11. RECYCLING

A. Recycling Assessments and Planning

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Participants:
This project is conducted as part of a Cooperative Research and Development Agreement (CRADA) among ANL, the Vehicle Recycling Partnership (VRP) of the United States Council for Automotive Research (USCAR), and the American Chemistry Council-Plastics Division (ACC-PD, formerly the American Plastics Council (APC)).

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Contractor: ANL
Contract No.: W-31-109-Eng-38

Objectives

- Eliminate any real or perceived recycling barriers that might preclude the use of advanced automotive materials.
- Enable the optimum recycling of all automotive materials, current and future, thereby obviating the need for legislative recycling mandates.
- Assess the critical needs for cost-effective recycling of automotive materials and components.
- Establish research priorities to enable cost-effective recycling of advanced automotive materials and components.
- Communicate a collaborative industry/government approach to issues related to the recycling of automotive materials.
- Coordinate research with other agencies and stakeholders in the United States, Europe, and Asia.

Approach

- Consult with automotive manufacturers and recycling industries, USCAR and its affiliates, national laboratories, universities, and other relevant organizations to assess critical recycling needs/barriers.

- Develop a recycling research plan that will serve as a “working document” to guide the U.S. Department of Energy (DOE) in establishing priority goals, with an initial emphasis on lightweighting body and chassis materials.
- Establish an outreach/communication function to enable cooperation amongst, and leveraging of resources with, all stakeholders and the international community.
- Assist DOE in establishing advanced recycling research and development (R&D) initiatives and provide technical oversight to ensure that priority objectives/goals are accomplished.

Accomplishments During this Reporting Period (10/1/06-9/30/07)

- Conducted by-monthly progress reviews with CRADA team.
- Identified new lightweighting materials being considered for vehicles of the future.
- Continued gap analysis with CRADA team.
- Prepared and presented several papers outlining the industry/government collaboration at national and international conferences.
- The CRADA team received the Society of Plastics Engineers (SPE), Environmental Division, Global Plastics Environmental Conference (GPEC) "Enabling Technologies in Processes & Procedures" 2007 Award. The award is in recognition of the work of the CRADA team in the development of enabling processes and procedures to facilitate the recovery and recycling of automotive plastics from end-of-life vehicles (ELVs).
- Continued liaison with the Institute of Scrap Recycling Industries (ISRI), the Automotive Recycling Association (ARA) and individual shredders.
- The American Chemistry Council-Plastics Division’s Operating Committee visited the Argonne pilot-plant.

Prior Accomplishments

Fiscal Year (FY) 2006

- Conducted quarterly progress reviews with CRADA team.
- Conducted annual project review and gap analysis with CRADA team.
- Developed CRADA Team presentation Brochure (was prepared by Energetics) and One-Pager.
- Launched US ELV CRADA Team Website (http://www.transportation.anl.gov/materials/crada_recycling.html). It includes an overview of the CRADA Team activities, downloadable CRADA Team brochures, a bibliography of recycle literature, presentations, and annual reports of the team.
- Presented papers outlining the industry/government collaboration at international conferences.
- The CRADA team held a media event for America Recycles Day. Press releases and related news stories are accessible through the CRADA team website.
- Continued liaison with the Institute of Scrap Recycling Industries (ISRI) and the Automotive Recycling Association (ARA) and held several meetings with the CRADA partners and representatives of ISRI and ARA.

FY 2005

- Conducted quarterly progress reviews with CRADA team.
- Conducted annual project review and gap analysis with CRADA team.
- Conducted one-day, peer-review progress review. It was attended by experts in the field, in addition to the CRADA partners.
- Conducted one-day Roadmap workshop to update the 2001 Roadmap for Recycling End-of-Life Vehicles (ELVs) of the Future.
- Continued liaison with ISRI and held several meetings with the CRADA partners and representatives of ISRI.

FY 2004

- Conducted quarterly progress reviews with CRADA team.
- Conducted annual project review and gap analysis with CRADA team.
- Held a CRADA announcement event at ANL on December 2, 2004. The event was attended by representatives of the press, industry, and government.
- Established liaison with ISRI and held several meetings with the CRADA partners and representatives of ISRI.

FY 2003

- Developed 5-year project plan.
- Negotiated a CRADA with the VRP, the APC, and ANL as partners. Effort under the CRADA was initiated in August 2003.

Future Direction

- Continue development and management of the R&D plan with the CRADA partners consistent with the recommendations of the updated Roadmap. As appropriate, new recycle R&D needs that are identified will be incorporated into the plan and projects for conducting the requisite R&D will be developed.
- Continue gap analysis with the CRADA partners.
- Conduct scheduled progress reviews.
- Maintain and update the US ELV CRADA team website.
- Continue ongoing efforts toward the milestones and objectives of the CRADA statement-of-work.
- Continue outreach efforts to broaden the basis for cooperation among stakeholders.
- Continue ongoing project efforts to assist DOE in preparation of planning documents, priority recycling R&D needs, proposal reviews, and related tasks.
- Identify research needs for recycling new lightweighting materials.
- Update the ELV Roadmap as necessary.
- Continue to prepare and publish papers.

Summary

The objective of this project is to establish priorities and develop cost-effective recycling technologies and strategies in support of the U.S. Department of Energy (DOE) Vehicle Technologies Program's (VTP's) long-term objectives and goals. The major goals of this research are to: 1) enable the optimum recycling of all automotive materials; 2) ensure that advanced automotive materials that improve the life-cycle energy use of vehicles are not precluded from use as a result of a perception that those materials are not recyclable; and 3) enable market-driven vehicle recycling.

Today, cars that reach the end of their useful service life in the United States are profitably processed for materials and parts recovery by an existing recycling infrastructure. That infrastructure includes automotive dismantlers, automotive remanufacturers and scrap processors (shredders). The dismantlers recover useable parts for repair and reuse. The dismantlers also recover some of the automotive fluids including the refrigerants and the engine oil. The refrigerants can be purified and reused. Facilities for recycling engine oil also exist. The oil can be used as an energy source or it could be refined and used to make new engine oil. Remanufacturers remanufacture a full range of components including starters, alternators, transmissions and engines to replace defective parts. The scrap processors recover ferrous and non-ferrous metals from the remaining auto "hulk."

The recyclability of the plastics and elastomers of the ELVs is limited at present by the lack of 1) commercially-proven technologies to identify and cost-effectively separate materials and components, and 2) profitable post-use markets. The presence of polychlorinated biphenyls (PCBs) on the plastics severely limits their end use. During the next 20 years, both the number and complexity of ELVs are expected to increase, posing significant challenges to the existing recycling infrastructure. The automobile of the future will use significantly greater amounts of lightweight materials (e.g., ultra-light steels, aluminum [Al], magnesium [Mg], titanium [Ti], plastics, and composites) and more sophisticated/complex components, such as fuel cell stacks, hydrogen storage systems, and electronic controls.

Roadmap Recommendations

A workshop to update the original roadmap, which was published in 2001, was held on September 14, 2005, at ANL. Representatives from DOE, key stakeholders, universities and other experts attended the meeting. The workshop evaluated the original Roadmap and its recommendations. The following were identified as some of the factors that can affect the recyclability of future shredder residue:

- Vehicles containing new materials of construction for lightweighting (composites, lightweight steel, Al alloys, Mg, and Ti);
- Catalysts for better environmental control; and
- Vehicles powered by fuel cells, electric batteries, hydrogen, and hybrids.

The key recommendations from the original roadmap, which was developed with input from key stakeholders to guide DOE's recycle research, were:

- Come together as a unified recycling community to cost-share in the development of required new technology.
- Incorporate reuse, remanufacturing, and recycling into the design phase for vehicles whenever possible.
- Recycle as early in the recycling stream as possible, while relying on the market to optimize the value and amount recycled at each step.
- Maintain a flexible recycling process that can adapt to diverse model lines fabricated with different techniques and materials from various suppliers.
- Develop automated ways to recover bulk materials.
- Emphasize R&D on post-shred material identification, sorting, and product recovery.
- Focus R&D efforts on materials not recycled today by sorters (e.g., post-shred plastics, glass, rubber, fluids, and textiles).
- Develop uses for recovered materials (whether in the same or different applications) and testing specifications.
- Encourage investment in the infrastructure needed to achieve the recyclability goal. Build on the existing infrastructure.
- Develop a means to prevent the entry of PCBs and other hazardous materials into the recycling stream and promote acceptable limits in shredder residues.
- Consider the recycling requirements of new technologies entering fleets as early as possible.

The Five-Year R&D Plan

On the basis of the roadmap and continuing discussions with key stakeholders, a five-year research plan was prepared. The plan includes three focus areas, as discussed below.

Area 1. Baseline Technology Assessment and Infrastructure Analysis

The focus of the work under this activity is to develop the tools and document the information necessary to make effective decisions relative to technology needs to facilitate sustainable future vehicle recycling and to make effective decisions regarding the allocation of R&D resources. (See 11.B)

Area 2. Materials Recovery Technology Development and Demonstration

Research to be conducted in this area will initially focus on addressing technology needs for post-shred materials recovery, including mechanical recycling and conversion to fuels and chemicals. (See 11.E) Projects that enhance pre-shred recovery—including disassembly for materials recovery and direct reuse and remanufacturing of components—will also be considered. In the long term, such components as fuel cells, advanced batteries, and onboard hydrogen reformers are more likely to enter the recycle stream through pre-shred recovery for remanufacturing, repair, and materials recovery. Research will be undertaken to determine the technology needs to ensure the recyclability of these advanced automotive components.

Area 3. Recovered Materials Performance and Market Evaluation

Understanding and enhancing recovered materials performance is an essential ingredient to a successful recycling program. This is especially true in automotive systems when the materials and components that are recovered have been in use for an average of from 10–15 years. Area 3 includes projects to quantify the relative performance of recovered materials vis-à-vis new or virgin materials; research on compatibilization of recovered polymers to improve performance properties (see 11.D); development of technologies to upgrade the recovered materials, such as separation of fibers from polymeric substrates; and development of applications for other recovered materials, such as rubber and glass.

CRADA Projects

A CRADA among ANL, the VRP, USCAR, and the APC (now the ACC-PD) has been structured to provide a core team of expertise and the resources to enable the optimum recycling of all automotive materials.

The CRADA team's R&D agenda focuses on the following key objectives:

- Develop and demonstrate sustainable technologies and processes for ELV recycling.
- Demonstrate the feasibility of resource recovery from shredder residue, including materials recovery for reuse in automotive and other applications, chemical conversion of residue to fuels and chemicals, and energy recovery.
- Develop viable strategies for the control and minimization or the elimination of substances of concern. (See 11.C)
- Benchmark recycling technology and provide data to stakeholders.
- Stimulate markets for reprocessed materials to support economic collection, processing, and transportation.
- Transfer technology to commercial practice.

This project provides for the overall management of the CRADA team activities and for communication and advocacy with other organizations. The other major projects that have been initiated under the CRADA include the following:

- Baseline Assessment of Recycling Systems and Technology.
- Post-Shred Materials Recovery Technology Development and Demonstration.
- Development of Technology for Removal of PCBs and Other Substances of Concern from Shredder Residue.
- Compatibilization/Compounding Evaluation of Recovered Polymers.

The objectives and progress on these projects are discussed in their respective sections of this report (see 11.B, 11.E, 11.C and 11.D, respectively). Effort under the CRADA was initiated in the fourth quarter of FY 2003.

Outreach Efforts

While the CRADA team provides a core of expertise, cooperation with other organizations is key to achieving the overall program objectives. In the United States, a market-driven recycling infrastructure is in place. The CRADA team is actively pursuing cooperation with the organizations and companies that are a part of that infrastructure. Cooperation with other stakeholders is also essential.

The CRADA team received the SPE, Environmental Division, GPEC Enabling Technologies in Processes & Procedures" 2007 Award. The award is given in recognition of the work of the CRADA team in the development of enabling processes and procedures to facilitate the recovery and recycling of automotive plastics from ELVs.

Many shredders, polymer recyclers, and the ACC-PD's operating committee visited Argonne's automotive recycling pilot-plant and discussed plastics-recycling activities.

A website was launched in March of 2006 to provide for better communication and networking with stakeholders and other research teams: (http://www.transportation.anl.gov/materials/crada _recycling.html). The website provides an update of the CRADA progress and provides access to relevant information and publications including a bibliography of mechanical, thermo-chemical conversion, and energy-recovery technologies for recycling automotive materials.

The CRADA team held a media event at Argonne for "America Recycles Day." It was attended by a number of media organizations. Articles featuring the work done by the CRADA team were written by the media members who attended. CRADA team members were interviewed by several radio stations after the media event.

Several presentations and publications were made to further communicate with interested parties, including a paper entitled, "Market Driven Technology Development for Sustainable End-of-Life Vehicle Recycling: A Perspective from the United States." This was presented by Edward Daniels at the 6th International Automobile Recycling Congress, Amsterdam, Netherlands, March 15-17, 2006. A joint DOE, USCAR, and APC paper on "Market Driven Recycling in North America" was presented as the keynote paper at the 2004 International Car Recycle Congress in Washington, D.C.

Several meetings with representatives of ISRI and ARA and with shredder operators were held to brief them on the CRADA objectives and projects and to elicit their participation.

To further communicate the U.S. approach to ELV recycling, a one-page CRADA summary and a CRADA brochure have been prepared and it is available at the CRADA website.

As previously mentioned, a review of the projects and ongoing efforts of the CRADA team was held on September 13, 2005, and a workshop was also held on September 14, 2005 to review and update the ELV Roadmap.

Publications

1. *The R&D of the FreedomCAR Materials Program*, Carpenter, J. A., Jr., E. J. Daniels, P. S. Sklad, C. D. Warren and M. T. Smith, Proc. Of the International Auto Body Congress, Novi, MI, September 19, 2006.
2. *Market Driven Technology Development for Sustainable End-of-Life Vehicle Recycling: A Perspective from the United States*, Daniels, E. J., Jody, B. J., Pomykala, J. A. Jr., and Spangenberger, J. S., presented at the 6th International Automobile Recycling Congress, Amsterdam, Netherlands, March 15-17, 2006.
3. *Industry and Government Collaboration to Facilitate Sustainable End-of-Life Vehicle Recycling*, Daniels, E. J., 2005 ASME International Mechanical Engineering Congress & Exposition, BRTD-4: Sustainability Applications in Product Design and Manufacture, Orlando, Florida, November 5-11, 2005.
4. *Market Driven Automotive Recycling in North America*, Duranceau, C., presented at the Institute of Scrap Recycling Industries Shredder Meeting, Dallas, TX (Oct. 30, 2004).
5. *Sustainable End-of-Life Vehicle Recycling: R&D Collaboration between Industry and the U.S. DOE*, Daniels, E. J., Carpenter, J. A. Jr., Duranceau, C., Fisher, M., Wheeler, C., and Winslow, G., JOM, The Mineral, Metals & Materials Society, vol. 56, no 8, pp 28-32 (Aug. 2004).
6. *Market Driven Automotive Recycling in North America*, Duranceau, C., USCAR, Carpenter, J., U.S. DOE, Fisher, M., American Plastics Council, keynote at the 2004 International Car Recycling Workshop, May 19, 2004, Washington D.C.
7. *Automotive Materials Recycling: A Status Report of U.S. DOE and Industry Collaboration*, Daniels, E. J., Ecomaterials and Ecoprocesses, Proc. of the International Symposium on Ecomaterials and Ecoprocesses, August 24-27, 2003, Vancouver, BC, Canada, pp 389-402.
8. *Effects of Transportation on the Ecosystem*, Carpenter, J.A., Jr., Ecomaterials and Ecoprocesses, Proc. of the International Symposium on Ecomaterials and Ecoprocesses, August 24-27, 2003, Vancouver, BC, Canada, pp 13-22.
9. *Automotive Technology: Looking Forward*, Sullivan, R., D. Hamilton and J.A. Carpenter, Jr., Ecomaterials and Ecoprocesses, Proc. of the International Symposium on Ecomaterials and Ecoprocesses, August 24-27, 2003, Vancouver, BC, Canada, pp 49-67.

10. *A Roadmap for Recycling End-of-Life Vehicles of the Future*, prepared by Energetics for the U.S. Department of Energy, Office of Advanced Automotive Technologies (May 2001).

B. Baseline Assessment of Recycling Systems and Technology

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Participants
This project is conducted as part of a Cooperative Research and Development Agreement (CRADA) among ANL, the Vehicle Recycling Partnership (VRP) of the United States Council for Automotive Research (USCAR), and the American Chemistry Council-Plastics Division (ACC-PD, formerly the American Plastics Council (APC)).

