

# ***STATE OF FLORIDA***



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## **EFFECT OF TIRE RUBBER GRINDING METHOD ON ASPHALT-RUBBER BINDER CHARACTERISTICS**

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**Research Report  
FL/DOT/SMO/96-410A**

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## EXECUTIVE SUMMARY

This report presents the results of a study carried out to evaluate the effect of the rubber grinding processes on the properties and characteristics of the resulting asphalt-rubber binder. Several ambient and cryogenic ground tire rubber (GTR) materials were evaluated using measurements of surface areas and bulk densities. The rubber materials were then, respectively, mixed with an AC-30 asphalt, and the resulting blends were tested to determine the corresponding viscosity, settlement during storage, and the potential for binder draindown.

The findings indicate that the asphalt-rubber binders produced with rubber from the different grinding processes have measurable differences in properties and storage characteristics which are critical to the performance of the binder in open-graded mixtures. The wet-ground rubber material had substantially lower bulk densities and larger surface areas than rubber resulting from other grinding methods. GTR materials with greater specific surface areas, and more irregular shaped particles produced asphalt-rubber binders having higher viscosities. Binders with the cryogenic ground rubber had the greatest amount of settlement and the least resistance to draindown.

**Key words:** Ground tire rubber, asphalt-rubber binder, ambient grinding, cryogenic grinding.

## **INTRODUCTION**

There are substantial differences in the existing methods for grinding tire rubber (1). Each method produces a unique rubber particle with specific characteristics such as surface area and texture, size, and shape. It is believed that the surface texture of the cryogenically processed rubber may not be beneficial for its use in asphalt pavements. The underlying hypothesis has been that the irregular texture of ambient ground rubber particles results in higher reaction rate, increased viscosity, and longer storage stability of asphalt-rubber binders. In contrast, glassy and angular cryogenic rubber particles have been suspected to have slower reaction rates, produce lower viscosities, and be more prone to settlement when blended with asphalt cement. Moreover, the development of asphalt rubber and long term performance data only exist for use of ambient ground rubber. Consequently, the present study is carried out to evaluate the effect of the rubber grinding processes on the properties and characteristics of the resulting asphalt-rubber binder. Several ambient and cryogenic ground tire rubber (GTR) materials were evaluated using measurements of surface areas and bulk densities. The rubber materials were then, respectively, mixed with an AC-30 asphalt, and the resulting blends were tested to determine the corresponding viscosity, settlement during storage, and the potential for binder draindown.

## **BACKGROUND**

### **Tire Rubber Grinding Methods**

Presently, there are four known methods for grinding scrap tire rubber (1). Each method results in unique GTR particles with specific characteristics. The grinding process can be either at ambient or cryogenic temperatures. The following is a brief description of each of the four methods:

### ***Crackermill Process***

This process is the most common method for producing GTR at the present time. The tearing of the scrap tire rubber is controlled by the spacing between the drums and their differential speeds. The size of the rubber is reduced by forcing the material through the rotating corrugated steel drums. The crackermill process generates irregularly shaped particles with large surface areas. It is performed at ambient temperatures.

### ***Granulator Process***

This method uses revolving steel plates that pass at close tolerance to cut the scrap tire rubber. The resulting GTR particles have a cubical and uniform shape with a low surface area. This process is also performed at ambient temperatures.

### ***Micro-mill Process***

This process further reduced the GTR particles to a very fine size. The GTR particles are mixed with water to make a slurry-like rubber. The slurry is forced through rotating abrasive disc to reduce the particle size. The processed slurry is then retrieved and dried to the final GTR product.

### ***Cryogenic Process***

In this process, the brittleness of the scrap tire is increased by submerging it in a liquid nitrogen bath. The rubber is then crushed to the desired size. It is believed that the surface texture of the cryogenically processed rubber may not be beneficial for its use in asphalt pavements.

