same equipment used to study both polyethylene and polypropylene, provide baseline emission data. The test conditions used will provide reasonable reference data for processors involved in similar extrusion operations. In some cases the emission factors determined in this study may overestimate or underestimate emissions from a particular process. For example, a recent 2-year study⁷ found, as would be expected, that a lower level of fume was generated by injection molding compared to extrusion-based processes in which the hot polymer is exposed to air. Therefore, professional judgment and conservative measures must be exercised when using the data for estimating emissions.

The samples used were mixtures of commercial copolymers from the sponsoring companies. The EVA mixtures, covering a range of 9 to 28% vinyl acetate, were composed of copolymers typically used in film forming, lamination, and hot-melt adhesive applications. The EMA mixtures containing 20% methyl acrylate were comprised of copolymers typically used in blown-film and extrusion coating applications. It should be noted that there are several variables related directly to the material being extruded that may influence the emissions. These variables include the age and type of resin, the additive package, and any additional materials added to the resin prior to extrusion. If a particular processor uses recycled materials, their thermal history is also an important factor. The test matrix used was designed to provide emissions data as a function of resin type and in some cases as a function of the operating temperature of the diehead assembly of the extruder. All of the EVA, LDPE, and EMA resins used were commercial grades. The average additive levels of the mixtures are shown in Table 1.

The equipment used was a small commercial extruder equipped with a 1.5-in. screw and fitted with an 8-strand die. The emissions were measured over a 30-minute period and were related to the weight of resin extruded. The emission factor for each substance measured was reported as pounds evolved to the atmosphere per million pounds of polymer processed [ppm(wt/wt)]. Processors using similar equipment can use these emission factors as reference points to assist in estimating emissions from their specific EVA-EMA application.

The 14 substances targeted for monitoring included particulate matter, total VOCs, light hydrocarbons (ethane, ethylene, and propylene), esters (vinyl acetate, and methyl acrylate), aldehydes (formaldehyde, acrolein, acetaldehyde, and propionaldehyde), ketones (acetone, and methylethyl ketone), and organic acids (formic, acetic, and acrylic acid). These are the analytes of interest, either because they are on the HAPs list, as stated earlier,

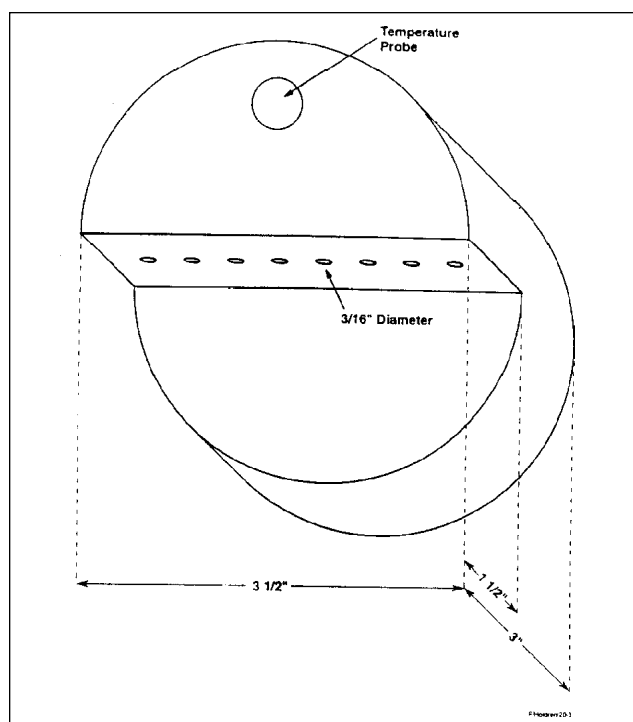


Figure 1. Extruder strand diehead used in EVA–EMA emissions testing program.

or they are the expected thermal breakdown products of the polymers tested.