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Contractor: ANL
Contract No.: W-31-109-Eng-38

Objective

- Establish the baseline or state-of-the-art for automotive materials recovery/recycling technology.

Approach

- Review the state-of-the-art of worldwide automotive-materials recovery/recycling technologies.
- Develop technology profiles of emerging automotive materials recycling technologies.
- Review international, federal, and state regulatory information regarding vehicle recyclability, substances of concern, and recycle laws and mandates.
- Conduct life-cycle studies to quantify the environmental burdens and benefits associated with various end-of-life recycling technologies.
- Conduct reference-case end-of-life recyclability studies.

Accomplishments during this Reporting Period (10/1/06-9/30/07)

- Updated the database of recycle technologies.
- Initiated the life-cycle study of current shredding operations.
- Prepared and published the document reviewing technologies and the state-of-the-art for recycling shredder residue.

Prior Accomplishments

FY 2006

- Completed compilation of recycle bibliography, structured as a pull-down PDF file, posted on US ELV CRADA Team website http://www.es.anl.gov/Energy_Systems/CRADA_Team_Link/Index.html.
- Completed Changing World Technologies (CWT) life-cycle case study.
- Completed life-cycle study of ANL process technology.
- Completed second draft of state-of-the-art assessment in recycling of vehicles and automotive materials.

FY 2005

- Conducted a literature search that identified mechanical, thermo-chemical conversion, and energy-recovery technologies and completed first draft of state-of-the-art assessment.
- Completed Salyp life-cycle case study, initiated CWT life-cycle case study.

FY 2004

- Compiled and structured recycle bibliography.
- Characterized North American recycle infrastructure.
- Conducted a review of U.S. regulatory issues.
- Initiated life-cycle studies of end-of-life recycle technologies (Salyp case study) .
- Completed reference recyclability calculations for reference cases and three lightweight alternatives: lightweight steel, composite materials, and aluminum (Al).

Future Direction

The focus of this task in FY 2008 will be on:

- Updating the database of recycle technologies.
- Update the document reviewing technologies for recycling shredder residue.
- Complete the life-cycle study of current shredding operations.

Summary

The objectives of this project are to benchmark the automotive materials recycling industry and to compile information in an accessible format regarding the status of existing and emerging recycling technology and research.

The focus of the work under this activity is (1) to develop the tools and document the information necessary to make effective decisions relative to technology needs to facilitate sustainable future vehicle recycling and (2) to make effective decisions regarding allocation of R&D resources.

The state-of-the-art of worldwide automotive-materials recovery/recycling technologies and associated resource-recovery infrastructures has been reviewed to identify technology gaps and needs and to identify differences in automotive recycling strategies among North America, Europe, and Asia. Technologies that are included in this review include, but are not limited to, post-shred materials-recovery technologies, pre-shred materials-recovery technologies, materials-identification technologies, automated-dismantling technologies, technologies for the recycling of specific components of vehicles (such as bumpers), and thermochemical-conversion technologies.

Life-cycle analyses of alternative recycle technologies have also been conducted to identify differences between technologies, such as mechanical recycling vis-à-vis thermochemical recycling, relative to energy and environmental benefits.

Regulations at the international, federal, and state levels are examined to identify the impact that proposed and existing regulations may have regarding recycling of automotive materials.

Reference-case recyclability calculations are made to quantify the expected recyclability of alternative vehicle designs.

Infrastructure

The North American recycling infrastructure has been characterized and a representative figure was shown in previous annual reports.

Technology Profiles

The recent literature has been reviewed, and summaries and profiles of available and emerging recycle technologies have been compiled into a draft working document that will be updated annually as new information becomes available.

A bibliography of abstracts of papers that discuss automotive recycling issues has been compiled (Table 1). The bibliography is organized in fifteen sections.

The bibliography was compiled from an extensive literature search, which included reviewing the following sources:

1. Society of Automotive Engineers (International) World Congresses from 1997 to 2007
2. Environmental Sustainability Conference and Exhibition, 2001
3. SPE:
   - ARC Conference from 1998 to 2000
   - GPEC Conference from 2002-2007
4. Other conference proceedings:
   - International Automobile Recycling Congress from 2001 to 2006
   - TMS Fourth International Symposium of Recycling of Metals and Engineered Materials, 2000.
   - Ecomaterials and Ecoprocesses, The Conference of Metallurgists, COM 2003

The bibliography has been posted on the US ELV CRADA Team website: http://www.es.anl.gov/Energy_Systems/CRADA_Team_Link/Index.html

More references will be added to the bibliography as they become available.

Table 1. Citations included in the recycling bibliography.

Bibliography Section	Number of Citations
Recycling infrastructure	20
Design for recycling	5
Legal and regulatory issues	23
Life-cycle analysis	12
Research programs	11
Substances of concern	5
Disassembly technologies and case studies	12
Reuse of automotive parts and subassemblies	1
Remanufacturing	0
Mechanical-separation technology	20
Thermochemical-conversion technology	14
Energy-recovery technology	11
Advanced materials recycle technology	9
Other technology	40
Case studies of materials recycled for auto applications	23
Total citations	206

Recycling Technologies: State-of-the-Art

The final document has been published and has been posted on the US ELV CRADA Team website: http://www.es.anl.gov/Energy_Systems/CRADA_Team_Link/Index.html.

Because post-shred residue contains residue from shredded white goods and other obsolete items in addition to vehicles, these were also discussed in the document.

Regulatory Situation

The European Union has issued End-of-Life Vehicle (ELV) Recycle Directives. The enforcement of these directives is, however, the responsibility of each member state. Although the United States has not developed a federal policy or mandate, regulations at the federal and state level can impact the technology needs for recycling automotive materials. For example, U.S. Environmental Protection Agency (EPA) regulations regarding polychlorinated biphenyl (PCB) limits the concentration of PCBs on recycled materials to below the detectable limit (i.e., 2 ppm). State regulations regarding other substances of concern (SOCs), such as mercury and polybrominated diphenyl ethers (PBDEs), can also impede materials recycling.

Life-Cycle Studies

The objective is to use life-cycle analysis to assess the environmental impacts of various mechanical-separation technologies and alternative end-of-life recycling technologies. This information will then be used to create a flexible, computerized, life-cycle inventory model, which is process-specific and yet can be modified to include additional recycling technologies and various material inputs. Life-cycle involves assessing all of the upstream burdens associated with the production of the materials and energies used in the process, including the transport of all materials to the facility.

PE Europe GmbH, a company that is experienced in conducting life-cycle assessments and in model development using its own GaBi (Ganzheitliche Bilanzerung) software, was contracted to perform these analyses. Three analyses have been completed for: 1) Salyp NV's mechanical-separation process; 2) CWT's thermal-conversion process; and 3) ANL mechanical and froth-flotation process. A fourth, life-cycle study of current shredding operations has been initiated. Data for the study are still being collected with the assistance of ISRI.

PE Europe has developed a flexible end-of-life model which was used to compare the two different approaches to recycling shredder-residue. The model allows the user to run simulations on shredder-residue separation within different boundary conditions. The following boundary conditions can be modified: 1) shredder-residue composition, 2) location of the facility, 3) type and distance of transportation, 4) market values for the separated fractions, 5) new potential applications for separated fractions, and 6) utilization ratio of the facility.

Salyp's separation process combined equipment developed by ANL and several others to create a facility that separates shredder residue into discrete fractions of metals, foam, mixed plastics, and fiber-rich and fines streams. On the other hand, the CWT process converts organic materials into hydrocarbon fuels and other potential products.

Data were collected for each of the three processes, including all energy, water, and material inputs, plus data on emissions to air and water, wastes, and products produced. The three sets of data were entered into the GaBi software to create a flexible model of the process.

In the case of the Salyp separation process, three different scenarios for handling the various materials recovered from shredder residue were determined. These scenarios included using specific material fractions as fuel for cement kilns (energy recovery), as well as using mixed plastics to replace such products as wood pallets and polypropylene (PP) pellets (material substitution). The various scenarios were assessed by using a variety of impact categories, including primary energy demand and CO_2 emissions. In the case of primary energy demand, all scenarios showed a net credit in total energy use. For the three scenarios studied, substituting recovered polypropylene/ polyethylene (PP/PE) in a new PP application had the greatest benefit. However, if the mixed plastic stream was used to replace wood (e.g., decking material, park benches, wood pallets, etc.), the benefits to primary energy demand were less than if the recovered materials were simply used for energy recovery. In terms of CO_2 emissions, the PP application again showed the greatest benefit. Substituting PP for wood applications was next with a lower benefit, while the energy-recovery scenario showed an increase in CO_2 emissions.

In the case of the CWT process, two basic scenarios were assessed. They involved using the light hydrocarbon oil generated by the process for fuel oil used in power plants to generate electricity and substituting light hydrocarbon oil for diesel oil (both with and without an added hot-oil processing step). While the oil product generated is more refined than an actual crude oil, it would require additional steps before it could be considered a true diesel oil. Therefore, reality is probably located somewhere between scenarios 1 and 2. In this study, the impact on primary energy demand resulted in a benefit in all cases. The benefits in the diesel substitution case were slightly greater than in the fuel-oil case. In the case of CO_2 emissions, all scenarios again showed an overall benefit. However, the diesel substitution case had a greater benefit than the fuel-oil substitution case.

Life-cycle analysis of the ANL process considered both the mechanical separation of the shredder residue to produce a polymer concentrate and recover residual metals, followed by froth-flotation to separate plastics from the polymer concentrate for recycling as plastics (material substitution). The analysis concluded both the mechanical and the froth-flotation processes resulted in environmental benefits, Figure 1. The environmental benefits of the ANL process were also compared with those of Salyp (Table 2) and CWT processes (Table 3). The environmental benefits are higher for the ANL process compared to the Salyp process except for the acidification potential and higher for the ANL process compared to the CWT process except for the impact category EP and NO_x emissions. Energy-wise the ANL process was the most advantageous. Interestingly, the best results can be obtained by combining both (ANL and CWT) processes, where organic fractions separated by ANL which do not meet the requirements for material substitution (such as mixed plastics and rubber by-products) are processed by CWT for fuel production.

Recyclability Studies

Recyclability studies were conducted to examine the effect of using automotive lightweighting material on recyclability. A Toyota Prius hybrid was selected as a reference case. This vehicle is a second-generation hybrid with a gas/electric powertrain. Evaluating the recyclability of this vehicle and its new technology will be a step in identifying changes that will impact end-of-life recycling of vehicles of the future.

In collaboration with Johnson Controls, Inc. (JCI), the VRP dismantled the vehicle according to VRP procedures to single-material components and entered data for each part into a database. A material list that identified the breakdown of materials into separate classifications (such as ferrous and nonferrous metals, as well as composite materials and plastics) was prepared.

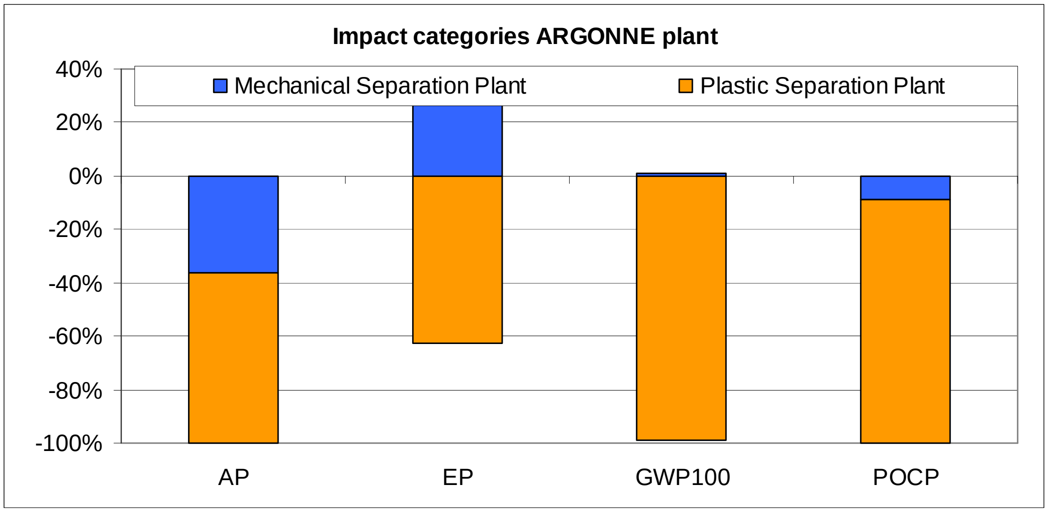

Figure 1. Impact categories of the ANL plant. (AP is acidification potential, EP is eutrophication (depletion of oxygen in water) potential, GWP is global-warning potential and POCP is photochemical ozone creation potential). Y axis indicates increase (+) or decrease (-) in the impact of the different categories.

Table 2. Comparison of the ANL and Salyp processes-Relative Environmental Impact. (A negative value indicates a reduction in the pollution category [an environmental benefit] while a positive value indicates an increase in the pollution category).

	ANL process (mechanical and Froth Flotation)	**Salyp process**
AP [lb SO_2-Equivalent.]	-0.0060	-0.0165
EP [lb Phosphate-Equivalent.]	-0.00011	0.00148
GWP100 [lb CO_2-Equivalent.]	-1.354	0.861
POCP [lb Ethene-Equivalent.]	-0.0026	0.0126

Table 3. Comparison of the ANL Froth Flotation and CWT processes-Relative Environmental Impact. Both processes require mechanical separation of the inorganic fraction. (A negative value indicates a reduction in the pollution category (an environmental benefit) while a positive value indicates an increase in the pollution category).

	ANL process (Froth Flotation*)	**CWT process**
AP [lb SO_2-Equiv.]	-0.01103	-0.00662
EP [lb Phosphate-Equiv.]	-0.00055	-0.00079
GWP100 [lb CO_2-Equiv.]	-4.167	-0.309
POCP [lb Ethene-Equiv.]	-0.0088	-0.0044

*Comparison is done here only with the froth- flotation process because both ANL's froth flotation process and CWT process require mechanical separation of the inorganic materials.

The materials breakdown is summarized in Table 4. By comparison, the materials composition of a production Ford Taurus is summarized in Table 5. Three different recyclability calculations were made (Table 6). The Federal Trade Commission (FTC) recyclability number is the percentage by weight of the material that is currently being recycled, and it includes metals, fluids less fuel, and batteries. The European guidelines include FTC materials plus fuel at 90% of a full tank, plastics that could be recycled, and up to 10% by weight energy recovery.

Table 4. 2004 Toyota Prius materials breakdown.

Materials	Mass (lb)	Percent
Ferrous metals	1713	60.6
Nonferrous metals	507	17.9
Plastics	341	12.1
Elastomers	87	3.1
Inorganic material	77	2.7
Other	62	2.2
Organic materials	42	1.5
Vehicle mass (less fluids)	2829	100.0

Table 5. 2004 Ford Taurus materials breakdown.

Materials	Mass (lb)	Percent
Ferrous metals	2223	70.4
Plastics	340	10.8
Nonferrous metals	312	9.9
Elastomers	152	4.8
Inorganic material	90	2.9
Other	38	1.2
Organic materials	4	0.1
Vehicle mass (less fluids)	3159	100.0

Note that Europe requires 95% recyclability for new vehicles. The feasibility-to-recycle number includes the FTC materials plus plastics that can be recycled. Changes to the current infrastructure would be required to increase recycling beyond the current FTC percentage.

To establish an indication of the impact of lightweight materials on the reference-case recyclability calculations, the 2004 Toyota Prius is compared with a proposed Al-intensive lightweight vehicle and a proposed composite lightweight vehicle, both of which are also based on the 2004 Prius. The production 2004 Toyota Prius hybrid vehicle body was steel with an Al hood and decklid. The suspension was of steel, except for an Al steering knuckle on the front suspension. This vehicle was used as the base for this study.

Table 6. Reference-case recyclability: 2004 Toyota Prius.

Calculation Method	Recyclability (%)
Federal Trade Commission	80.86
European	97.61
Feasibility of recycling	85.58
Ref. 2000 Ford Taurus	80.50

The Al alternative is for a 2004 Toyota Prius with an Al body and a magnesium (Mg) engine cradle and a rear axle substituted for the production parts. In addition, seat frames, body brackets, and the instrument panel cross-car beam have been changed from steel to Al. As a result, the weight has been reduced by approximately 630 lb or 21%. Because the weight reduction is entirely in the currently- recycled portion of the vehicle, the recyclability is adversely affected and is reduced from 80.86% to 76.10%. No changes were made to the currently non-recycled portion of the vehicle. Al replaced steel at 50% by weight of the original steel.