### **Current FDOT Specifications**

Specifications developed and implemented by the Florida Department of Transportation require that ground tire rubber (GTR) used in asphalt pavements be produced such that the final grinding process occurs at ambient temperatures. Such a requirement is based on recommendations

from national experts (2,3) and the findings of local experimentation with asphalt rubber and its application for highway construction (4,5,6). The underlying hypothesis has been that the irregular texture of ambient ground rubber particles results in higher reaction rate, increased viscosity, and longer storage stability of asphalt-rubber binders. In contrast, glassy and angular cryogenic rubber particles have been suspected to have slower reaction rates, produce lower viscosities, and be more prone to settlement when blended with asphalt cement. Moreover, the development of asphalt rubber and long term performance data only exist for use of ambient ground rubber. Consequently, to date, all of the experimentation and development work dealing with the asphalt-rubber in Florida has been based on GTR produced by ambient grinding methods.

### **OBJECTIVES**

Since the implementation of GTR specifications in Florida, several companies involved in cryogenic grinding processes have proposed significantly lower costs in the cryogenic ground tire rubber production. However, further technical information on the effect of cryogenic ground rubber on the properties of asphalt rubber mixtures is needed before making rational decisions about materials selection based on comparative cost-effectiveness. Thus, the present study was initiated to (a) determine if measurable differences exist between the rubber produced by ambient and cryogenic grinding processes and (b) evaluate the effect of the rubber generated by the respective grinding methods on the asphalt-rubber binder properties and characteristics for the Florida applications.

Several ambient and cryogenic ground tire rubber materials were evaluated using measurements of surface areas and bulk densities. The rubber materials were then respectively mixed with an AC-30 asphalt, and the resulting blends were tested to determine viscosity and



settlement during storage. The potential for binder draindown in a typical Florida open-graded friction course mixture was also investigated.

## **LABORATORY TESTING PROGRAM**

### **Materials**

Tire rubber samples were obtained from four GTR processors listed in Table 1. Each processor was asked to supply approximately 11 kg (25 lbs) of GTR generally meeting the FDOT Type B gradation specifications. The suppliers used different grinding equipment and techniques to produce the test samples. GTR from Rouse Rubber, Inc. was produced with a wet-grinding process at ambient temperatures while American Tire Recyclers (ATR), Inc., produced the rubber using a series of shredders and dry grinding mills at ambient temperatures. Cryogenic ground rubber was obtained from Eco<sup>2</sup>, Inc., which processed the fine size rubber using liquid-nitrogen cooled cryogenic hammer mills. Recovery Technologies, Inc., also produced the rubber through a liquid-nitrogen cryogenic grinding process. However, in the latter, the temperature of the final cryogenic mill was allowed to increase above the embrittlement temperature of the rubber. This process was proposed as a combination cryogenic-ambient grinding process that would result in more irregular or ambient-like shaped rubber particles.

### **Sample Preparation**

To have a good base for comparison between the different sources of GTR, it was deemed necessary to prepare all the samples using the same particle size distributions. A target gradation near the mid-range of the Florida Type B specifications was selected. A sufficient quantity of GTR from each source was separated into pass-retained fractions by washing and sieving. The pass-retained fractions were then reblended using the correct proportions to meet the set gradation target.

It should be noted that the sample received from Recovery Tech. had a very limited quantity of material retained on the 425  $\mu\text{m}$  (No.40) sieve. Due to this limitation, some tests on the fractionated GTR samples were not performed on this fraction so that the available material could be used in tests in which particle size distribution was important.

The GTR samples, proportioned to the target gradation, were then respectively blended with an AC-30 asphalt cement from Coastal Fuels terminal located in Jacksonville, Florida. The asphalt was preheated to a homogeneous temperature of 163°C (325 °F) in 1.9 L (half-gallon) cans. Twelve percent GTR (by weight of asphalt) was thoroughly mixed with the asphalt cement using a mechanical blender for 10 minutes. Each asphalt-rubber mix sample was returned to a 163°C (325 °F) oven for one hour with intermittent reblending at 15 minute intervals. The asphalt-rubber samples were then removed from the oven and allowed to cool to room temperature until time for further testing. Prior to testing, the blended asphalt-rubber samples were reheated to 149°C (300°F) and thoroughly remixed with the mechanical blender.