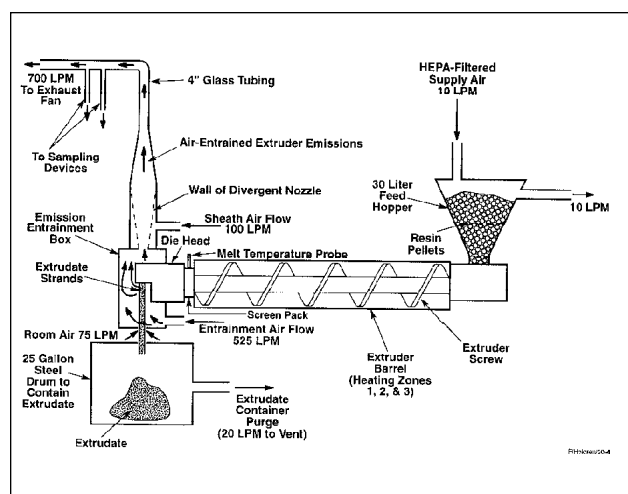


Figure 2. View of the extruder system and the various sampling locations.

EXPERIMENTAL PROCEDURES

Experimental Process Conditions

An HPM Corporation 15-horsepower unvented extruder was used to process the EVA and EMA test sample mixtures at Battelle. The extruder was equipped with a 1.5-in. single screw (L/D ratio of 30:1) and fitted with an 8-strand die (Figures 1 and 2). Extruded resin strands were allowed to flow into a stainless steel drum located directly under the

Table 2. Resin throughput and key flow parameters during the EVA and EMA extrusion runs.

TEST RUN NO.	1A	1B	2	3	4	5	6
RESIN TYPE	Low-Density Polyethylene	Low-Density Polyethylene	EVA 18% VA	EVA 28% VA	EVA 9% VA	EMA 20% MA	EMA 20% MA
EXTRUDER CONDITIONS							
Melt Flow Rate	2	2	2	6	2	2	7
Average Diehead Melt Temperature, °F	340	340	340	340	435	350	565
Zone 3 Temperature, °F	292	301	301	301	415	300	547
Zone 2 Temperature, °F	296	297	297	297	365	300	449
Zone 1 Temperature, °F	275	274	275	274	275	275	275
Pressure, psig	1300	1500	1000	750	600	1750	<50
Resin Throughput [(lb/hr) (g/min)]	28.4/215	26.9/204	34.0/257	35.7/270	34.8/263	32.8/248	35.1/265
Rotor Speed, rpm	75	75	75	75	90	75	83
Run Duration, min	30	30	30	30	30	30	30
AIR FLOWS							
Total Manifold Flow, L/min	700	700	700	700	700	700	700
Flow Rate Into Sheath Area, L/min	100	100	100	100	100	100	100
Flow Rate Into Entrainment Area, L/min	525	525	525	525	525	525	525
Flow Rate Through Hopper, L/min		10	10	10	10	10	10 10
Flow Through Tubes for Carbonyls, L/min	0.5	0.5	0.5	0.5	0.5	0.5	0.5
Flow Through Tubes for Organic Acids, L/min	5	5	5	5	5	5	5
Flow Into Canisters, L/min	0.16	0.16	0.16	0.16	0.16	0.16	0.16
Flow Through 402 THC Analyzer, L/min	1	1	1	1	1	1	1
Flow Through Filter Holder, L/min	15	15	15	15	15	15	15

Table 4. Sample collection scheme for EVA and EMA test runs.

SUBSTANCES MONITORED		Organic Acids	Aldehydes/ Ketones	Particulate	VOCs		
					HHC	LHC	
COLLECTION MEDIA	KOH Impregnated Filter		DNPH Tube	Glass Fiber Filter	SUMMA Canister		
ANALYTICAL METHOD	Desorption with Dilute H ₂ O ₄ and Analysis by Ion Exclusion Chromatography/UV		Desorption with Acetonitrile and Analysis by HPLC	Gravimetric	Modified TO-14		
					HP-1 Fused Silica Capillary Column GC/MS	Al ₂ O ₃ /Na ₂ SO ₄ Capillary Column GC/FID	
SAMPLING LOCATION							
		Manifold					
Melt Temp (°F)	Run No.	Number of Samples Analyzed					
340	1A	2	2	1	1	2	1
340	1B	2	2	1	1	2	1
340	2	2	2	1	1	2	1
340	3	2	2	1	1	2	1
435	4	2	2	1	1	2	1
350	5	2	2	1	1	2	1
565	7	2	2	1	1	2	1

Note: No processing aids were used.