The composite alternative is for a 2004 Toyota Prius that consists of: 1) a carbon-fiber body with 40% carbon fiber and 60% thermoset polyurethane/urea resin by volume, 49.72% carbon, and 50.28% thermoset polyurethane/urea resin by weight, and 2) a Mg engine cradle and rear axle substituted for the production parts. In addition, seat frames, body brackets, and the instrument-panel cross-car beam have been changed from steel to composite. As a result, the weight has been reduced by approximately 711 lb, or 24%. Because the weight reduction is entirely in the currently-recycled portion of the vehicle, the recyclability is adversely affected and is reduced from 80.86% to 57.20% if none of the composite is recycled or 74% if all of the composite material is recycled. No changes were made to the currently non-recycled portion of the vehicle. The composite material replaced steel at 40 wt% of the original steel.

There are reductions in all three recyclability calculations for lightweighted vehicles, even though the rest of the vehicle is not changed (Table 7). Where the Al and composite material is being recycled, the same amount of material would be disposed of in landfills in each of the three scenarios. The only difference is that the recycled portion of the lightweighted vehicles would be lighter. Although the recyclability would be less, there would be no difference in the amount of material disposed of in landfills, and the lighter vehicles would use less fuel during their life. As can be seen, lightweighting presents challenges in the European market. Note that these calculations do not take into account the downsizing of related components that would accompany any lightweight vehicle, such as powertrains, brakes, and tires. Because the downsized components are high in metallic content, downsizing will further reduce recyclability and make it difficult to meet the European 95% requirement.

In conjunction with this study, additional evaluations are planned by using these data as a starting point for assessing the recyclability of cars of the future. The impact of vehicle lightweighting and material selection on recyclability will be evaluated. In addition, the impact of powertrain changes in future vehicles (including hybrid and fuel-cell alternatives) on recyclability will be determined in comparison to powertrains in current vehicles. An assessment of various alternatives on recycling and the effect on the current recycling infrastructure will be produced. No downsizing of other components was included in this study. Future studies will reflect the downsizing of powertrains, brakes, tires, and other components in recyclability calculations. Items requiring further study resulting from these assessments will support future projects to determine the feasibility of various alternative vehicle configurations and choices of materials.

These results demonstrate the need for technology to recycle new automotive material if recycling mandates are to be met and to ensure that lightweighting materials are not excluded because of the inability to recycle them.

A seminar to address recyclability and recycled content in view of changing automotive materials has been planned.

Table 7. 2004 Toyota Prius recyclability, reference case vs. Al and composite body materials.

Calculation Method	As Produced (%)	Al Body (%)	Composite Body (%)
FTC	80.9	76.1	74.0[a]
European	97.6	96.0	94.5[a]
Recycling feasibility	88.3	85.6	83.9[a]

[a] If the composite material were not recycled, then the numbers would be FTC, 57.2%; European, 78.2%; and feasibility of recycling, 67.1%. Recycling of the composite material would require significant changes in the current recycling infrastructure. In addition, a market for the recycled carbon fibers would need to be developed. Current technology for recycling carbon fibers results in a 20% loss in fiber properties and would limit their reuse to short fiber applications.

Publications

1. *A Life Cycle Look at Making Oil From End-of-Life Vehicles.*, Wheeler, C.S., Simon, N.L., Binder, M., Winslow, G. R., Duranceau, C.M., SAE 2006 World Congress, Detroit, Michigan, 2006. SAE-2006-01-0374.
2. *Modular Life Cycle Model — Basis for Analyzing the Environmental Performance of Different Vehicle End-of-Life Options*, Binder, M.; Simon, N. L.; Duranceau, C. M.; Wheeler, C. S.; Winslow, G. R., Proc. of the 5th International Automobile Recycling Congress, Amsterdam (Mar. 9-11, 2005).
3. Modular Life Cycle Model of Vehicle End-of-Life Phase — Basis for Analysis of Environmental Performance, Wheeler, C. S.; Simon, N. L.; Duranceau, C. M.; Winslow, G. R.; Binder, M., SAE Paper 2005-01-0847.
4. *United States National Life Cycle Inventory Database Project, A Status Report*, Sullivan, J. L.; Wheeler, C. S.; and Simon, N. L., SAE Paper 2005-01-0852.

C. Development of Technology for Removal of PCBs and Other Substances of Concern (SOCs) from Shredder Residue

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Contractor: ANL
Contract No.: W-31-109-Eng-38

Objective

- Develop viable strategies and technology for the control and minimization or elimination of polychlorinated biphenyls (PCBs) and other substances of concern (SOCs) from recycled automotive materials.

Approach

- Identify efficient and environmentally-acceptable process solutions for removal of contaminants, including PCBs, from materials recovered from shredder residue.
- Define variances in analytical procedures/test results for PCB analysis.

Accomplishments during this Reporting Period (10/1/06 - 9/30/07)

- Additional bench-scale testing of ANL's two-stage cleaning process has been conducted and has consistently produced plastics from the polypropylene/polyethylene (PP/PE) product with <2 parts per million (ppm) PCBs.

- Tested new methods to clean the plastics and remove the PCBs including:

  -- Conducted preliminary testing of a proprietary process developed by an independent organization

  -- A catalytic process tested at ANL

  -- Cleaning using a bio-degradable solvent

- Developed a design for an apparatus to test ANL's two-stage process at a larger scale (15-25 lb/hr). Quotes for system components have been obtained.
- Hosted and participated in forums and seminars on SOCs including perfluorooctanoic acid (PFOA)/ perfluorooctanesulfonates (PFOS), and brominated fire retardants, emerging lead- and mercury-free technologies.

Prior Accomplishments

<u>Fiscal year (FY) 2006</u>

- ANL developed a two-stage cleaning process which in bench-scale tests has consistently produced plastics from the PP/PE product with <2 ppm PCBs.
- Completed tests in commercial solvent washing equipment using proprietary solvent-based solutions and CO_2
- Cooperated with the Bromine Science and Environmental Forum (BSEF) in preparing the BSEF brochure entitled "Deca-BDE Flame Retardant"

<u>FY 2005</u>

- Completed the study to explain discrepancies in PCBs analytical results
- Initiated testing of commercial solvent washing processes for cleaning plastics
- Conducted a study to understand the interactions between PCBs and plastics
- Identified and initiated testing of alternative methods for PCB removal

<u>FY 2004</u>

- Completed the solvent/detergent screening study for removing PCBs and heavy metals from plastics
- Completed aqueous cleaning tests in commercially-available equipment
- Initiated a study to explain discrepancies in PCBs analytical results

Future Direction

The FY 2008 plan includes:

- Test ANL's two-stage process at a larger scale (15-25 pound test) for the cleaning of the polyolefin concentrate
- Develop a conceptual design and perform cost analysis of the ANL process
- If successful, incorporate the process into the overall process design for recovering materials from shredder residue
- Conduct testing at ECO2 using their proprietary process
- Test a proprietary process developed by an independent organization
- Test vacuum de-volatilization in a screw to remove PCBs

ANL's two-stage process has successfully produced recovered plastics, at least PP/PE materials, with less than 2 ppm PCBs in multiple tests. However, this has been shown only in small-scale experiments. In FY 2008, larger-scale experiments will be conducted to investigate the scalability of the process including required residence time and operating temperatures.

Summary

The objective of this project is to develop techniques and/or technology to identify and/or cost-effectively remove PCBs and other SOCs from recycled automotive materials.

SOCs can impact the recyclability of automotive materials in a number of ways. Certainly, their presence in either recycled materials and/or materials source streams impacts the overall costs of recovering recyclable materials. In some cases, their presence at ppm, such as in the case of PCBs, can prevent the reuse of the recovered materials.

The strategy that is required for control of the SOCs may vary globally. For example, requirements are different in Europe, North America, and Asia for various SOCs. Strategies for controlling SOCs can also depend on the technology used for recycling the automotive material and the materials end use.

The presence of SOCs in vehicles and/or in other durable goods that are presently recycled with end-of-life vehicles (ELVs) is likely to continue to impact the materials recycle stream for the foreseeable future. Consequently, the control of certain SOCs will require technology that will effectively remove the SOCs from recovered materials consistent with current regulatory requirements and consistent with the market requirement for the recovered material.

Other materials that have been in automobiles could also become SOCs in the near future. Examples include some of the brominated biphenyls.

The focus of the work in this project is on the development of technology for the removal of PCBs from potentially recyclable materials recovered from shredder residue. PCBs, at ppm levels, are routinely found in shredder residue. The source of the PCBs is not completely understood, but historically it has been associated with liquid PCB-containing capacitors, ballasts and transformers that inadvertently escape the inspection and control process at the shredders. Unless PCBs are removed from the recovered materials, with the exception of metals, introducing the recovered materials into commerce will not be possible.

Development and Testing of a Two-Stage Process at ANL

The work done so far on washing of polymers recovered from shredder residue suggests that different washing methods and solutions appear to reduce the PCBs concentration down to about 5-10 ppm in a reasonably short time. Further reduction in the concentration of PCBs requires more extensive and prolonged washing in fresh solution. The prolonged washing, particularly in organic solvents, is further complicated by the absorption of the solvent by the plastics which alters the properties and the value of the plastics.

This behavior of PCBs suggests that the PCBs on the plastics are by two different mechanisms. First, some are in the oils and dirt that are on the plastics. Second, some of the PCBs are adsorbed on the plastics and they do not desorb easily during washing. We tested this hypothesis in the lab. We conducted washing tests using several washing solutions and solvents including a non-flammable solvent to wash the plastics under conditions that minimized the absorption of the solvent by the plastics. This reduced the concentrations of PCBs from about 30 ppm to about 5-10 ppm under a range of operating conditions. The washed plastics were then processed in an environment that induces desorption (high temperature and/or reduced pressure). The PCBs concentration was consistently reduced from 5-10 ppm to below 2 ppm. Tests were also conducted where unwashed samples were exposed to the same environment that induces desorption. The PCBs concentration could not be reduced below 2 ppm under the same operating conditions.

A two-stage process, based on this concept, has been developed and tested at the bench scale at ANL It has repeatedly reduced the PCBs concentration in PP/PE samples to less than 2 ppm. For example, at conditions that do not cause the plastics to oxidize, we were able to reduce the PCBs concentration on the recovered polyolefins from about 32 ppm to as low as 0.088 ppm. We developed a preliminary design for an apparatus to test this process at a larger scale (15-25 lb/hr).

Commercially-available equipment that could be adopted with modification to this process has been identified. Quotes for some of the system components have been obtained.

Testing of a New Proprietary Process

An independent organization tested a proprietary process that they developed for cleaning the plastics. Initial tests showed reduction in PCBs on polyolefins from about 22 ppm to 3 ppm. More tests are planned.

We also conducted preliminary tests on a catalytic process to determine if the PCBs can be selectively degraded using ultraviolet (UV) radiation. The initial results were not encouraging. We also conducted tests using a biodegradable solvent. It was successful in reducing the concentration to about 5-10 ppm, but further reduction to below 2 ppm did not appear likely.

The VRP awarded a contract to ECO_2 to conduct tests using their proprietary process.

The criteria for selection of a cleaning method must include: 1) impact of the cleaning process on the properties and marketability of the polymers, 2) nature and cost of disposal of the waste generated by the process, and 3) overall cost of the cleaning process.

Evaluation and Testing of Solvent-Based Washing Systems

Three companies with equipment and/or proprietary washing solvents and solutions that could potentially be used for non-aqueous removal of PCBs from plastics were identified by Troy Polymers, Inc. (TPI):

- Environmental Technology Unlimited (Wilmington, North Carolina);
- Cool Clean Technologies, Inc. (Burnsville, Minnesota); and
- itec Environmental Group, Inc. (Oakdale, California).

Each company was supplied with a sample of plastics with the determined concentration of PCBs of 11 ppm. Samples were washed at the three companies, and the washed samples were evaluated for PCB levels.

Environmental Technology Unlimited performed six treatments of shredder residue plastics, and five out of the six washed samples reduced the PCBs concentration to below 2 ppm. Unfortunately, the company does not have equipment to conduct large-scale testing of the process using plastics. Cool Clean Technologies technology used CO_2 only. The washing failed to remove the PCBs. itec Environmental Group (name changed to ECO_2) reduced PCB levels in the plastics from 11 ppm to 2.8 ppm via solvent washing; no CO_2 treatment, which normally follows the basic process, was used. Further testing was conducted by itec. The plastics were washed using itec's proprietary solvent and then with liquid CO_2. Two samples received by ANL were analyzed and both showed residual PCBs concentration on the order of 5 ppm. Another series of trials was conducted at different process conditions. The residual PCBs were still higher than 2 ppm.

Evaluation and Testing of Commercially-Available, Aqueous-Based Washing Systems

Before testing the solvent-based systems, large-scale cleaning/washing tests were conducted using plastics from shredder residue using aqueous solutions and a surfactant. The objective was to identify the limitations of the various types of existing washing equipment. Testing was done using an ALMCO rotary-drum washer equipped with a dryer and SeKoN centrifuge equipment. The tests were carried out on about 100 lb of plastic chips each. The particles were between 0.2 and 0.5 in. in size. Under a CRADA contract, GraPar Corporation built, for Troy Polymers, Inc. (TPI), and tested a specially designed machine that has a design capacity of about 300 lb/hour of plastics. TPI conducted further testing on this machine in its facilities.

In each of these tests, the washed material was "visually" clean. However, PCBs analyses were highly variable and indicated that, in some cases, the PCBs concentration had increased after washing. As a result, it was determined that the PCBs analysis procedures should be reexamined, as is discussed in the next section.

The results suggest that existing aqueous-based equipment, as is, is not likely to reduce the concentration of PCBs to acceptable levels.

Evaluation of the Variability of PCB Sampling and Analytical Procedures

Experiments were performed to explain the variability in the results, and to develop a consistent procedure for the determination of the concentration of PCBs.

The variability may be due to a number of factors, including sample size, plastics particle size, PCBs extraction procedure, analytical procedures, and/or interference from other compounds. A one-day seminar was held and attended by analytical experts from the United States and overseas to develop recommendations for improved sampling and analysis techniques specific to plastics chips.

To investigate the possible interference of phthalates in the PCBs analysis, a sample of plastics chips derived from shredder residue was thoroughly mixed and then divided into four parts. The first part was analyzed by using gas chromatography and an electron-capture detector (GC-ECD) and by using gas chromatography/mass spectroscopy (GC-MS). The other three parts were spiked with different quantities of phthalates, as shown in Table 1, and the spiked samples were analyzed by using the same two methods. The results show no interference of the phthalates in the PCBs analysis. Interestingly, the GC/MS results were always higher than the GC-ECD results.

Table 1. Effect of phthalates on PCBs analysis.

Weight-Percent of Phthalates added	PCBs Concentration (ppm) by GC/ECD	PCBs Concentration (ppm) by GC/MS
0	4.6+/-0.3	7.9+/-1.0
0.5	4.7+/-0.3	7.4+/-0.2
1.0	5.1+/-0.6	7.0+/-0.4
2.5	4.8+/-0.3	7.4+/-0.3

To investigate the effects of plastics particle size on extraction efficiency of PCBs, a series of laboratory experiments were conducted at TPI on 300-g samples of plastics with two different particle sizes (one made of chips about 0.2 in. in size and the other was granulated to about 0.04 to 0.08 in. in size). Typically in PCBs analyses, extractions are done on a few grams of material, even though the dirt, oil, and the PCBs are not evenly distributed on the shredder residue plastics.

Samples of the plastics before and after washing were analyzed directly by three different laboratories by using standard PCBs analytical procedures. Extracts from nine sonications of 300-g samples were also analyzed for PCBs by three laboratories. The results show that:

1. The results from the three labs are fairly consistent for each set of samples.
2. Direct analysis of the samples from the three labs showed that the concentration of PCBs in the granulated plastics was about 5 ppm and, for the un-granulated, it was 10 ppm. Obviously, the granulated samples have larger surface area per unit mass than the other samples. Therefore, more efficient extraction of PCBs from the plastics would be expected in the case of the granulated chips. Because this was not the case, the results indicate that the particle size does not affect the PCB results. After extraction, the samples all had less than 2 ppm of PCBs, except for one sample that showed 2.8 ppm.
3. Calculation of the concentration of PCBs in the original samples based on the determined PCBs in the hexane extracts (prepared via nine sonications of 300-g samples) showed that the concentrations of PCBs in the granulated samples were comparable with those of the un-granulated samples.
4. These results further indicate that the PCBs are predominantly on the surface and not absorbed in the plastics, otherwise the granulated samples would have shown higher concentrations.