### **Laboratory Evaluation**

For the laboratory experiment conducted in this study, the following tests were performed:

#### ***Sieve Analysis***

The particle size distributions of the as-received GTR samples were characterized by the sieve analyses performed in accordance with the Florida Method 5-559, *Testing of Ground Tire Rubber*.

#### ***Surface Area Measurements***

Surface area measurements on the fractionized sizes of the GTR from each source were performed by Porous Materials Inc., Ithaca, NY., under the direction of Dr. Gupta. The Brunauer,

Emmett, and Teller (BET) multi-point surface analyzer was used to determine the specific surface area (area per mass) of the GTR particles. This method utilizes the low-temperature adsorption of krypton gas onto the surface of the rubber particles at various partial pressures (7,8). Using the BET theory, the specific surface area is determined by measuring the quantity of krypton gas adsorbed onto the surface of a known mass of GTR at various relative pressures.

### ***Bulk Density Determination***

Bulk densities of GTR samples were determined using the funnel stand and cylindrical measure apparatus developed for the NAA fine aggregate angularity test, AASHTO Provisional Standard TP33-93. To determine the bulk density of the GTR samples, the nominal 100 mL cylindrical measure was filled by allowing the sample to pour through the funnel from a fixed height. The sample was gently struck-off even with top of the measure and then weighed. The bulk density was determined as the mass of the sample divided by the volume of the cylindrical measure. The test was performed on two size fractions from each source; one fraction consisting of the material passing the 850  $\mu\text{m}$  (No. 20) sieve and retained on the 425  $\mu\text{m}$  (No. 40) sieve, and the other fraction of passing the 425  $\mu\text{m}$  (No. 40) sieve and retained on the 180  $\mu\text{m}$  (No. 80 sieve). Triplicate tests were performed for each fraction.

### ***Viscosity Measurement***

The viscosity of each asphalt-rubber mix sample was determined in accordance with ASTM D 4402 (9). Viscosities were respectively measured at 135 °C, 149 °C, and 163 °C (275 °F, 300 °F, and 325 °F) using a Brookfield viscometer model DV-II with a thermocel temperature controller.

### ***Settlement Test***

Settlement tests, also known as separation tests, were performed using a procedure modified

from section 9.3 of AASHTO PP5-93, *Practice for the Laboratory Evaluation of Modified Asphalt Systems (10)*. The settlement test consisted of filling aluminum tubes, 25 mm (1 in) in diameter and 900 mm (3ft) long, with the asphalt-rubber samples. The ends of the aluminum tubes were tightly crimped. The filled tubes were then placed upright in an oven at 163 °C (325 °F) for 48 hours. At the end of the heating period, the tubes were removed from the oven and immediately placed upright in a low temperature chamber set at -10 °C (14 °F) for at least 16 hours to allow the asphalt-rubber to solidify. The tubes were then sawed into 300 mm (one-foot) segments and labeled as top, middle, and bottom. The asphalt-rubber binder was recovered from each segment and extracted using a terpene-based solvent to recover the GTR. The percentage of GTR in each segment was calculated and used to compare the relative amount of settlement for each source of GTR.

#### ***Draindown Test***

A draindown test procedure developed for Stone Matrix Asphalt (SMA) mixtures (11) was used to evaluate the potential for binder migration in open-graded friction course samples. For this study, prepared oolitic limestone aggregates and asphalt-rubber binder samples were preheated for mixing to approximately 149 °C (300 °F). The asphalt-rubber binders were added to the aggregate samples to achieve a total binder content of 6.3 percent, then mixed rapidly until all aggregate particles were thoroughly coated. The loose, hot-mix samples were poured into pre-weighed wire baskets and positioned on non-absorptive paper sheets. The samples, basket, and paper were placed in a forced-draft oven at 143 °C (290 °F) for one hour. After one hour, the paper was removed from beneath the basket and weighed. The amount of binder draindown was measured as the asphalt-rubber binder which dripped from the sample and deposited on the paper sheet. Two tests were performed with each GTR source.