Diehead emissions were transported by the 700-L/min airflow to a sampling point 10 ft. downstream of the diehead using 4-inch-diameter glass tubing. The location for this sampling point (Figure 2) was based on previous studies performed at Battelle that involved design, engineering, implementation, and proof-of-principle stages for the pilot plant system.²

Two separate sampling manifolds were used at the sampling location: one for collecting gases and vapors and the

other for collecting particulates (Figure 4). For gases and vapors, a 10-L/min substream was diverted from the main emission entrainment stream using a 0.5-in. stainless steel tube (0.425-in. i.d.) wrapped with heating tape and maintained at 50 °C. VOCs and oxygenates were sampled from this manifold. Similarly, particulates were sampled from a separate 15-L/min substream using a 0.25-in. stainless unheated steel probe (0.1375-in. i.d.).

This study did not include any measurements of emissions from the drum collection area, as all commercial extrusion processes quench the molten resin shortly after it exits the die. Emissions from the extrudate in the collection drum were prevented from entering the diehead entrainment area by drawing air from the drum at 20 L/min and venting to the exhaust duct.

VALIDATION OF THE ANALYTICAL METHOD

The purpose of the manifold spiking experiments was to determine the collection and recovery efficiencies of the canister, acid, and carbonyl collection methods. During the first spiking experiment, all three collection methods were evaluated. Results are reported in detail elsewhere.² During the second

Table 5. Results from spiking experiments.

ANALYTE	METHOD	SPIKE LEVEL $\mu\text{g/L}$	RECOVERY $\mu\text{g/L}$		AVERAGE PERCENT RECOVERED*
			Set 1	Set 2	
FIRST EXPERIMENT ^a					
Formic Acid	KOH filters	0.71	0.987	0.733	122±18
Acetic Acid	KOH filters	0.77	1.023	0.640	121±12
Acrylic Acid	KOH filters	0.59	0.687	0.567	107±11
Formaldehyde	DNPH Cartridge	1.63	2.20	2.03	130±5
Benzene-d ₆	Canister	0.092	0.088	0.086	95±2
SECOND EXPERIMENT ^b					
Benzene-d ₆	Canister	0.24	0.27	0.25	108±4
Benzene	Canister	0.22	0.22	0.22	100
Methyl Acrylate-d ₃	Canister	0.25	0.26	0.24	100±4
Methyl Acrylate	Canister	0.25	0.25	0.23	95±4
Vinyl Acetate	Canister	0.24	0.28	0.25	110±6

*Relative error is the relative percent difference: the absolute difference in the two samples multiplied by 100 and then divided by their average.

^a Reference 2; ^b Reference 3

Table 6. Summary of EVA and EMA thermal process emissions for generic resin grades (µg/g).

TEST RUN NO.	1A	1B	2	3	4	5	6
Resin Type	Low-Density Polyethylene	Low-Density Polyethylene	EVA 18% VA	EVA 28% VA	EVA 9% VA	EMA 20% MA 3 MI	EMA 20% MA 6 MI
Die Melt Temperature (°F)	340	340	340	340	435	350	565
Particulate Matter	<1	1.5	<1	<1	<1	4.1	61.5
VOLATILE ORGANIC COMPOUNDS							
Beckman 402-THC*	106.7	106.9	128.2	123.4	99.7	45.7	117.2
Heavy Hydrocarbons (HHC) 86.0		83.0	108.3	109.9	86.4	44.2	90.0
LIGHT HYDROCARBONS (LHC)							
Ethane	0.02	0.02	0.01	0.01	0.03	0.02	0.49
Ethylene	0.01	0.01	0.01	0.01	0.02	0.02	0.36
Propylene	0.01	0.01	0.01	0.01	0.01	0.01	0.14
ESTERS							
Vinyl Acetate	<0.01	<0.01	<0.01	6.22	<0.01	<0.01	<0.01
Methyl Acrylate	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
ALDEHYDES							
Formaldehyde†	0.42	0.28	0.08	0.08	0.13	0.09	1.07
Acrolein†	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.10
Acetaldehyde†	0.09	0.07	0.04	0.03	0.10	0.03	0.77
Propionaldehyde†	0.02	0.01	0.01	0.01	0.02	<0.01	0.31
Butyraldehyde	0.02	0.02	0.01	0.01	0.04	0.02	0.49
Benzaldehyde	0.02	0.02	0.03	0.05	0.05	0.03	0.23
KETONES							
Acetone	0.15	0.13	0.10	0.10	0.13	0.10	0.34
Methyl Ethyl Ketone†	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
ORGANIC ACIDS							
Formic Acid	0.27	0.22	3.85	3.11	6.05	4.40	4.66
Acetic Acid	0.44	0.44	7.40	2.89	5.32	2.06	3.23
Acrylic Acid†	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02

Note: No processing aids were used.