In addition, two of the laboratories identified Aroclor 1242 as the only PCB present, while the third laboratory identified Aroclors 1232 and 1254 as the only two present. TPI also conducted an analysis of various plastics samples by using GC-ECD and GC-MS methods. The results are compared in Table 2. Results from the two methods are in reasonable agreement, even though the GC-MS method showed higher values.

Evaluation of Soxhlet Method for PCBs Extraction

Successful commercialization of technology for recovering polymers from shredder residue depends on a reliable and inexpensive technique to analyze samples for PCBs in the field. The U.S. EPA and European protocols for PCBs analysis were reviewed and experiments were conducted to understand the requirements for on-site analysis. A Soxhlet-based method appears to be appropriate for testing because of its simplicity and because it is among the methods specified in both the U.S. EPA protocols and in the European protocols (Table 3). Limited experiments to define the operating conditions for the Soxhlet method were conducted. The results are discussed below.

Selection of a Solvent

Two solvents were tested: hexane and toluene. Three 120-g samples were extracted with hexane for 8 hours, and another three 120-g samples were extracted with hexane for 24 hours. Similarly, three 120-g samples were extracted with toluene for 8 hours, and another three 120-g samples were extracted with toluene for 24 hours. All extractions were carried out while maintaining the siphoning time at 8–10-minute intervals. This procedure resulted in 24 samples of extracts and 12 samples of extracted plastics that were analyzed. The results indicated that hexane is a better solvent than toluene.

Table 2. Comparison of PCBs analysis using GC-ECD and GC-MS methods (extraction was carried out using hexane at 2,000 pounds per square inch absolute (PSIA) and 100^{o}C).

Sample Type	PCB Concentration, Using GC-ECD (ppm)	PCB Concentration, Using GC-MS (ppm)
Ungranulated Chips	7.55	9.67
Ungranulated Chips	3.70	5.07
Ungranulated Chips	1.50	3.3
Ungranulated Chips	1.35	2.66
Granulated Chips	7.56	9.37
Granulated Chips	0.93	1.82
Granulated Chips	0.82	2.11
Hexane Solution	9.93	9.50
Hexane Solution	8.3	11.13
Hexane Solution	1.41	1.72
Hexane Solution	0.78	0.92
Hexane Solution	0.53	0.65

Table 3. Protocols for PCBs analysis.

Parameter	European Protocols	U.S. EPA's Protocols	Recommended Protocols
Particle size (mm)	0.5	Not specified	1
Sample size for extraction (g)	3	30	30
Extraction equipment	Soxhlet	Sonication Soxhlet Pressurized fluid	Soxhlet
Extraction time	Not specified	Not specified	>/= 4 h Siphoning cycles at 8–10-min intervals
Solvent	Toluene	Hexane 50/50 Hexane/acetone 50/50 Methylene chloride/acetone	Hexane
Analytical method	MS	GC/ECD MS	MS
Quantification method	6 congeners multiplied by 5	Aroclors	Aroclors

Determination of Extraction Time

Three additional 120-g samples were extracted with hexane for 4 hours each. This procedure resulted in six samples of extracts and three samples of extracted plastics that were analyzed. The results indicate that a Soxhlet extraction time of 4 hours is adequate because it reduced the PCBs concentration in the extracted plastics to below the detectable limits in two of the three samples and reduced it in the third to 1 ppm, even though these samples apparently had more PCBs initially, as evidenced by the higher level of PCBs in the solvent.

Determination of Adequate Sample Size

In addition to the six 120-gram samples extracted for 24 hours discussed above, six additional 60-gram samples and six additional 30-g samples were processed and sampled in the same manner as before (24-hour extraction time and same siphoning intervals) by using hexane. The results indicate that a sample size of 30 g appears to be adequate.

Comparison of the U.S. EPA and the European Quantification Methods

Four of the extracts from the 120-g samples that were extracted with hexane for 24 hours and two of the 120-g samples that were extracted with hexane for 8 hours were also quantified by using the European method. The results were essentially identical within analytical errors (Table 4). These results lead to the following conclusions:

1. A conventional Soxhlet extractor using hexane is effective for PCBs extraction from plastics.
2. A total extraction time of 4 hours with siphoning intervals of 8–10 min is adequate for complete extraction of the PCBs.
3. The EPA and the European quantification methodologies yield close results.

Table 4. Comparison of the U.S. EPA and the European quantification methods.

Extraction Time (h)	PCBs According to the EPA Method (ppm)	PCBs According to the European Method (ppm)
24	10.8	9.8
24	9.8	10.9
24	8.0	10.7
24	11.2	11.5
8	11.7	12.3
8	10.8	10.8

Other Accomplishments

Because of potential concern over other SOCs, the VRP hosted and participated in forums and seminars on several related topics including PFOA/PFOS, brominated fire retardants and emerging lead- and mercury-free technologies.

Publications

1. *Overview of Washing Systems for Commercial Cleaning of Plastics Separated from Automotive Shredder Residue*, Sendijarevic, I.; Sendijarevic, V.; Winslow, G.R.; Duranceau, C.M.; Simon, N.L.; Niemiec, S. F.; and Wheeler, C.S., SAE Paper No. 2005-01-0851.
2. *Screening Study to Evaluate Shredder Residue Materials*, Sendijarevec, V.; Simon, N.; Duranceau, C.; Winslow, G.; Williams, R.; Wheeler, C.; Niemiec, S.; and Schomer, D., SAE Paper No. 2004-01-0468.

D. Compatibilization/Compounding Evaluation of Recovered Polymers

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Contractor: ANL
Contract No.: W-31-109-Eng-38

Objectives

- Evaluate the market opportunity for polymers recovered from shredder residue.
- Identify limitations associated with the reuse of the materials as-recovered and determine the need for post-processing technology to upgrade the recovered materials to meet the requirements of the market.

Approach

- Specify standard protocols for material testing, content characterization, and performance properties.
- Determine properties of recovered polymers.
- Conduct blending and pelletizing trials of the recovered polymers.
- Conduct mold trials using recovered polymers.

Accomplishments During This Reporting Period (10/1/06-9/30/07)

- Determined the physical properties of the MBA recovered fractions.

- Provided samples and held discussions with three companies to determine value of recovered polymers as a function of composition of the recovered material.

Prior Accomplishments

Fiscal year (FY) 2006

- Determined the physical properties of the 70% filled acrylonitrile/butadiene/styrene (ABS) fraction.
- Determined the physical properties of two blends of the 70% filled ABS fraction with virgin ABS (10% recovered/90% virgin and 25% recovered/75% virgin).

FY 2005

- Midland Compounding developed a protocol for evaluating the physical properties of recovered plastics.
- Compiled a physical properties database for virgin plastics.
- Determined physical properties of the polyethylene/polypropylene (PP/PE) product recovered from the ANL froth-flotation process and from the PP/PE fraction recovered by the Salyp process.
- Pelletized 1000 pounds of a blend of the ANL-recovered PP/PE product.
- Conducted mold trials of the recovered PP/PE product.

Future Direction

FY 2008 activities will focus on:

- Determine physical properties of upgraded ABS, polystyrene (PS) and polycarbonate(PC)-ABS/PC alloy.
- Pelletize a blend of recovered filled ABS with virgin ABS and compare the properties of the blend with the properties of the virgin material.

Summary

The objectives of this project are (1) to characterize the properties of potentially-recyclable automotive materials and (2) to confirm the technical and economic feasibilities of using those materials in value-added applications.

The project initially focused on establishing the properties of polymeric materials that are recovered as part of the Post-Shred Materials Recovery Technology Development project (see 11.E).

Regardless of the effectiveness of any automotive-materials recovery technology, the materials that will be recovered will be on average 10–15 years old and derived from different sources (automobiles, home appliances and others). In this project, the performance properties of recovered polymers will be compared vis-à-vis new or virgin materials and blends of virgin and recovered materials to establish a database of the properties of recovered automotive polymers. At present, there are few data about the physical properties of polymers recovered from post-consumer durable goods. Absent such data, it is unlikely that sustainable applications for recycled materials will be either identified or developed.

Physical properties testing has been conducted by Midland Compounding, Inc. Midland also conducts composition testing, the results of which are compared with the results of compositional analysis done on recovered materials by ANL.

Blending and pelletizing of the PP/PE recovered from shredder residue by ANL has been tested by Palmer Plastics, Inc. More blending and compounding tests will be done, as required, to achieve the desired performance properties of the recovered materials for target applications.
Mold trials using the recovered PP/PE were also done by MGV Enterprises. More molding tests are planned to confirm the technical and economic feasibilities of using recycled polymers in specific applications.

Polymer Physical Properties and Materials Composition Analysis

Typically, 10-lb samples of recovered materials are used to define physical properties and to characterize the composition of the material.

To quantify the physical properties of the recovered material, a sample is extruded on a single-screw extruder, melt-screened through a 40-mesh screen, molded into American Society for Testing and Materials (ASTM) test bars and plaques, and tested. The molded parts and a random selection of regrind chips from each sample are evaluated for material identification by using infrared (IR) spectroscopy.

Common physical properties that are measured for each sample include the following:

- Melt flow rate (MFR),
- Izod impact,
- Flexural modulus,
- Tensile strength at yield,
- Tensile strength at rupture,
- Elongation at rupture,
- Deflection temperature under load (DTUL),
- Gardner impact, and
- Specific gravity (SG).

Physical Properties and Composition of the PP/PE Recovered from Shredder Residue

The physical properties of PP/PE recovered from different shredder residues by ANL and by Salyp were determined for several samples. The results for the ANL materials are given in Table 1. Properties of commercially-available PP and PE virgin (low-density PE (LDPE) and high-density PE (HDPE)) are presented in Table 2 for comparison. The Izod impact of the recovered material is about three times that of the virgin resins, while the tensile strength of the recovered material is lower than the tensile strength of the virgin resins by about 30%. This phenomenon may be attributed, at least in part, to the presence of thermoplastic olefins (TPO) and rubber in the recovered material, which act as impact modifiers. Recovered samples 8, 9, and 10 listed in Table 1 contained about 2% rubber, while samples 1 through 7 contained about 4% rubber.

The results for the more than 20 PP/PE samples recovered by Salyp from different European and U.S. shredder residues are given in Table 3. The properties of the Salyp-recovered PP/PE are equivalent to the properties of the ANL-recovered PP/PE.

Physical Properties and Composition of the Recovered Filled ABS

Filled ABS recovered by the ANL froth-flotation process followed by removal of the rubber by the ANL dry mechanical process contained 70% filled ABS (specific gravity greater than 1.07 and less than 1.1), 1.5% PS, 8% polyphenylene oxide (PPO), 3% rubber, 3% PP, 7% nylon and 7.5% others. The physical properties of this recovered filled ABS were determined. The results are given in Table 4 and are compared with properties of a commercially-available virgin ABS. Table 4 also shows the properties of two blends of the recovered ABS with virgin ABS (25% recovered/75% virgin and 10% recovered/90% virgin). Interestingly, except for elongation-at-rupture and Gardner impact, the properties of the blends were very close to the properties of the virgin material. Operating condition to upgrade the recovered filled ABS to over 90% has been determined. Properties of the upgraded material will also be established.

Physical Properties of the Polymers Recovered by MBA Polymers

During this reporting period the physical properties of the MBA-recovered fractions were determined by MBA Polymers. The results are given in Table 5.

Table 1. Properties of PP/PE recovered by ANL from different shredder residues.

Property	**Sample 1**	**Sample 2**	**Sample 3**	**Sample 4**	**Sample 5**	**Sample 6**	**Sample 7**	**Sample 8**	**Sample 9**	**Sample 10**	**Average**
MFR, g/10min, 230°C, 2.16 kg	10.5	14.9	7.7	10.1	11.4	7.2	8.7	7.2	8.7	7.2	9.4
Izod impact, ft-lb/in., 73°F	12.3	10.5	11.9	10.8	9	10.7	13.2	1.7	2.8	3.3	8.6
Flex mod., 1% secant, 1,000 psi	83	73	89	84	82	101	112	126	127	113	99.0
Tensile strength at yield, 1,000 psi	2.6	2.2	2.7	2.6	2.4	2.8	3.1	3.4	3.3	3.1	2.8
Tensile strength at rupture, 1,000 psi	0.8	1.2	2.1	1.9	1.4	2.5	2.0	3.1	3.1	2.9	2.1
Elongation at yield, %	23.0	20.8	21.1	22.8	20. 6	20. 6	17.1	**	**	**	24.3
Elongation at rupture, %	132	78	233	154	82	251	229	12	14	13	119.8
DTUL, °F (at 66 psi)	131	131	134	134	138	147	155	**	171	160	145
Gardner impact, 73°F, in.- lb	104	88	136	96	56	144	184	20	32	40	90.0
SG, g/cc	0.94	0.95	0.94	0.95	0.94	0.93	0.93	0.94	0.94	0.94	0.94

** Not tested

Table 2. Comparison of recovered PP/PE with commercial grades of PP & PE (Boedeker) (http://www.boedeker.com/mtable.htm), unless specified otherwise.

Property	**PP Homo-Polymer**	**PP Co-Polymer**	**PP Flame Retarded**	**Standard PP Co-polymer**	**LDPE**	**HDPE**
MFR, (g/10 min), 230°C	0.5-136*					
Izod impact, ft-lb/in.	1.9	7.5	0.65	0.7	No Break	3
Flex Mod, 1,000 psi	180	160	145	120	200	125
Tensile Strength, 1,000 psi	4.8	4.8	4.3	5.2	2.0	4.6
Elongation, %	12	23	28	600	600	900
DTUL, °F (at 66 psi)	210	173	106	210	110	--
SG, g/cc	0.905	0.897	0.988	0.90	0.92	0.95
Gardner impact, 73°F, in.-lb	0.9-22*					

- Data from http://www.ed-cam.com/materials/propylene_molded.asp. Ranges are for with and without additives.

Table 3. Properties of PP/PE recovered by Salyp from different shredder residues.

Property	**Salyp Data**
MFR, (g/10 min), 230°C	2.3–4.6
Izod impact, (ft-lb/in.) 73°F	4.7–13.3
Flex mod., 1%, secant, 1,000 psi	81.7–116.5
Tensile strength at yield, 1,000 psi	2.4–2.9
Tensile strength at rupture, 1,000 psi	2.2–2.8
Elongation at rupture, %	19–57
DTUL, °F (at 66 psi)	150–169
Gardner impact, 73°F, in.-lb	190–240
SG, g/cc	0.93

Table 4. Properties of recovered filled ABS, virgin ABS and blends of the two materials.

Property	Recovered Filled ABS	Virgin ABS (342 EZ)	90%Virgin/ 10% Recovered	75% Virgin/ 25% Recovered
MFR, g/10min, 230C, 3.8 kg	3.9	6.5	7.6	6.4
Izod Impact, ft.lbs./in., 73F	0.9	3.8	3.0	2.6
Flex Mod, 1% secant, 1,000 psi	324	296	299	302
Tensile strength at yield, psi	4982	5546	5392	5312
Tensile strength at rupture, psi	4956	4459	4544	4930
Elongation at rupture, %	2	56	9	6
DTUL, °F (at 264 psi)	162	165	166	164
Gardner Impact, 73F, in.lbs.	0	>320	32	8
SG, g/cc	1.08	1.05	1.05	1.06

During this reporting period, we also sent samples for evaluation to two plastics recyclers: MRC Polymers, Inc. in Illinois and Enviroplas, Inc. in Indiana. We also held discussions with Standard Plaque, Inc. in Michigan to discuss sample-preparation methodologies for analysis of recovered materials with different levels of impurities.

Polymer Physical Properties Database

A physical properties database has been compiled so that the physical properties of the recovered polymers can be compared with general purpose virgin polymers.

General-purpose physical properties have been compiled for the following plastics:

- ABS,
- Nylon (6 cast, 6/6 extruded, 30% glass filled),
- PPO (unfilled, 30% glass filled),
- PC
- PE, LDPE, HDPE, ultra-high-molecular-weight [UHMW] PE,
- PP,
- PS (general-purpose, high-impact), and
- Polyvinyl chloride (PVC).