## DATA ANALYSIS

### Sieve Analysis

The results of the sieve analyses are shown in Table 2. It can be seen that the respective as-received gradations of the GTR samples from the four suppliers were significantly different. The only material meeting the FDOT Type B Gradation was the GTR from Rouse. In comparison, the samples from ATR and Eco<sup>2</sup> were considerably coarser while the sample from Recovery Tech. was significantly finer than the specification range. The respective particle textures and shapes resulting from the grinding processes are illustrated in Figures 1 and 2. These figures are 25x magnified photographs of two pass-retained fractions (425/180 and 180/150  $\mu\text{m}$ ) of GTR from each source.

### Surface Area Measurements

The results of the specific surface area measurements are summarized in Table 3. It can be observed that the ambient, wet-ground GTR from Rouse has the largest surface area for all size fractions. The ambient, dry-ground GTR from ATR has substantially lower surface areas than the Rouse GTR and is more comparable to the GTR from Recovery Tech.. Holland et al. (8), which reported comparable results on unfractionated GTR samples, give standard deviations typically in the range of 0.004 to 0.006  $\text{m}^2/\text{g}$ , based on five replicate tests. The results also demonstrate that specific surface area increases as particle sizes decrease. As a perspective on the scale of these surface areas, the specific surface area for a sample of 165 $\mu\text{m}$  (midpoint of 180/150  $\mu\text{m}$  fraction) cubes of GTR would be 0.0316  $\text{m}^2/\text{g}$ .

### Bulk Density Measurements

The data on the bulk density in Table 4 show that the ambient wet-ground GTR from Rouse had the lowest density values while the cryogenic Eco<sup>2</sup> rubber had the highest. This trend supports

the intuitive logic that the irregular particles will be deposited with more voids (lower bulk density) than smooth-faced rubber particles. A good repeatability of the test can also be observed. Furthermore, the available data seem to suggest that the size fraction has no effect on the bulk density of the GTR.

### **Viscosity Tests**

The results of the viscosity tests are summarized in Table 5. The data show significant differences in viscosities for the asphalt-rubber binders from different sources. Figure 3 illustrates the GTR source effect on the viscosity of the asphalt-rubber binders. The current specification requires a minimum of 1 Pa-s (10 Poises) viscosity for ARB-12 at 149 °C (300 °F). The binders containing the GTR from Eco<sup>2</sup> and Recovery Technologies failed to meet this requirement.

### **Settlement Tests**

The results of the settlement tests are given in Table 6. It is apparent that under the test conditions, settlement is a natural phenomenon for all sources of GTR. However, these results also clearly show that the cryogenic ground GTR had the greatest amount of settlement. Comparison of the percentages of GTR in the bottom segments shows that the Eco<sup>2</sup> GTR had the highest percentage while Rouse GTR had the lowest. ATR and Recovery Tech. had comparable results.

### **Draindown Test**

The average draindown weight and percent draindown for each sample are summarized in Table 7. These results appear to indicate that each of the GTR sources is effective in mitigating the potential for binder draindown. As reference, the maximum allowable draindown is 0.3 percent of total mix for SMA mixtures. These test results also indicate that the ambient ground GTR materials provide, comparatively, better resistance to binder draindown in the open-graded friction course mixture.

## CONCLUSIONS

The findings of this study indicate that the process used to grind fine rubber materials from waste tires has a significant effect on the shape, texture, and certain physical properties of the rubber particles. Consequently, asphalt-rubber binders produced with rubber from different grinding methods will also have measurable differences in properties and storage characteristics which are critical to the performance of the binder in open-graded mixtures.

On the basis of the analysis of the laboratory data, the following conclusions can be drawn:

1. Surface area measurements and high magnification photographs of GTR samples indicate that the “ambient-grind” specification may not always insure that the rubber particles are irregular shaped. The wet grinding process appears to produce GTR with highly irregular particles having relatively large surface areas. The dry grinding process and the cryogenic grinding methods produce more flat sided particles with lower specific surface areas.
2. Bulk densities of GTR samples, determined using the NAA fine aggregate angularity apparatus, may provide a simple quantitative means of evaluating GTR particle textures. Results show that the very irregular, wet-ground GTR material has substantially lower bulk densities than GTR material from other grinding methods. A maximum bulk density of  $0.40 \text{ g/cm}^3$  appears to distinguish between GTR materials that are smooth and blocky from materials that have highly irregular particle textures. However, the ambient, dry-ground GTR from American Tire Recyclers will not meet this criteria.
3. GTR particle textures have a distinctive effect on the resulting asphalt-rubber viscosities. GTR materials with greater specific surface areas, and more irregular shaped particles will produce asphalt-rubber binders with higher viscosities. The current FDOT specification limit of a

minimum of 1 Pa-s (10 poises) appears to distinguish between asphalt-rubbers (ARB-12) produced with ambient versus cryogenic ground GTR materials.

4. Settlement tests show that the GTR particle textures have a measurable effect on how the GTR will settle in a blend of asphalt-rubber. Under the test condition of no agitation for 48 hours at 163 °C (325°F), the results show that all types of GTR will settle. However, the cryogenic ground GTR had the greatest amount of settlement and the highly irregular-shaped ambient GTR had the least amount of settlement.
5. The draindown test used to evaluate the potential for binder migration in the FC-2 open-graded friction course indicates that the ambient ground GTR materials are more resistant to draindown than cryogenic ground GTR. However, it appears that the criteria of 0.3 percent maximum allowable draindown established for SMA mixtures may not be appropriate for evaluating the GTR modified FC-2 mixtures. This may be due to the high mineral filler content of the SMA binder mastics which significantly increases the weight of the material that may drain during the test. The maximum allowable draindown for FC-2 binders should be reduced. This would require additional research.

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Table 1 Suppliers of Ground Tire Rubber Samples

Processor/Supplier	Address	Report Abbreviation
Rouse Rubber Industries, Inc.	P.O. Box 831, Hwy. 61 South Vicksburg, MS 39181	Rouse
American Tire Recyclers, Inc.	8525 Mallory Road Jacksonville, FL 32220	ATR
Eco <sup>2</sup> , Inc.	P.O. Box 1120 Hawthorne, FL 32640	Eco <sup>2</sup>
Recovery Technologies, Inc.	1225 Franklin Blvd. Cambridge, Ontario N1R 7E5	Recovery Tech

Table 2 Gradations of Supplied GTR Samples

Sieve	Type B Range	Rouse	ATR	Eco <sup>2</sup>	Recovery Tech.	Target
No.20 (850 $\mu$ m)	100	100	100	91	100	100
No.40 (425 $\mu$ m)	85-100	97	76	65	99	75
No.80 (180 $\mu$ m)	10-50	36	10	24	70	25
No.100 (150 $\mu$ m)	5-30	25	4	16	51	5

Table 3 BET Surface Areas, (m<sup>2</sup>/g)

Sample Fraction (Pass/Ret, $\mu$ m)	Rouse	ATR	Eco <sup>2</sup>	Recovery Tech.
850/425	0.0971	0.06451	0.06573	*
425/180	0.14258	0.12363	0.10441	0.11911
180/150	0.25167	0.13814	0.11861	0.15626
150/75	0.30201	0.21710	0.15690	0.19657

\* insufficient material to test this fraction

Table 4 Results of Bulk Density Tests on GTR Samples

Sample Fraction (Pass/Ret)	Bulk Density (g/cm <sup>3</sup> )			
	Rouse	ATR	Eco <sup>2</sup>	Recovery Tech.
425/180 μm	0.3462	0.4130	0.4818	insufficient material to perform this test
	0.3462	0.4030	0.4839	
	0.3452	0.4030	0.4809	
180/150 μm	0.3452	0.4250	0.4659	0.4160
	0.3482	0.4260	0.4649	0.4130
	0.3482	0.4240	0.4639	0.4150