* THC = Total hydrocarbons minus methane.

† Hazardous air pollutants (HAPs).

spiking experiment, collection and recovery efficiencies were determined only for the canister sampling method. The results from the two spiking experiments are summarized in Table 5. The analytes measured by the spiking experiments are listed in Column 1. Column 3 shows the calculated concentrations of the spiked compounds in the air stream of the manifold. The concentrations found from duplicate sampling and analyses, and corrected for background levels, are shown in the next two columns. Finally, the average percent recovered is given in the last column.

The results from the first experiment are summarized as follows: all three collection methods showed very good

recoveries of the manifold spiked compounds; the three organic acids were spiked at a nominal air concentration of about 0.6 to 0.8 µg/L; recoveries using the KOH-coated filters ranged from 107 to 122%; formaldehyde (1.63 µ/L) served as the surrogate for the aldehyde–ketone species and the DNPH cartridge method showed a recovery of 130%; deuterated benzene (0.092 µg/L) served as the representative compound for the canister collection method; and the amount recovered was 95%.

During the second experiment, additional recovery data points were obtained for the canister method using an expanded list of compounds. The additional compounds

Table 7. Coefficient for equations predicting EMA emission levels, $Y = MT + C$, where T is extrusion temperature ($^{\circ}\text{F}$) and Y is emission quantity in lbs per million lbs of resin.

EMA (20% Copolymer)	Temperature Range	M Slope	C (y intercept)
VOC (402 method)	350 - 565 $^{\circ}\text{F}$	0.33	-70.7
Particulates	350 - 565 $^{\circ}\text{F}$	0.27	-89.3
Formaldehyde	350 - 565 $^{\circ}\text{F}$	0.0046	-1.15
Acetaldehyde	350 - 565 $^{\circ}\text{F}$	0.0034	-1.17
Formic Acid	350 - 565 $^{\circ}\text{F}$	0.0012	3.98
Acetic Acid	350 - 565 $^{\circ}\text{F}$	0.0054	0.16

Other hydrocarbons and acids were detected, but were below the 0.75 ppm cut-off point.

included deuterated benzene for comparison with the first experiment, as well as benzene, methyl acrylate, deuterated methyl acrylate, and vinyl acetate. The expected spike level of these five species was nominally 0.24 μL . As the results indicate, excellent recoveries were obtained for all compounds. Mass ions from the mass spectrometric detector that were specific for each compound were used in calculating recovery efficiencies because the five species were not well resolved with the analytical column (e.g., the two methyl acrylates were seen as one peak when monitoring the flame ionization detector).

EMISSION FACTOR RESULTS

Ethylene Vinyl Acetate Copolymers

The emission results are presented in Table 6. Overall, VOCs and particulates for all three EVA test resins had much higher emission rates than the oxygenates. VOC emissions ranged from 100 to 130 ppm (wt/wt), while particulates were less than 1 ppm. The higher test temperature produced higher levels of aldehydes, but lower overall VOCs. However, this result is confounded because different EVA resins were used.

As discussed in the experimental section, two different methods were used to measure VOC emissions. One was the Beckman 402 Hydrocarbon Analyzer which continually analyzed the air emission stream throughout the run and provided a direct reading of all VOC substances responding to the flame ionization detector. The other method used an evacuated canister for sample collection and gas chromatography for analysis. With this method, total VOCs were determined by summing the Heavy Hydrocarbon (HHC) and Light Hydrocarbon (LHC) results.

As can be seen in Table 6, the Beckman 402 results are consistently higher than the HHC and LHC results. There are a number of possible explanations for these discrepancies, as the techniques are inherently different, but that discussion is beyond the scope of this paper. However, as a conservative measure, it is recommended that the higher result be used when estimating emission quantities.