The VRP had previously compiled physical properties data on selected polymers that were recovered during the U.S. field trials. These materials were recovered by disassembly. The data from these polymers are included in the database so that the physical properties of materials recovered by disassembly can be compared with those of materials that are recovered from post-shred operations, Table 6 ("USCAR U.S. Field Trial for Automotive Polymers Recycling," by W.W. Gallmeyer, C.M. Duranceau, R. L. Williams and G.R. Winslow, SAE Paper # 2003-01-0645, 2003). Table 5 gives the properties of PP dismantled from automobiles as part of the USCAR U.S. field trial. The recovered PP was reported to have a specific gravity of 0.915 and it is made of 99.2% PP, 0.4% PE and 0.4% ABS. The differences in the properties of the dismantled PP and the PP/PE recovered from shredder residue are also compared in Table 6.

Table 5. Properties of plastics recovered by MBA.

Sample	ABS	HIPS*	PP filled	PP unfilled
MFR, g/10min, 230C, 3.8 kg	N/A	N/A	N/A	5.5
MFR, g/10min, 200C, 5 kg	N/A	N/A	2.5	N/A
MFR, g/10min, 230C, 2.16 kg	4.6	7.9	N/A	N/A
Izod Impact, ft.lbs./in., 73F	10.3	1.6	1.8	3.4
Flex Mod, 1% secant, 1,000 psi	130	293	315	361
Tensile strength at yield, psi	3029	3779	4319	6291
Tensile strength at rupture, psi	1773	2365	4011	5623
Elongation at rupture, %	50	32	19	6
DTUL, °F (at 264 psi)	N/A	N/A	162	170
DTUL, °F (at 66 psi ,	150	232	N/A	N/A
Gardner Impact, 73F, in.lbs.	216	64	16	16
SG, g/cc	0.94	1.07	1.05	1.06

* HIPS = high-impact polystyrene

Table 6. Properties of PP Dismantled of Cars as Part of the USCAR U.S. Field Trial.

Property	Recovered, Extruded	Recovered Flakes, Sample #1	Average Properties of Recovered PP/PE*
MFR, (g/10 min), 230°C	19.9	17	9.4
Izod impact (ft-lb/in.) 73°F	1.8	1.8	8.6
Flex. Mod., 1%, secant, 1,000 psi	136.9	131.9	99
Tensile Strength at Yield, 1,000 psi	3.130	3.136	2.8
Elongation at Yield, %	19	18	24.3
Tensile Strength at Rupture, 1000 psi	2.388	2.384	2.1
Elongation at Rupture, %	59	60	119.8
DTUL, ^{o}F (at 66 psi)	129.7	136.5	145

* From Table 1

The differences are not significant and do not affect the usefulness of the material. For example, the specific gravity of the material recovered at ANL is about 0.94 compare to 0.915 for the dismantled material. The MFR reflects the largest difference: 17 for one of the two samples of the dismantled flakes versus about 9.4 for the material recovered from shredder residue. The USCAR study also found that the properties of the dismantled PP responded as expected when additives were added to the PP. For example, the Izod impact increased from less than 2 ft-lb/in. to about 11 when 10% of an impact modifier was added, and to about 14 when 20% were added (USCAR U.S. Field Trial for Automotive Polymers Recycling: Interim Findings", by W. W. Orr, SAE Paper # 2000-01-0735, 2000).

Blending and Pelletizing of Recovered PP/PE

250 pounds of PP/PE recovered by ANL were blended with 750 lb of supplemental PP copolymer regrind for 15 minutes. The blended material was then run through an extruder and pelletized. The general appearance of the final pellet was excellent (Figure 1). Properties of the recovered material used in blending and the properties of the regrind and of the resulting pellets are shown in Table 7. Standard pelletizing conditions were used. Barrel heats were set from 365°F at the rear barrel zone and increased progressively to 390°F at the front, with six heat zones in between. Screen-changer and breaker-plate heats were set at 405°F, and die heats were set at 395°F. Melt temperature was recorded as 460°F, and drive-load and screw speed were set at 60% and 67.5% of the maximum values, respectively.

Figure 1. Pelletized PP/PE product recovered from shredder residue.

Material output was recorded as 1,400 lb/h. Extra-fine screen packs were used (20/20/20/60/100/20 mesh screens) to remove impurities because this was the first time this material has been tried.

In addition, while screen changes are typically performed at pressure differences between 500 psi and 1,000 psi, in this test, changes were performed when the pressure exceeded 500 psi to safeguard against puncturing a screen pack and losing material. Because extra-fine screen packs were used in the test, screen changes were required approximately every five minutes. The results indicated that the recovered PP/PE can be blended with other olefinic regrind and pelletized by using standard processes and equipment.

Mold Trials

Three types of auto parts were molded by MGV Enterprises using ANL-recovered PP/PE from shredder residue: knee bolsters, battery trays, and steering column covers (Figure 2). A standard molding machine was used in these trails. No changes to the standard conditions were required to run the recovered material. The limited testing done on the recovered PP/PE fraction shows that quality products, including auto parts, may be produced from the recovered materials. Additives and/or modifiers may be added to meet the specifications of some products.

Recovered Rubber/Plastics Material

A mixed-rubber fraction with about 20% by weight mixed plastics was recovered. A sample of the recovered material was sent for testing by the "TireCycle" process used for recycling rubber. Preliminary tests done on the recovered material indicated that it may be suitable for making construction products, such as roofing shingles. The presence of the plastics in the mixed-rubber material appeared to improve its overall properties, especially its stiffness.

Publications

None.

Table 7. Properties of recovered PP/PE when mixed with regrind.

Property	Argonne, As-Recovered Sample 9 (see Table 1)	Regrind As-Is	Pelletized Blend
MFR (g/10 min), 230°C	8.7	3.1	9.2
Izod impact (ft-lb/in.) 73°F	2.8	13.6	10.4
Flex. mod., 1%, secant, 1,000 psi	127	157	136
Tensile strength at yield, 1,000 psi	3.3	3.7	3.4
Tensile strength at rupture, 1,000 psi	3.1	2.9	2.3
Elongation at rupture, %	14	125	57
DTUL, °F (at 66 psi)	171	197	176
Gardner impact, 73°F, in.-lb	32	>320	132
SG, g/cc	0.94	0.91	0.92

Figure 2. Auto parts molded from PP/PE recovered from shredder residue.

E. Post-Shred Materials-Recovery Technology Development

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Participants:
This project is conducted as part of a Cooperative Research and Development Agreement (CRADA) among ANL, the Vehicle Recycling Partnership (VRP) of the United States Council for Automotive Research (USCAR), and the American Chemistry Council-Plastics Division (ACC-PD, formerly the American Plastics Council (APC)).

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Changing World Technologies (CWT) is cost-sharing on the evaluation of its thermal depolymerization process. The Polyurethanes Recycle and Recovery Council (PURCC) is also participating and cost-sharing on the evaluation of the Troy Polymers, Inc. (TPI), polyurethane glycolysis process.

Contractor: ANL
Contract No.: W-31-109-Eng-38

Objective

- Develop technology for the cost-effective recovery of materials from post-shred residues.

Approach

- Characterize shredder residue (SR) from a number of sources to determine composition variability.

- Conduct bench-scale and large-scale process/technology tests to benchmark technology.

- Build and operate a pilot-plant for the separation of SR to produce recovered materials for market evaluation and to provide “control” samples of materials for testing of alternative technologies, as appropriate.

- Conduct cost and performance analysis of alternative technologies to establish the business case for the technologies and to identify technology gaps.

Accomplishments During this Reporting Period (10/1/06-9/30/07)

- ANL was approached by two shredders who expressed interest in building large-scale plants to validate the results obtained in the ANL pilot-plant. Discussions with the shredders are underway.

- Achievements during this reporting period are given below.

Mechanical Separation of Shredder Residue

- Completed testing of 10,000 pounds of SR out of a 30,000 pound batch from a shredder who is interested in building a plant to validate the ANL technology.
- Completed engineering designs of the basic Mechanical Separation System including equipment specifications, equipment cost, operating requirements, and utility requirements.
- Conducted cost analysis of the process including sensitivity of the cost of producing the polymer concentrate to yield, value of by-products (ferrous, non-ferrous metals) and cost of utilities.
- Completed the recovery of plastics and metals from fines (< 0.25 inch).

Froth-Flotation Separation of the Plastics

- Designed, built and tested a 5,000 lb/hr plastics separation sink/float module.
- Defined process conditions for upgrading the polycarbonate(PC)-acrylonotrile-butadiene-styrene(ABS)/PC alloy concentrate to 90-95%.
- Completed engineering designs of the basic froth-flotation process to include equipment specifications, equipment cost, operating requirements, and utility requirements.
- Proved the technical feasibility of upgrading the filled ABS from 70% to greater than 90% and of the unfilled ABS and polystyrene (PS) from the ABS/PS concentrate, to greater than 90% and 85%, respectively.

CWT's Thermochemical Process for Producing Hydrocarbon Liquids

- Evaluation of the CWT technology was continued. Preparation for the 2,000 lb. test was started.

Emerging Technologies for the Rapid Identification and Sorting of Plastics

- Conducted performance testing and cost analysis of color sorter and near infrared (NIR) systems for plastics, wood and rubber separation.

MBA's Plastics Separation Test

- Tests have been completed and the report was submitted.

Energy Anew Process for Recycling Fines

- Energy Anew completed a study to recover metals and polymers from fines (<7/8 in. in size).

On To Technologies

- The VRP has awarded a contract to OnTo Technology of Bend, Oregon to develop technology to recycle batteries used in hybrid and electric vehicles. Testing has started. Initial results are promising.

Prior Accomplishments

Fiscal Year (FY) 2006

Mechanical Separation of Shredder Residue

- Ran one (1) 10-ton production campaign (fractions supplied to commercial equipment vendors for performance verification).
- Initiated engineering design for full-scale, bulk-separation system.
- Obtained budgetary quotes from vendors for major equipment.
- Confirmed performance of commercial equipment with field trials by vendors/ANL of the as-is SR and of the fractions generated at ANL.
- Conducted preliminary investigation of costs and performance of commercial color sorters, electrostatic separators, and IR sorters for removal of wood and rubber.

- Developed a dry process for separating rubber from plastics.

Froth-Flotation Process for Recovering Plastics

- Ran first campaign of the middling plastics fraction.
- Recovered a 60%-unfilled ABS/PS and a 50%-filled ABS concentrates from the middling plastics.
- Upgraded the filled ABS concentrate from 50% to 70% and defined process conditions to further upgrade this fraction to 90%.
- Defined process conditions for separating and recovering unfilled ABS and PS from the unfilled ABS/PS concentrate, to greater than 90% and 85%, respectively.
- Defined process conditions for recovering an 85% PC-ABS/PC alloy from the middling plastics.

Other Accomplishments

- Completed testing of Troy Polymers' (TPI) process for conversion of urethane foam to polyol initiators. Over 1,200 lb of foam were used and produced over 100 gallons of polyol initiators.
- Conducted pilot-scale testing of CWT process for converting SR to fuels. Pre-processed SR from another shredder was evaluated and shipped to CWT for further testing.
- Completed a large-scale, plastics-separation test at MBA Polymers Inc. using a plastic concentrate produced by Salyp's mechanical-separation system.
- Completed testing of the VW-SiCon plastics-separation process.

FY 2005

Mechanical Separation of SR

- Ran four (4) 15-ton production campaigns and conducted material balance on all runs.
- Modified bulk-separation operation resulting in an increase in polymer yield in concentrate from 40% to over 90%.
- Conducted trials on gravity tables, mineral jigs, and a kinetic density separator in the U.S. and Europe primarily for removal of wood and rubber.

Froth-Flotation Process for Recovering Plastics

- Conducted bench-scale research on settling velocities and density distributions of actual SR polymers including the wood and rubber.
- Ran a production campaign of the base process with polymer concentrate to yield three polymer fractions; the polyolefin fraction: middling plastic fraction, and the heavies plastic fraction.
- Developed a wet process for removal of wood and rubber from the recovered polyolefin fraction.
- Recovered 5,000 pounds of polypropylene(PP)/polyethylene(PE) essentially free of wood and rubber.

Other Accomplishments

- Conducted bench-scale tests and in a five-gallon reactor of TPI's glycolysis process for conversion of urethane foam to polyol initiators.
- Conducted bench and pilot-scale testing of CWT's thermal depolymerization process for converting SR to fuels.
- Developed an Excel-based process cost model that incorporates two primary modules for the recovery of automotive plastics. The first module includes the unit operations required for recovering a plastics concentrate from SR and the second module includes the unit operations required to recover selected plastics from the mixed plastics concentrates.

FY 2004

Mechanical Separation of SR

- Completed construction, shakedown and start-up of the bulk-separation facility.
- Ran six (6) 5-ton trial campaigns.

Froth-Flotation Process for Recovering Plastics

- Completed construction and shakedown of the pilot-plant with electronics plastics.
- Ran a shakedown campaign with SR polymer concentrate.

Other Accomplishments

- Completed large-scale tests of Salyp's "thermoplastics sorting" technology by using residue from two European locations and one U.S. location as feed materials

FY 2003

- Initiated construction of bulk-separation facility.
- Initiated construction of froth-flotation pilot-plant.

Future Direction

The FY 2008 plans are as follows:

Mechanical Separation of SR

- Include recovery of materials from fines in the engineering design.
- Process SR generated by controlled shredding of selected items (autos only).
- Shred automotive nano-composites to determine potential release of nano particles.
- Produce fractions of SR in support of other collaborators.

Froth-Flotation Process for Recovering Plastics

- Produce polymer fractions for property determination and market evaluation.
- Continue development of a predictive simulation model for determining appropriate separation operating and solution conditions that can affect gravity and froth-flotation separation of selected polymer materials for separation and recovery of polymers from a SR concentrate.
- Development of bench-scale experimental design to provide requisite empirical data for the predictive simulation model.
- Conduct process-improvement studies to increase value and reduce cost.

Other Future Plans

- Complete the 2,000-lb sample run. Evaluation of the CWT technology will be completed and recommendations for path forward will be made.
- Develop NIR spectroscopy libraries for SR plastics.
- Evaluate new processes for recycling SR including conversion to fuels and chemicals.
- Determine recycling research needs of new lightweighting materials.
- Identify an industrial partner to demonstrate the ANL technology.
- Continue development of the OnTo technology for battery recycling and Energy Anew fines recycling.

Summary

The objective of this project is to develop technology for the cost-effective recovery of materials from post-shred residues. Research will provide data essential to establishing a business case for sustainable recycling of automotive materials from post-shred residue. Technologies specific to the recovery of materials from post-shred material streams are being evaluated and demonstrated to determine their commercial viability. The performance (e.g., yield, purity, efficiency, and cost) of emerging technologies will be determined to enable the development of an integrated process for recovering materials from shredder residue.

Research has been completed on the Salyp, MBA, Troy Polymers, VW-SiCon and ANL mechanical-separation processes. Research is ongoing on the ANL froth-flotation, CWT, OnTo, and Energy Anew processes.

Two shredders have expressed interest in building a plant on their sites to validate the results obtained in ANL's pilot-plant. About 5 tons of residue from one of their modern plants was processed to determine if a business case exists.

Characterization of SR

Over 100 tons of SRs from six facilities were processed in ANL's mechanical-separation plant. Table 1 shows the average composition of the different fractions produced. We observed:

- Large variations in fines, metals, rubber and wood, and
- Less variation in the composition of the plastics fraction.

The mass fractions of the polymer concentrate separated from different shredder residues showed little variation, and the weight percent (wt%) of the polymer concentrate recovered from 8 runs totaling 80,000 lbs of shredder residue from a given source conducted over a six-month period was reasonably consistent (41%, 26%, 36%, 39%, 45%, 37%, 43% and 45%,; average 40%). The composition of the different polymer concentrates was also similar, Figure 1.

ANL Pilot-Plant

ANL's pilot-plant consists of a dry mechanical-separation facility and a wet-density/froth-flotation facility. The pilot-plant is used to:

1. Recover materials from shredder residue,
2. Conduct process improvement studies
3. Generate design and scale-up data
4. Produce samples for evaluation,
5. Define the effectiveness of alternative separation technologies and systems, and
6. Serve as a user/demonstration facility.

Mechanical Separation Pilot-Plant

The mechanical-separation facility processes raw SR to yield a polymer concentrate, ferrous and non-ferrous concentrates, a fines fraction and other fractions. The plant achieved over 90% recovery of the thermoplastics targeted for recovery as a polymer concentrate and over 95% recovery of the metals in the SR.