Table 5 Viscosities of Asphalt Rubber Samples Containing 12% GTR

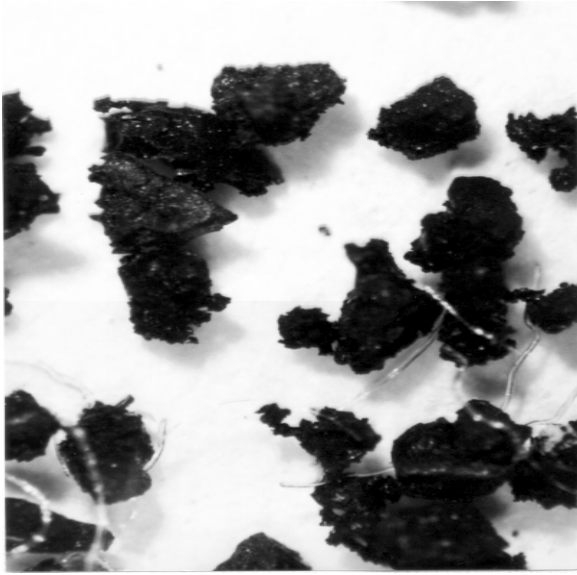
Temperature, °C	Absolute Viscosity, Pa-s			
	Rouse	ATR	Eco <sup>2</sup>	Recovery Tech.
135	2.64	1.98	1.61	1.75
149	1.34	1.10	0.82	0.91
163	0.79	0.72	0.57	0.57

Table 6 Results of Asphalt-Rubber Settlement Tests

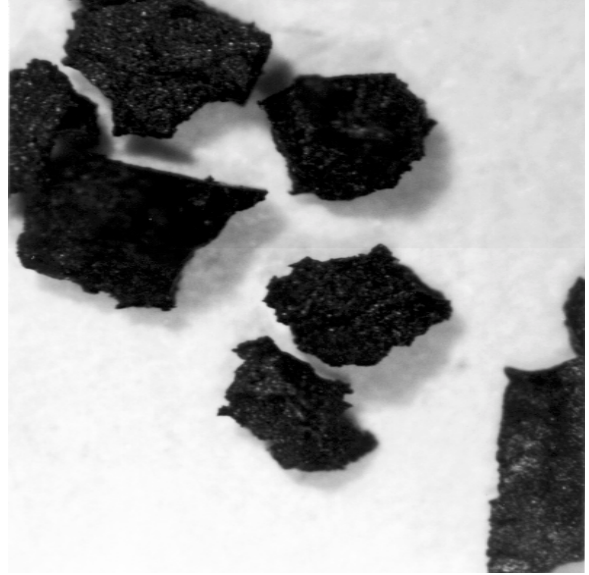
Segment of Settlement Tube	Percent GTR by Weight of Asphalt			
	Rouse	ATR	Eco <sup>2</sup>	Recovery Tech.
Top	0.8	1.4	0.2	0.6
Middle	8.6	11.4	5.8	7.9
Bottom	14.2	17.1	21.6	16.7

Table 7 Results of the Open-Graded Friction Course Draindown Tests

	Percent Draindown by Weight of Total Mix			
	Rouse	ATR	Eco <sup>2</sup>	Recovery Tech.
Sample 1	0.01	0.05	0.02	0.07
Sample 2	0.06	0.04	0.20	0.10
Average	0.04	0.05	0.11	0.09



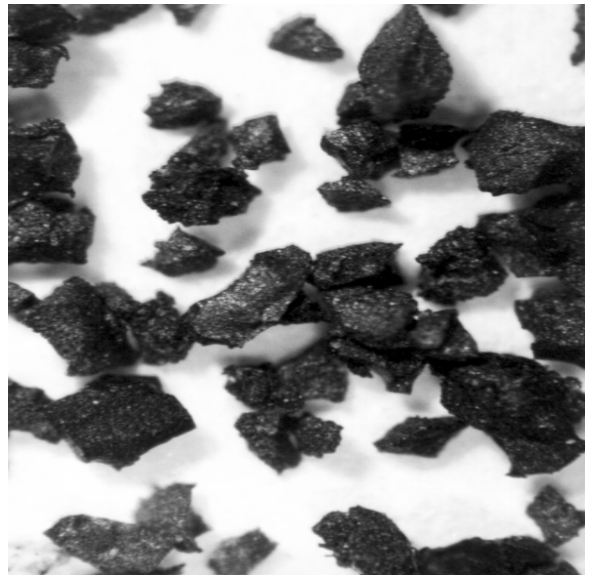
(a) Rouse Rubber, Inc.