One advantage of the canister method is that it can provide emission data on total VOCs as well as individual compounds. Based on visual observation of the

VOC chromatograms, the VOC measurements were due to the additive response of many individual compounds. The majority of individual VOCs were well below 1 ppm (wt/wt). The exceptions were the organic acids, which were in the range of 6 to 12 ppm total. Variations in the amounts of organic acids evolved did not follow either the die-melt temperature or the percent bound vinyl acetate. This may have been simply a reflection of the variability of the method, or the effect of different samples being used at different temperatures. Organic acid emissions were, however, significantly higher than those observed in an earlier study on LDPE resins.²

Vinyl acetate was detected in only one of the test runs, that of the high vinyl acetate copolymer in Run #3. It is thought that this may have been an artifact of the test apparatus in which fewer VOCs may have adhered to the canister wall during sample storage and were not completely released during sample analysis.

Ethylene-Methyl Acrylate Copolymers

The emission factor results for the EMA copolymers are presented in Table 6. Extrusions were performed at 350 and 565 $^{\circ}\text{F}$, corresponding to blown film and extrusion coating temperatures, respectively. Overall, the VOCs for the test resins had higher emission rates than the oxygenates. VOC emissions ranged from 45 to 117 ppm (wt/wt) and the particulates from 4 to 61 ppm (wt/wt). As expected, the higher test temperatures generally produced the higher emission factors. Even at the highest test temperature, the majority of individual VOCs were below 1 ppm (wt/wt) and no single VOC compound exceeded 5 ppm (wt/wt). Those that exceeded 1 ppm were aliphatic hydrocarbons in the C_{10} to C_{16} range.

Oxygenated VOCs were present in the emissions at both temperatures, but generally at values <1 ppm (wt/wt). The exceptions were formic acid, and acetic acid detected at levels of <5 ppm at both extrusion temperatures, and formaldehyde, detected at a level of approximately 1 ppm at 565 $^{\circ}\text{F}$ extrusion temperature. From the structure of the ethylene-methyl acrylate copolymer shown below, it was thought that methanol would be generated during extrusion at the highest temperature.

H H H H

-C-C-C-C-

H H H
C = O

O

CH_3

However, specific evaluation of the GC-MS runs for methanol showed this compound to be absent in runs made at both extrusion temperatures. The oxygenated compounds on the HAPs list are designated as such in Table 6.

Predicting Emissions within Experimental Temperature Range

The data in Table 6 were reduced to the following equation for EMA that predicts the level of emissions at a specific extrusion temperature:

$$Y = (M \times T) + C \quad (1)$$

where Y = emissions in pounds per million pounds of processed resin, and T = melt temperature in °F. M and C constants are shown in Table 7 for each analyte.

Inserting the melt temperature (°F) into the equation will provide an estimate of the number of pounds of emissions per one million pounds of processed polymer. This equation is only valid within the temperature ranges and conditions used in this study and is not recommended for predicting emissions for temperatures outside this range. A similar equation was not derived for EVA because of the limitations of test temperatures.

CONCLUSION

Significance of Emission Factors from SPI Study

This study provides published emission rate data collected during extrusion of EVA and EMA under specific operating conditions.

The significance of this data becomes apparent when placed into context of the 1990 Clean Air Amendment's definition of a "major" source for VOC emissions. Categorization of an emission source as a "major" source subjects it to more stringent permitting requirements. The definition of a "major" source varies with the severity of the ozone nonattainment situation of the area where the source is located. The current VOC emission limits are 10 tons per year for a source in the severe classification, and 50 tons per year for a source in the serious classification. Currently, the only extreme nonattainment area in the United States is the Los Angeles, California area.

The utility of this data can be illustrated in the following example. Based on the emissions data and equations developed in this effort, a processor with equipment and conditions similar to those in this study can extrude up to 156 million pounds of EVA or 171 million pounds of EMA, and using the maximum emissions discovered in this study without exceeding the 10-ton-per-year limit for an extreme ozone nonattainment area. However, before using the data in this paper to estimate emissions, one must consider a number of other parameters, such as increased additive levels, which may impact the type and quantity of emissions as discussed in the Introduction.

These results cannot be used for industrial hygiene purposes.

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