The polymer concentrate included high and varying amounts of wood and rubber. Wood was about 1–4 wt%. In Figure 1, the wood is included in the "non-polymers."

Separation of Wood and Rubber

Trials were conducted on commercially-available air aspirators, air classifiers, gravity tables, and mineral jigs to remove wood and/or rubber from the polymer concentrate. This equipment did not yield satisfactory results. Trials using modified wet-separation approaches removed almost 100% of the wood and over 90% of the rubber with a nominal loss (~ 5%) of the plastics.

A modular dry process for separating rubber from plastics has been tested at rates of up to 200 lb/hr of polymer concentrate. The process is able to separate over 75% of the rubber and produce a rubber fraction containing less than 10% of non-rubber material.

Table 1. Streams produced by mechanical separation of an average shredder residue.

	Shredder Residue	Oversized Heavies	Oversized Foam rich	Fines+	Ferrous Rich	Non-Ferrous Rich	Lights	Polymer Concentrate
Weight (lbs)	40,000	2,148	756	17,640	656	1,468	1,968	10,044
PP	1,075	0	0	0	17	33	129	897
PP (filled)	403	0	0	0	0	0	9	393
ABS	763	0	0	0	5	9	13	737
PE	941	0	0	0	9	18	85	830
HIPS	261	0	0	0	4	8	15	234
Nylon	379	0	0	0	4	9	19	347
PVC	512	0	0	0	0	0	0	511
PPO	139	0	0	0	0	0	4	135
PC-ABS	151	0	0	0	0	0	1	150
PC	212	0	0	0	0	0	12	200
Other Plastics	597	0	0	0	1	0	17	579
Rubber	4,505	20	0	0	6	172	61	4,246
PU	273	3	0	0	1	23	9	237
Wood	239	0	0	0	0	0	0	239
Metals	2,911	1,117	0	0	590	954	0	249
Foam, Fiber and others	21,320	1,008	756	17,640 +	19	241	1,597	59
Moisture	5,320	0	0	0	0	0	0	0
Total	40,000	2,148	756	17,640	656	1,468	1,968	10,044

+ Fines are material smaller than 0.25 inch in size and also contain some polymers and metals. HIPS=high-impact polystyrene, PVC=polyvinyl chloride, PU=polyurethane. See text for other defintions.

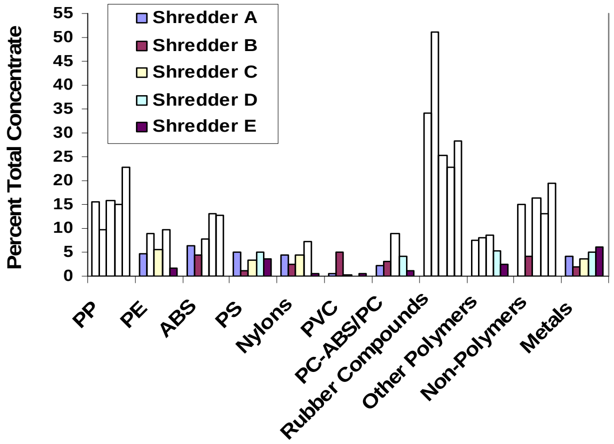

Figure 1. Composition of polymer concentrates from different shredder residues.

Froth-Flotation Pilot Plant

This facility includes six continuous stages for the separation of targeted plastics from the polymer concentrate. A shakedown of the facility was conducted using 4,000 lb of post-consumer electronics and appliance mixed plastics and by using a mixture of colored plastics. These trials confirmed the effectiveness of the basic system. Over 30,000 lb of polymer concentrate from shredder residue has been processed in this facility. The recovered fractions are described below:

Recovered PP/PE Fraction

More than 5,000 lb of an unfilled PP/PE fraction that is over 95% PP/PE have been consistently produced. It contains less than 0.2% wood and less than 4% rubber. However, when the material was palletized, most of the rubber in this fraction was compatible with the PP/PE. The recovered PP/PE has properties similar to those of some commercially-available PP materials. The unfilled PP/PE product is about 5%–6% of the starting shredder residue weight. Table 2 summarizes the recoverable plastics from 10,000 pounds of typical shredder residue.

Filled ABS Fraction
ABS that has a specific gravity between 1.07 and 1.1 was isolated by the basic froth-flotation process as an ABS concentrate, Table 2. It contains 50% ABS, 20% rubber, 10% rigid urethane rubber, and 20% of other materials. Removing wood and rubber increased the ABS concentration to 70% and reduced the rubber and urethane to 3% and 2%, respectively. When this material was blended with virgin ABS at 10% and 25% recovered material, the properties of the blends were slightly different from the properties of the virgin ABS. Laboratory tests have established process conditions to increase the ABS concentration to over 90%.

Unfilled ABS and PS
A fraction of ABS, PS and PPO that has a specific gravity between 1.0 and 1.07 (43% ABS, 22% PS, 7% PPO and 28% other materials including rubber and some wood) was produced by the basic process. Lab tests separated this fraction and produced fractions with over 90% ABS and over 85% PS/PPO.

PC-ABS/PC Alloy
Laboratory tests produced a PC-ABS/PC fraction having a combined concentration of over 85%. Work is ongoing to isolate a large sample of ABS/PC-PC fraction for further evaluation.

PVC: Recovery of the above fractions leaves behind a fraction made of high-specific-gravity materials. Rubber constitutes over 50% of the total and metals about 5%. Separating rubber, metals, glass and rocks from this stream leaves a fraction containing over 50% PVC. This fraction is also rich in glass-filled nylons.

Rubber
Recovered-rubber concentrate from the basic process was upgraded by the ANL dry rubber-separation process to over 90% rubber. The other 10% was mostly plastics.

In summary, we have recovered the unfilled polyolefins as a potentially useable product and isolated the filled ABS, unfilled ABS/PS, PC-ABS/PC and PVC into more manageable fractions.

The basic wet process has also produced a styrenics fraction containing over 75% by weight "targeted plastics" primarily ABS, PS, PPO, and filled PP. MBA Polymers established that this fraction can be separated to produce recyclable products. This may be a more cost-effective approach than trying to separate it by froth flotation to produce high-grade ABS and PS.

A 5,000 lb/hr continuous-flotation module has been designed, built and tested. Testing of this module using over 10,000 pounds of various polymer fractions provided necessary information for designing and building a full-scale plant.

Development of a Process Flowsheet

A process conceptual design for a 20 ton/hr mechanical-separation system has been developed. The key steps in the process include: 1) a device to separate large metal chunks and rocks; 2) a screen separator to separate large non-metallic pieces such as fabrics, PU foam and tire rubber pieces; 3) a shredder to size reduce the material; 4) a vibrating screen or a trommel to separate "fines", smaller than ¼ inch; 5) a magnetic separator to recover ferrous metals; 6) an eddy-current separator to recover non-ferrous metals; 7) a granulator to granulate the remaining material; and, 8) an air classifier to remove "lights" from the granulated material. Tests using SR were conducted to evaluate the cost, performance, and maintenance requirements of various equipment that was proposed for the conceptual design.

We also conducted sensitivity of the cost of producing the polymer concentrate to yield per ton of SR, value of the by-products (ferrous, non-ferrous, etc.), cost of utilities, number of operating shifts, cost-of-capital, etc. The production cost can be as low as $0.02 per pound of polymer concentrate recovered for a plant operating three shifts per day and the polymer concentrate is about 45% of SR, to over $0.075 per pound for a plant operating one shift and the polymer concentrate is only 15% of SR. The value of the recovered metals has a significant impact on the overall cost. The value of the metals will depend on their recoverable concentration in the SR and on the composition of the metal concentrates produced.

Table 2. Composition of an average polymer concentrate and recovered polymer fractions.

	Polymer Concentrate	PP/PE Product	ABS Product	ABS/PC Product	Rubber Product	HIPS/ABS Concentrate	Mixed Plastics	Mixed Stream*
Weight (lbs)	10,044	1,736	141	108	689	856	1,203	5,311
PP	897	827	0	0	0	0	63	7
PP (filled)	393	0	0	0	11	43	194	146
ABS	737	0	105	2	0	365	176	88
PE	830	787	0	0	10	12	21	0
HIPS	234	0	2	0	0	186	25	21
Nylon	347	0	5	0	0	5	42	296
PVC	511	0	0	0	3	0	123	385
PPO	135	0	13	1	0	62	21	37
PC-ABS	150	0	0	6	0	0	0	143
PC	200	0	0	85	1	0	19	94
Other Plastics	579	0	9	2	2	12	8	547
Rubber	4,246	90	2	9	628	104	263	3,149
PU	237	21	4	2	18	0	96	96
Wood	239	0	1	0	17	66	146	8
Metals	249	0	0	0	0	0	0	249
Foam, Fiber and others	59	10	0	0	0	1	5	42
Total	10,044	1,736	141	108	689	856	1,203	5,311

* Rubber and metals are to be recovered from these streams

We conducted performance testing and cost analysis of color sorter and IR sorters. The following systems were tested: 1) Satake (chip monochromatic and IR); 2) Key (chip full-color and laser); 3) MSS (full-color and IR); 4) S+S Separation and Sorting Technology (full-color and IR); 5) NRT (chip full-spectrum and IR – See 11.F); and 6) PROTEC (chip full-spectrum). Based on the results of these tests, the ANL wet separation for wood and rubber removal is the most economical for separating the wood and the rubber from the plastics. The primary reason is the high loss of the targeted plastics by the optical sorters. Further, at this time, these systems can not produce marketable polymer products from the polymer concentrate.

Recycling of Fines

ANL conducted tests to recover polymers and metals from the <0.25 in. fines. The polymer concentrate recovered from the material in the 2-mm to 6-mm size range was about 50 wt% of the weight of the fines fraction. It contained up to 50% by wt rubber and about 20 wt% plastics. A sample of this concentrate was processed in the wet-separation system to produce a polyolefin concentrate, a styrenics concentrate, a mixed plastic and rubber concentrate, and recover the residual metals as a metal concentrate. Twenty percent of the plastics had specific gravity < 1.

The ferrous material, including iron oxides, in the fines has been reduced significantly compared to years ago. This is due to shredders using more-efficient metal-separation equipment.

Processing of Polymer Concentrate at MBA

Salyp built a mechanical-separation plant that started with ANL's original mechanical-separation system and added an optical sorter and a plastics washing system. Salyp's starting SR contained less rubber and wood than the U.S. residue. MBA processed about 40,000 pounds of polymer concentrate produced by Salyp. Five materials were recovered: 1) Polyolefin "A", 2) Polyolefin "B", 3) filled PP, 4) ABS and 5) HIPS. The total yield of these products was estimated to be 48.5%

of the plastics-rich fraction. This yield is 88% of the amounts of these plastics in the feed material. The products were extruded, molded and tested. The properties were reported to be "encouraging" and it is expected that most of the products could be used in some durable-goods applications.

CWT

CWT processed a mixed SR stream after separating the < 1/16-in. fines (~36% by weight). About 700 lb of the remaining material were processed along with 80 lb of tires and 1,700 lb of used motor oil. The products were hydrocarbon oil (84%), a fuel-gas (10%), and a solid carbon product (6%). Distillation of the oil generated gasoline (12%), diesel (32%), heavy-hydrocarbon oils (15%), and gas (3%).

Polychlorinated biphenyls (PCBs) in the input SR were 21.8 parts per million (ppm); however, in the products, the PCBs were below the detection limits. This indicated that PCBs degrade during the process. No measurable concentrations of heavy metals were found in distillate-cut #3. The char contained significant amounts of several metals. The heavy oil from the dissolver contained about 3,200 ppm of total chlorine, but no chlorine was found in the light distillates; only 14 ppm was found in the heavier distillate and 11 ppm in the distillate bottoms. Bromine was found in the heavy oil from the dissolver (~135 ppm). No bromine was found in the output liquid products. Bromine was found in char (87 ppm).

The tire rubber and the oil samples contained 17,200 ppm and 1,600 ppm of sulfur, respectively. This indicates that sulfur compounds degraded in the process.

A pre-processed organic fraction (about 2,000 pounds) derived from SR has been shipped to CWT for next trials. CWT removed the inorganic fines from this material. The prepared organic fraction will be processed to validate the earlier results.

TPI

Foam from SR was converted by the TPI process to polyol initiators at yields of about 88% and 72%, for clean and dirty foam, respectively. Activated carbons reduced the concentration of PCBs in the products to < 2 ppm. Over 1,200 lb of shredder residue foam have been processed in a 5-gallon reactor, and produced over 100 gal of polyol initiator.

Twenty gallons of the polyol initiator were propoxylated. The recycled polyols were successfully tested for making rigid foams. They required less or no catalysts and had better flame resistance than the foams made with virgin polyols. The results established the technical feasibility of the process. Initial economic analysis of the process indicated that the glycolysis process is potentially economical. TPI is working with PU-foam producers to move the technology forward.

Energy Anew Recycling of Fines

Energy Anew conducted tests on 300-pound samples of fines (smaller than 1 in.). SR was screened using a 7/8-in. screen and the material that passed the screen was processed to yield organic- and inorganic-rich fractions. A combination of hydrocycloning, screening, rising current, wet tabling, magnet, and grinding were utilized to give ferrous and non-ferrous metal, organic, and inorganic/sand fractions. Economic analysis of the process showed that recovery of the metal and sand may be viable. Energy Anew is working to scale-up the process.

The ACC-PD has co-funded a study with Plastics Europe to study the synergies of co-combustion of plastics-rich fuels and biomass. The study will review technologies, co-combustion experience, environmental compatibility and synergies of biomass plastics co-combustion

OnTo Technology

The VRP has awarded a contract to OnTo Technology of Bend, Oregon to research and develop technology to recover materials from batteries used in hybrid and electric vehicles. Preliminary tests were conducted during this reporting period and the results are promising.

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F. Development of High-Speed Multispectral Imaging for Sorting Automotive Plastics

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Objective

- Develop technology for the cost-effective sorting of automotive plastics recovered from post-shred residues.

Approach

- Determine types of automobile shredder residue (SR) plastics that potentially can be sorted using the proposed multispectral sensing technology.
- Construct a bench-scale unit comprising rudimentary features of the new technology.
- Conduct bench-scale evaluations to determine technical feasibility for development of the proposed technology.
- In Phase II, develop the new high-peed multispectral sensing technology required to perform commercial-scale sorting of automotive plastics recoverable from SR.
- In Phase II, build and test a prototype sorter incorporating the new multispectral sensing technology for application to sorting of plastics recovered from SR.
- Conduct cost and performance analysis of the new sorting technology to establish technical viability and commercial feasibility for deployment and use of the new sorting technology.

Accomplishments During this Reporting Period (10/1/06-9/30/07)

The Phase I research program performance period was from 06/28/2006 through 03/27/2007 and progress reported herein covers this performance period.

Types of Plastics for Sorting Derived from Automobile SR

- Met with personnel from the Transportation Technology R&D Center at Argonne National Laboratory (ANL), toured the Vehicle Recycling Partnership (VRP) plant, and learned about various plastics derived from automobile shredder residue.
- Received and preliminarily evaluated SR plastics samples from ANL.

Bench-Scale System Demonstration and Feasibility Determination

- Determined preliminary technical requirements for an optical module for integration with a sensing module.
- Determined technical feasibility of developing a primary beam-shaping module which collects, collimates, and condenses broadband radiation for transmission to a sensing module.

- Determined the technical requirements for an analog amplification circuit that converts the detector response into an appropriate voltage signal.
- Designed and constructed a bench-scale unit comprising rudimentary features of the proposed detection system.
- Demonstrated technical feasibility for development of the new sensing technology.

Future Direction

Effort in FY2008 will be performed through a recently-awarded SBIR Phase II project. In summary, the Phase II plan is as follows:

- Develop the new high-speed multispectral sensing technology required to perform commercial scale sorting of automotive plastics recoverable from SR.
- Integrate the new sensing technology with an electronics and computing platform incorporating a real-time operating system interfaced to a precision air-jet ejection array system in order to enable high-speed sorting.
- Develop and incorporate sensing, detection, and sorting algorithms and a run-time, human-to-machine, input/output (I/O) interface for system configuration and control.
- Build and test a prototype sorting system incorporating the new multispectral sensing technology with the operating system platform, software, and sorting hardware for application to sorting of plastics recovered from shredder residue.
- Conduct cost and performance analysis of the new sorting technology to establish technical viability and commercial feasibility for deployment and use of the new sorting technology.