(b) American Tire Recyclers, Inc.

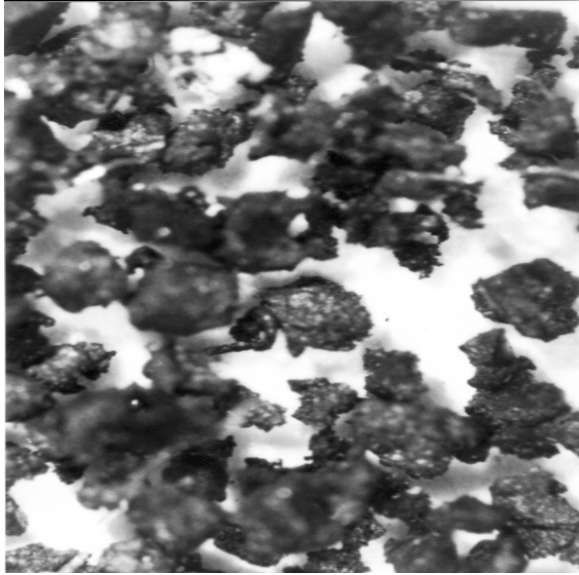


(c) Eco<sup>2</sup>, Inc.

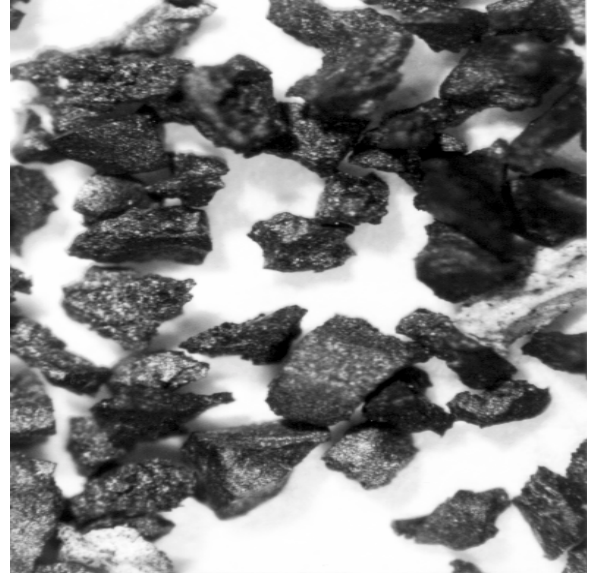


(d) Recovery Technologies, Inc.

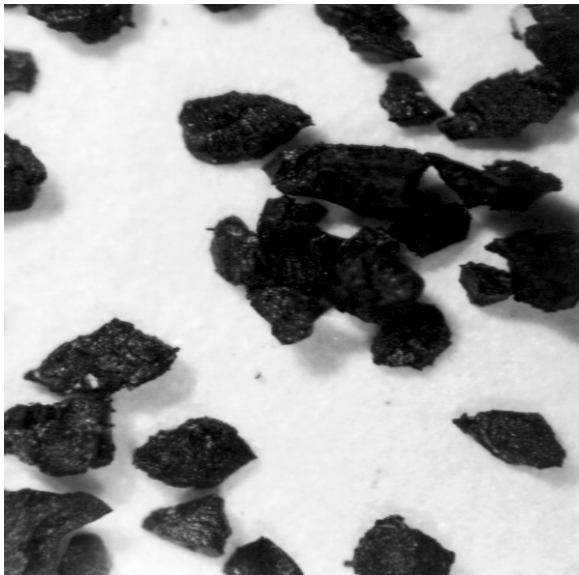
Figure 1 Magnified Illustrations of the Effect of Grinding Methods on the Texture and Shape of GTR Sample Particles Passing 425  $\mu\text{m}$  and Retained on 180  $\mu\text{m}$