Summary

In the Phase I research, NRT has designed and constructed a bench-scale sensing and detection system capable of simultaneously acquiring detection signals from multiple spatial locations thereby potentially enabling high-volume sorting of automotive plastics derived from SR. As a “proof of technology” prototype, this system has enabled us to explore and evaluate the challenges arising from concurrent acquisitions of signals from multiple sensing sites. Assessment of this prototype leads us to believe that it is possible to construct a low-cost, compact, rugged system for industrial processing of automotive plastics.

The project had the following technical objectives:

1. Determine the technical requirements for an optical module that collects the broadband radiation from a plastic sample and couples this information into an optical signal-transmission system. The module should accommodate a “reasonable” field-of-view and depth-of-focus.
2. Determine the technical requirements for a photonic containment array. This array provides for further optical signal processing and allows concurrent monitoring of various spatial locations.
3. Determine the technical feasibility of developing a beam-shaping module which collects and condenses the broadband radiation transmitted from the containment array and presents it to a detector system.
4. Determine the technical requirements for a detector containment array which will be configured to align with the photonic containment array.
5. Determine the technical requirements for an analog amplification circuit that converts the detector response into an “adequate” voltage signal.
6. Construct a rudimentary prototype for the verification of objectives 1 through 5 and determine overall technical feasibility for development of the proposed high-speed multispectral imaging system.

Brief System Description

The multispectral imaging system can be functionally segmented into five modules. These include a collection module, an optical- signal input module incorporating a photonic containment array, an optical dispersion module, an output optical-signal module, and electro-optical conversion module.

The collection module collects radiation emanating from an irradiated sample and conditions the radiation for presentation to the optical-signal input module. The input module further conditions the optical signals through noise suppression and beam-shaping photonic containment array for redirection and presentation to an optical dispersion module. In the optical dispersion module, the conditioned and redirected optical signals collected from the irradiated sample are dispersed according to energy level and the dispersed optical signals are directed into an output optical signal module. The output optical signal module conditions the dispersed optical signals and aligns the optical signals for presentation to an electro-optical conversion module incorporating a containment detector array which converts the optical signals into electrical signals for analyses by an associated computing system.

System Evaluation

After assembling the prototype and carefully adjusting all mechanical parameters, a “white” and “black” energy spectrum was obtained. The “white” spectrum is acquired when viewing only our radiation source and provides a “perceived” energy signature for that source after transitioning the imaging system. Likewise, the “black” spectrum is acquired when the radiation source is off and provides a “background” signature. Any subsequent data must be “normalized” by subtracting the “black” spectrum, then dividing by the difference of the “white” and “black” spectrum.

Since the primary goal of the prototype was to validate the procedure of simultaneously acquiring independent data from multiple sources, quantifying the crosstalk between channels is essential to evaluating its performance. In particular, the crosstalk we considered could be categorized as electrical, thermal or optical. Electrical crosstalk between the channels is characterized by a voltage change in one channel as a result of applying a signal to another channel. By visual inspection of the various signals, electrical crosstalk seemed negligible.

Thermal crosstalk between the channels is characterized by a voltage change in one channel as a result of the thermoelectric (TE) cooling in another channel. This interaction was difficult to measure, since there seemed to be some signal variance even within a single detector/cooler pair. Perhaps this “drift” was a result of our TE cooling circuitry. However, when the detectors were embedded in an aluminum heat sink, about a one percent signal distortion due to thermal effects was observed.

Optical crosstalk between the channels is characterized by a voltage change in one channel as a result of an intensity change in another channel. To measure the crosstalk of channel *A* on *B*, the radiation entering channel *A* is “chopped” (i.e., cycled on and off) and the change in *B* indicates the distortion. Surprisingly, even after careful alignment of the various system components, the optical crosstalk seemed to range from ten to twenty percent. It is believed that much of the crosstalk is the result of surface effects and that anti-scattering measures, to be incorporated in Phase II, will significantly reduce this distortion.

The next step in system validation involved acquiring “spectra” of some automotive polymers with the prototype and comparing them to “references”. To exercise the system, polystyrene (PS), polycarbonate (PC) and polyvinyl chloride (PVC) were selected for this testing. It should be noted that the data analyzed by the system for these polymer samples were acquired simultaneously.

Many physical characteristics (e.g., surface texture, fillers, color) affect the overall shape of the “spectra”; as a result, the system data and reference signals were not expected to be identical. However, certain signatures specific to the polymers were clearly be identifiable. Using these data, the polymers were readily distinguished from one another.

Conclusions

The Phase I research enabled us to investigate a low-cost, high-speed, and effective alternative to current polymer sorting systems. In particular, the prototype has demonstrated that independent, unique signatures can be simultaneously acquired from multiple spatial locations using a multispectral device thereby establishing technical feasibility for development of an operational, near-commercial-scale prototype system in Phase II for high-volume sorting of automotive plastics recovered from automobile SR.

Research using the Phase I bench-scale prototype unit has also highlighted three areas that require more attention in Phase II before constructing an effective industrial-grade sorting system: 1) acquisition speed, 2) channel crosstalk, and 3) number of channels. The prototype system can only acquire approximately 10^3 samples per second per channel; this rate needs to be increased to around 10^4 in the final system. Crosstalk was seen to be as high as twenty percent; a decrease to less than five percent is desired. Finally, the number of simultaneous channels should be increased to at least eight in order to satisfy desired processing rates.

Publications

Phase I Final Report, *Development of High Speed Multispectral Imaging for Sorting Automotive Plastics*, U.S. Department of Energy SBIR Program, Contract DE-FG02-06ER84559, National Recovery Technologies, Inc., Nashville, TN, March 2007.

G. Microwave Assisted Gasification for Recycling Polymer Matrix Composites

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Contract No. DE-FG02-07ER84878 Small Business Innovative Research (SBIR) and Small Business Technology Transfer (STTR) Program

Objective

- Develop technology for the cost-effective recovery of high strength, full size fibers from polymer matrix composites (PMC).

Approach

- Gasify polymer compositions using microwave gasification and conventional heating.
- Evaluate gas production, liquid composition, and char percentages.
- Determine microwave susceptibility and dielectric heating characteristics of common fiber and particulate materials.
- Determine composite strength using virgin and recycled reinforcements.
- Evaluate whether microwave assisted gasification of PMCs is more energy efficient and economical than conventional pyrolysis.

Accomplishments during This Reporting Period (6/20/07-2/22/08)

Eltron Research and Development has shown proof-of-concept of microwave assisted gasification of polymer matrix composites during this Phase I project. Achievements during this reporting period are given below.

Dielectric Heating Assessment of Fiber and Particulate Compositions

- Selected fiber and particulate reinforcement compositions for testing.
- Determined optimal microwave conditions for heating each composition.
- Prepared new particulate materials with superior dielectric heating capabilities.
- Assessed heating rate and temperature achieved for each composition.

Microwave Assisted Gasification of Polymer Matrix Composites

- Designed, built and tested a laboratory scale microwave gasification reactor.
- Developed microwave thermocouple system.
- Developed experimental method to screen particulate and fiber loading and composition.
- Completed matrix of experiments which show the effect of particulate loading and composition on gas, liquid, and char formation.

Future Direction

The remaining Phase I effort will include the following:

Compare Microwave Gasification to Conventional Pyrolysis

- Design, build and test a laboratory scale pyrolysis reactor using a conventional furnace.
- Complete experiment which shows the effect of heating type (microwave vs. conventional heating) on gas, liquid, and char formation.

Microwave Assisted Gasification of Polymer Matrix Composites

- Continue experiments which show the effect of reinforcement composition, addition of air flow, microwave output power, and polymer composition on gas, liquid, and char formation.

Strength Testing of Composites with Recycled Reinforcements

- Prepare composite samples with virgin and recycled fibers by resin transfer molding.
- Characterize tensile properties according to ASTM D 638-01 for Tensile Properties of Plastics.
- Characterize interlaminar shear strength according to ASTM D2344 for short beam shear testing of high-modulus composite materials.

Feasibility Study Quality Economics for Comparison of Technologies

- Compare the economics of microwave gasification of polymer matrix composites to conventional pyrolysis.

Anticipated Phase II Objectives

- Design, build and test small pilot-scale microwave assisted gasification reactor for scale-up tests to more closely mimic a commercial system.
- Develop methods to microwave gasify composite samples and reclaim fibers from existing commercial products.
- Ascertain the effect of polymer composition, mixed polymer samples, and metal or ceramic content on gas product generation and fiber strength.
- Continue to increase the compositional stability and catalytic activity of particulate reinforcements with dielectric heating capability.
- Determine the feasibility of microwave assisted gasification for other automotive feedstocks such as tires, natural-fiber/polymer composites, and neat polymers.
- Develop a marketing and business plan for commercialization of the technology including feasibility study quality economics for comparison of technologies.

Summary

The objective of this project is to develop a Microwave Assisted Gasification (MAG) process for converting polymer matrix composites (PMC) such as fiber reinforced plastics (FRP) to viable value added products without reducing the size, strength or stiffness of the reinforcement fibers. This will include the development of new fiber or particulate reinforcement compositions that act as internal dielectric heaters when exposed to microwaves.

In this project, the MAG process will efficiently convert the organic matrix of PMCs to usable chemicals or fuels without the degradation in strength or size of the inorganic reinforcement, allowing it to be directly recycled into new composite structures. The microwaves will increase the efficiency and reaction rate of the gasification of the polymer matrix by enhancing the catalytic activity. Fiber or particulate reinforcements will be selected for microwave susceptibility to dielectric heating, so that when exposed to microwaves, the reinforcements will heat the plastic matrix from the inside.

Economically, the MAG process has many benefits to offer versus conventional pyrolysis or mechanical separation methods. Energy costs for microwave heating are comparable to conventional heating methods, but for the proposed MAG process, the costs are offset by quicker, more efficient gasification and savings by eliminating the need for separation, purification, or flotation processes. The MAG process also provides for full-size, high strength and stiffness fiber recycling.

Dielectric Heating Assessment of Fiber and Particulate Compositions

The dielectric heating susceptibility of 27 particulate and fiber compositions were tested at 50% microwave power. As shown in Table 1, six types of compounds were tested. Common particulate additives for polymer matrix composites showed low dielectric heating susceptibility.

Most commercial glass formulations showed low dielectric heating susceptibility. One formulation heated to >50°C, which shows that glass compositions could be optimized for dielectric heating susceptibility.

Metal oxide powders showed varying dielectric heating susceptibility (see Figure 1). The extent of heating depended on metal selection and metal oxidation state. One metal oxide which is a combination of +2 and +3 oxidation states showed a decrease in dielectric heating susceptibility as the M^{+2} oxidized to M^{+3}.

All powders which are well-known dielectric heaters showed heating to >100°C within 5 minutes at 50% microwave power (see Figure 2). These tests illustrated the effect of powder particle size and sample size.

If enough sample was provided, composite reinforcement fibers showed good dielectric heating within 1-2 minutes (see Figure 3).

The best dielectric heaters were proprietary oxygen-storage catalysts (see Figure 4). Most heated to >500°C within 3 minutes at 50% microwave power.

Table 1. Summary of materials tested for dielectric heating.

Type of Particulate or Fiber	Number of Sample Compositions Tested
Commercially available oxides	6
Common particulate reinforcements for composites	2
Well-known dielectric heaters	4
Fibers	3
Proprietary oxygen-storage catalysts	5
Glass compositions	7

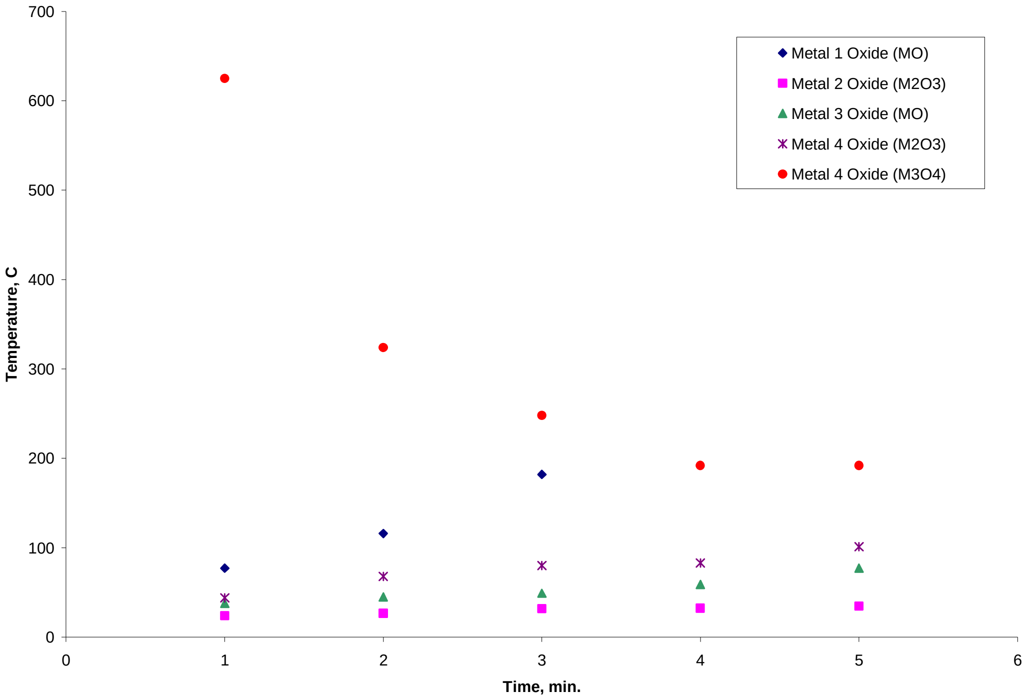

Figure 1. Dielectric heating susceptibility of 5 metal oxide powders.

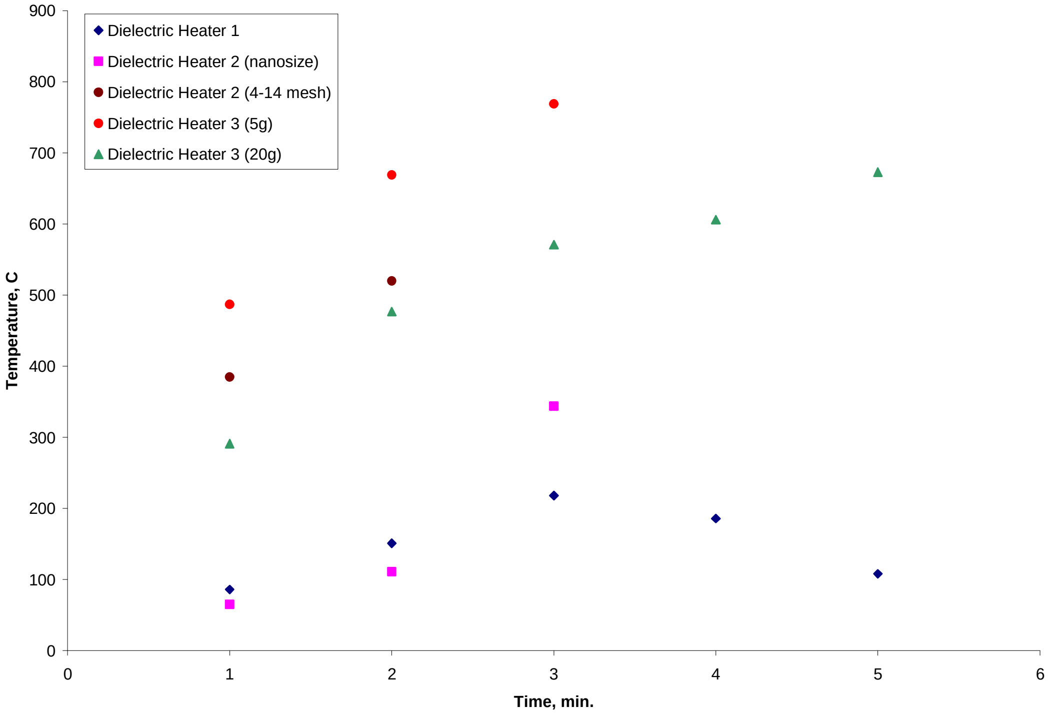

Figure 2. Dielectric heating susceptibility of well-known dielectric heaters.

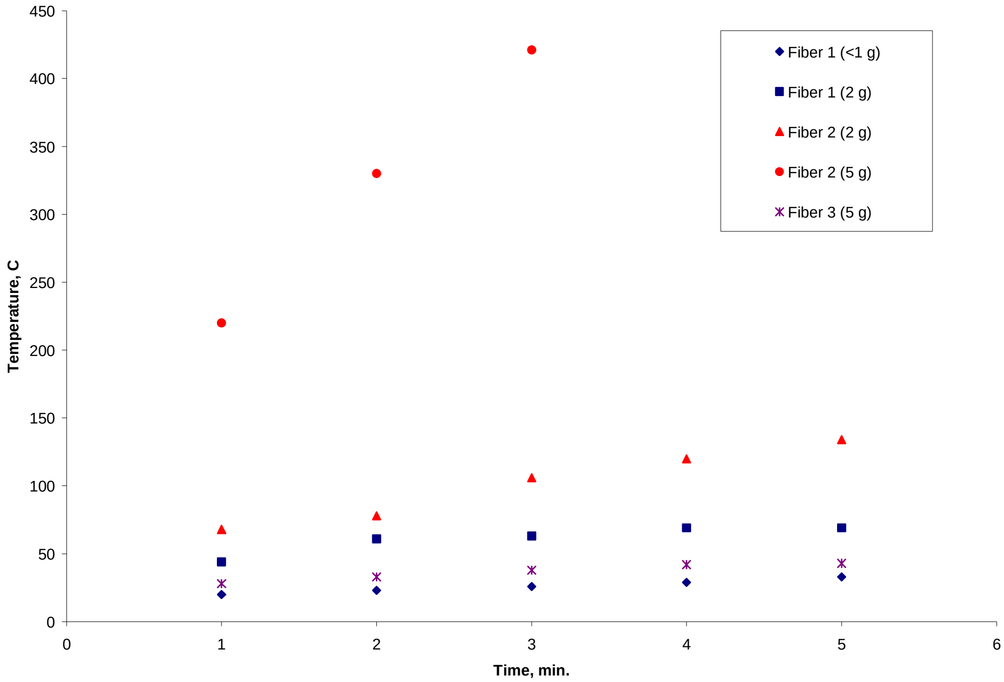

Figure 3. Dielectric heating susceptibility of reinforcement fiber compositions.

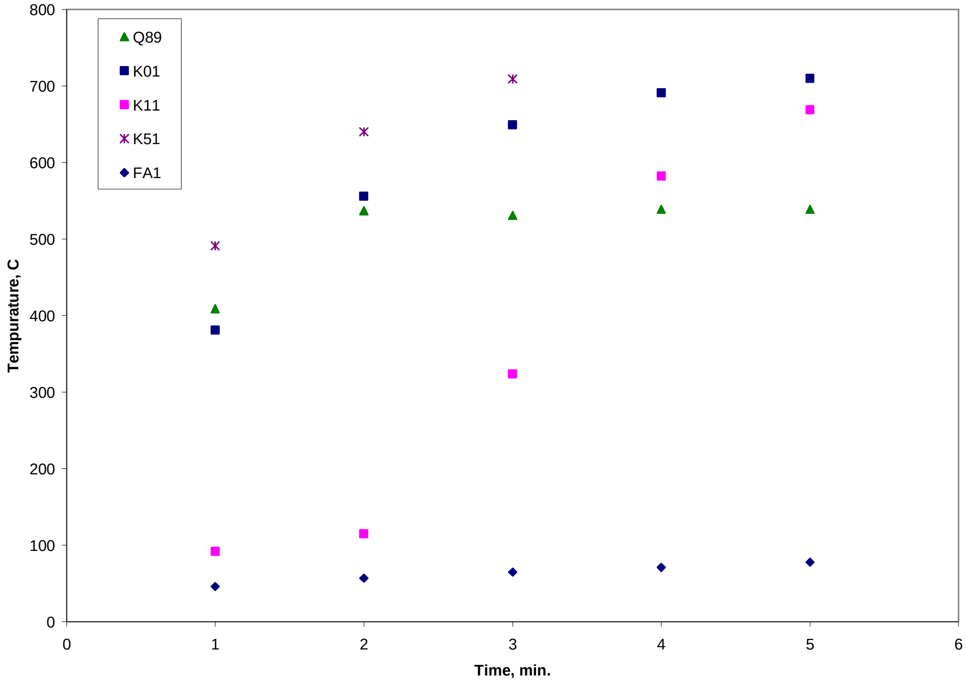

Figure 4. Dielectric heating susceptibility of proprietary oxygen-storage catalysts.

Microwave Assisted Gasification of Polymer Matrix Composites

Many research groups are working on pyrolysis of the organic matrix which is decomposing the polymer in the absence of oxygen. Heating a polymer matrix to 450–500°C can be sufficient to break down many different polymers into a wide range of products including CO, H_2, and hydrocarbons including methane and ethane. However, pyrolysis at 500°C has many waste products that are difficult to separate from the fibers. Raising the pyrolysis temperature can minimize the creation of byproducts but at higher temperatures, fibers become brittle and lose strength.

Microwave Assisted Gasification can be used to optimize CO and H_2 generation over other gaseous products such as CH_4 or CO_2, increase gaseous versus liquid product generation, and reduce operating temperatures and waste products to generate full-size, high strength fibers with clean surfaces.

At 275–325°C, Microwave Assisted Gasification of epoxy samples showed mostly CH_4 gas generation (see Figure 5). The addition of 1% oxygen-storage catalyst increases the H_2 generation (see Figure 6). Fiber-reinforced composite samples showed the best gas product composition (see Figure 7) and after gasification of the polymer matrix, fibers were undamaged with clean surfaces and no byproducts (see Figure 8).

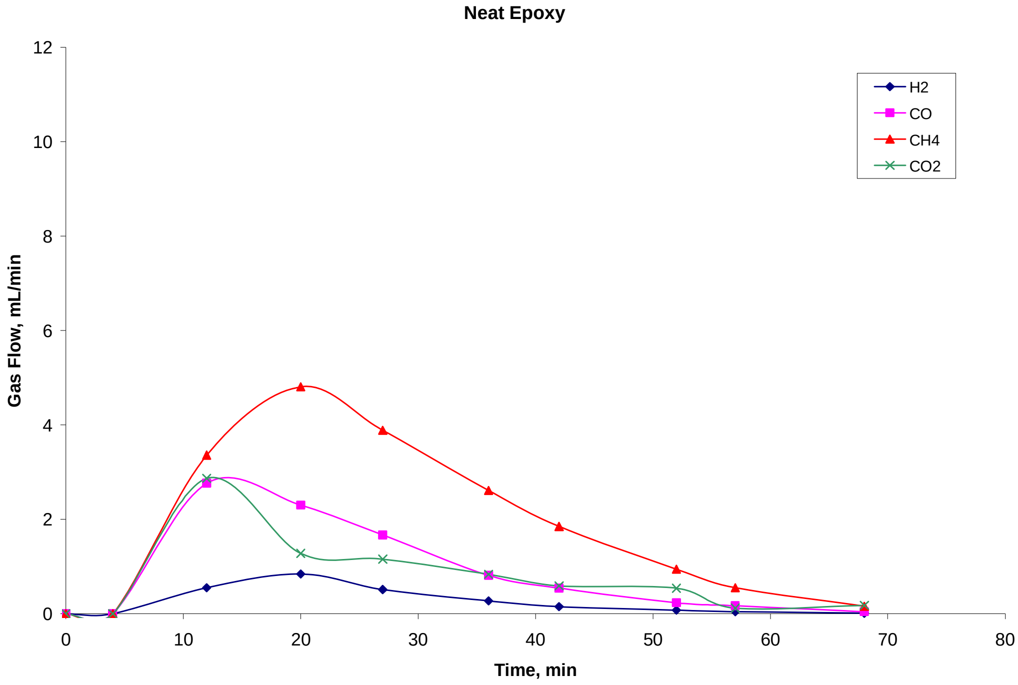

Figure 5. Microwave Assisted Gasification of epoxy sample.

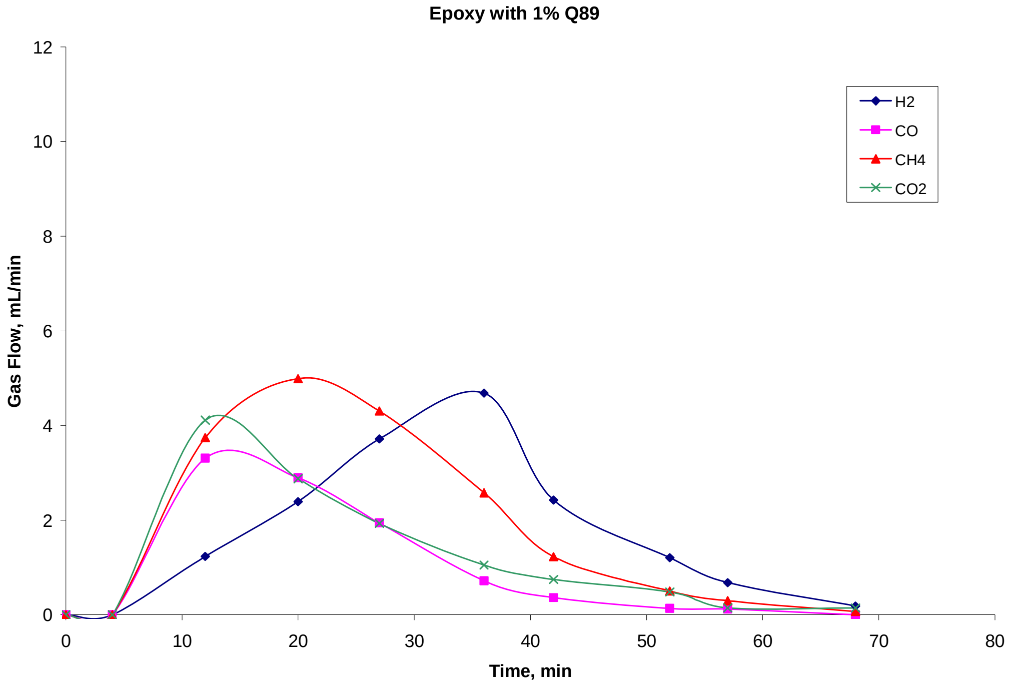

Figure 6. Microwave Assisted Gasification of epoxy sample with 1% Q89 catalyst.

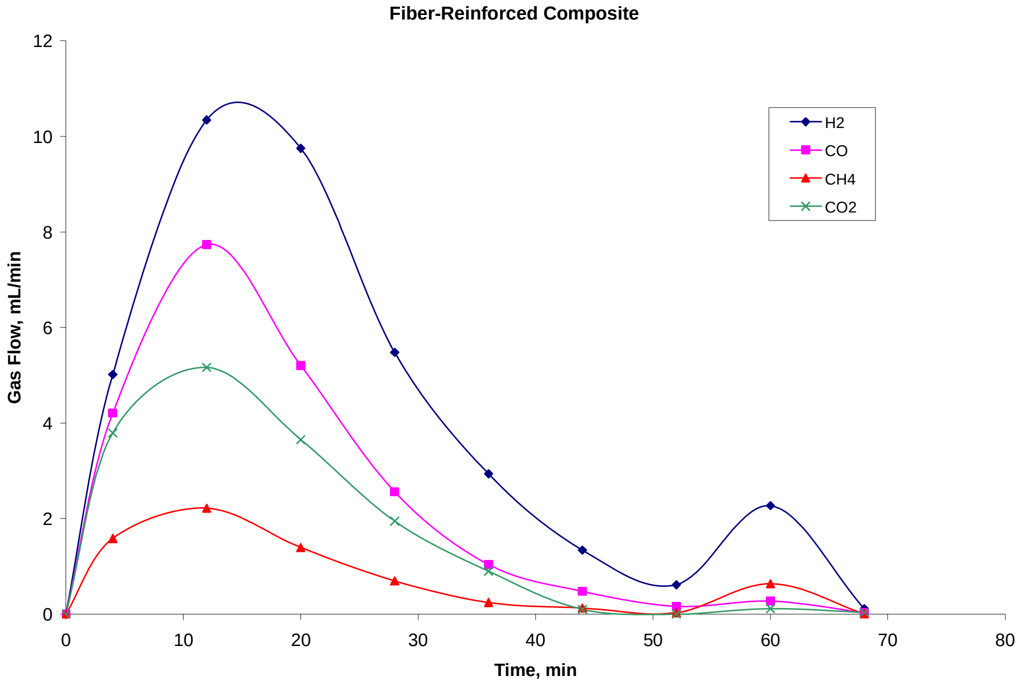

Figure 7. Microwave Assisted Gasification of fiber-reinforced composite sample.

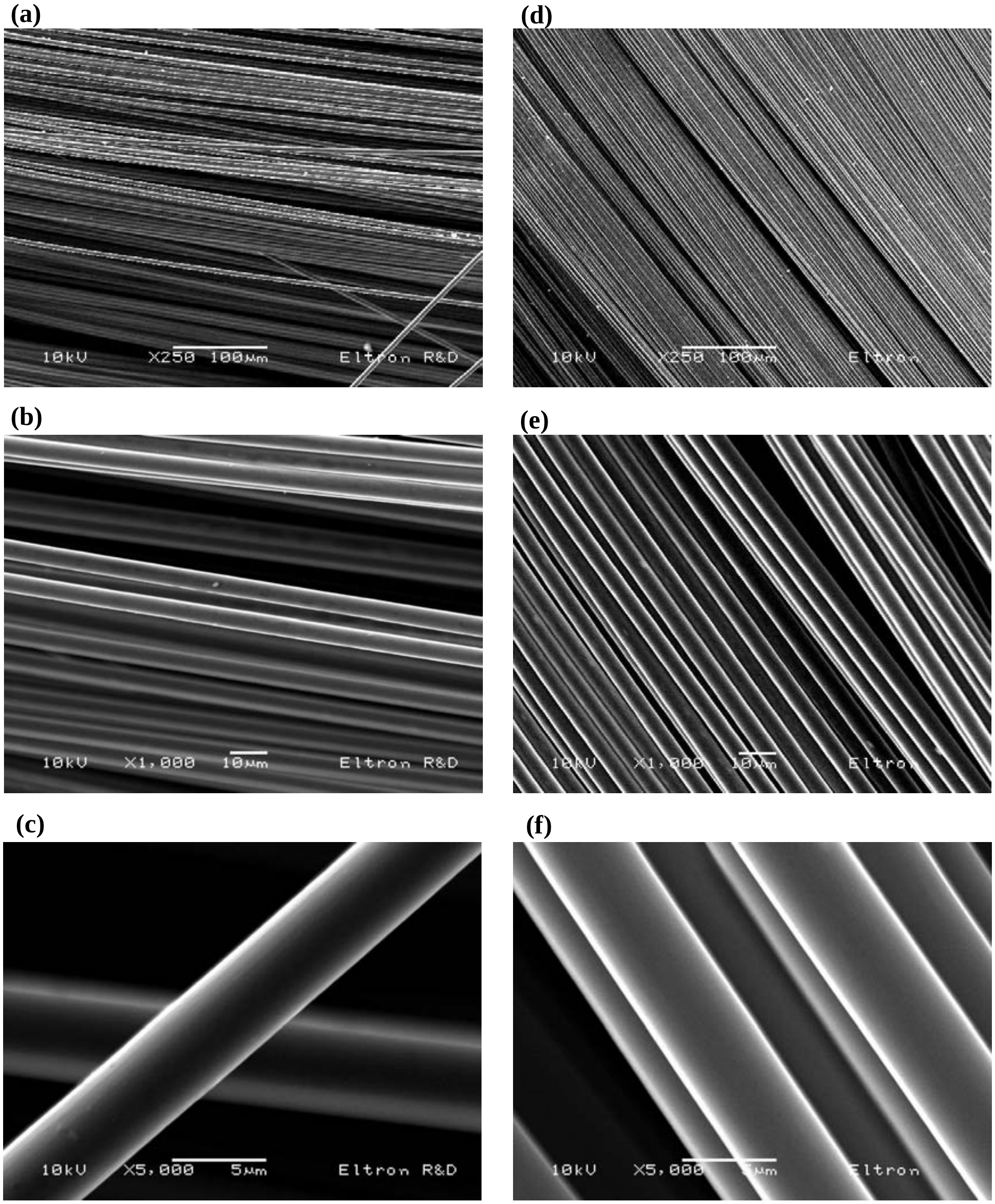

Figure 8. SEM photos of virgin fibers (a, b, c) compared to fibers after Microwave Assisted Gasification of the polymer matrix (d, e, f).