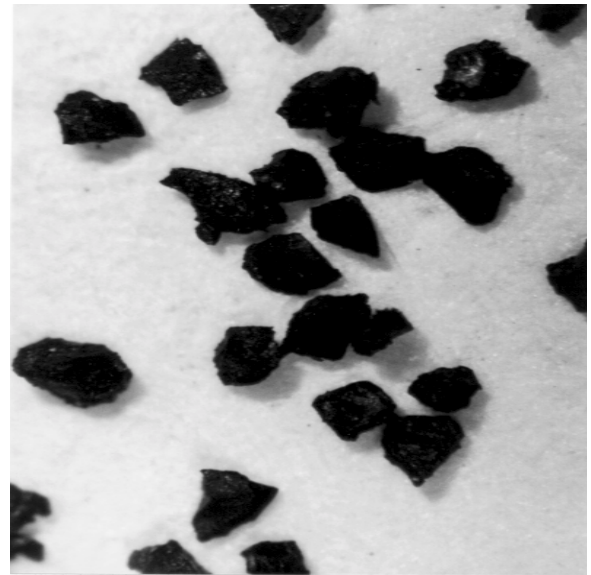
(a) Rouse Rubber, Inc.



(b) American Tire Recyclers, Inc.



(c) Eco<sup>2</sup>, Inc.



(d) Recovery Technologies, Inc.

Figure 2 Magnified Illustrations of the Effect of Grinding Methods on the Texture and Shape of GTR Sample Particles Passing 180  $\mu\text{m}$  and Retained on 150  $\mu\text{m}$

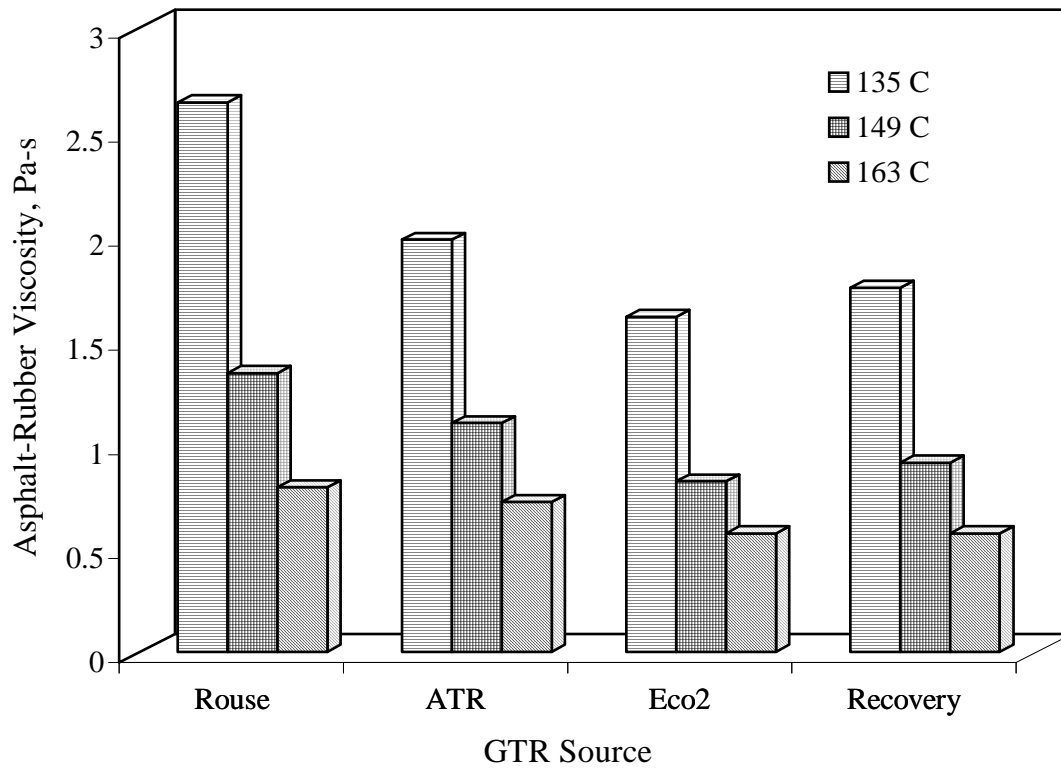


Figure 3 Effect of GTR Source on the Viscosity of Asphalt-Rubber Binders