

11. RECYCLING

A. Recycling Assessments and Planning

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Participants:

This project is conducted as part of the cooperative research and development agreement among Argonne National Laboratory (ANL); United States Council for Automotive Research's Vehicle Recycling Partnership, LLC; and the American Chemistry Council Plastics Division

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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 (10/1/07–9/30/08)

- Conducted progress review meetings with CRADA team members.
- The Engineering Project Oversight Committee (EPOC) of the VRP also held meetings twice a month and an ANL representative attended as necessary to discuss CRADA issues.
- Made presentation to the VRP management committee which holds monthly meetings and to the Environmental Technology Leadership Council of USCAR to update them on the CRADA accomplishments.
- Presented progress at the 2008 Office of Vehicle Technologies Annual Merit Review meeting (Feb 27, 2008).
- Held meetings with the FreedomCAR Materials Technical Team.
- Conducted a workshop (September 24–25, 2008) to develop a roadmap for recycling lightweighting metals (aluminum, magnesium and titanium) and to identify R&D needs for recycling these metals.
- Updated the CRADA website: http://www.es.anl.gov/Energy_systems/CRADA_Team_Link/Index.html.
- Continued gap analysis with CRADA team.
- Reached agreement with a shredder to build a 20-ton-per-hour validation plant to confirm process yields, process economics, and to confirm qualities of the recovered polymers.
- Modified the Annual Operating Plan in order to assist the shredder who is building a 20-ton-hr validation plant based on the ANL recycling technology.
- Prepared the statement of work for a new 5-year extension of the CRADA.
- Identified new lightweighting materials being considered for vehicles of the future.
- Prepared and/or presented technical papers on recycling automotive materials. One of the papers was published in the November 2007 issue of the *Journal of Metals* and has been selected to receive the 2009 Technology Award of the Extraction and Processing Division of The Minerals, Metals & Materials Society (TMS).
- Continued liaison with the Institute of Scrap Recycling Industries (ISRI), the Automotive Recycling Association (ARA) and visited the facilities of two automotive dismantlers and a shredder.
- Reviewed Small Business Innovation Research proposals for DOE.

Prior Accomplishments

FY 2007

- Conducted bimonthly progress reviews with CRADA Team.
- Identified new automotive lightweighting materials that are being considered for vehicles of the future.
- Continued gap analysis with CRADA team.
- Prepared several papers outlining the industry/government collaboration at national and international conferences.
- The CRADA team received the (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.
- Continued liaison with ISRI, ARA, and individual shredders.
- The ACC-PD Operating Committee visited the ANL pilot plant.

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 United States (U.S.) End-of-Life Vehicles (ELVs) CRADA Team Website. It includes an overview of the CRADA team activities, downloadable CRADA team brochures, a bibliography of recycle literature, and 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 ISRI and 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 ELVs of the Future.
- Continued liaison with the 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 and the American Plastics Council (APC. Now the ACC-PD) and ANL as partners; effort under the CRADA was initiated in August 2003.

Future Direction

- Pursue extension of the present CRADA for 5 more years.
- Continue development and management of the R&D plan with the CRADA partners.
- Continue ongoing efforts toward the milestones and objectives of the CRADA.
- Conduct scheduled progress reviews and continue gap analysis with the CRADA partners.
- Maintain and update the U.S. ELVs CRADA team website.
- 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.

Introduction

The objective of this project is to establish priorities and develop cost-effective recycling technologies and strategies in support of DOE's Energy Efficiency and Renewable Energy (EERE) Vehicle Technologies 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 nonferrous 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 lightweighting

materials (e.g., ultra-high strength 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, 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, 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.

A one-and-a-half day seminar was conducted on September 24 and 25, 2008 to develop a roadmap and identify R&D needs for recycling lightweighting metals (Al, Mg, and Ti). A roadmap report with recommendations will be issued during the next reporting period.

The Five-Year Research and Development Plan

On the basis of the 2001 roadmap and continuing discussions with key stakeholders, a 5-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.

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. 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; 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, and 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.
- 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 (Recycling Assessments and Planning) 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. 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 a 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.

We were contacted by a U. S. shredder who was willing to validate ANL's process for recycling materials from shredder residue by building a validation plant at one of his yards. During this reporting period, we reached agreement with the shredder to build a 20-ton-per-hour validation

plant to confirm process yields, process economics, and to confirm qualities of the recovered polymers. In order to support the shredder's effort, we prepared a modified operating plan and the modified plan was approved.

The CRADA team members also held their regular review meetings to review progress and identify technology gaps. Members from ANL and the VRP attended two Materials Technical Team meetings and discussed the validation plant plans. We also attended the 2008 Office of Vehicle Technologies Annual Merit Review meeting in Bethesda, Maryland, in February of 2008 and presented our progress to the review panel.

The CRADA team received the SPE Environmental Division's GPEC, "Enabling Technologies in Processes & Procedures," 2007 Award. The award was 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. The team also prepared and/or presented a number of papers on recycling automotive materials. One of the papers was published in the November 2007 issue of the *Journal of Metals* and has been selected to receive the 2009 Technology Award of the Extraction and Processing Division of TMS.

Many shredders, polymer recyclers, and the ACC-PD's operating committee visited ANL's automotive recycling pilot plant and discussed plastics recycling activities. Representatives of ANL, VRP, and ACC-PD also held discussions with ARA and visited two dismantlers and a shredder and discussed present recycling activities and the industry's needs for expanding these activities.

The VRP established a Communications/ Outreach project to create more visibility and awareness among the public, industry, and legislative audiences regarding the work the VRP performs. This outreach occurred via participation at conferences, seminars, and auto shows and featured trade show booths, brochures, videos, mouse pads, and tote bags. It is believed that communicating with the public, industry, and

legislative audiences will bring in new collaborators, project partners, and even more leveraged funding. The project is preparing a video reflecting the recycling infrastructure in the United States. The video provides a quick way to explain the vehicle recycling infrastructure and the role VRP initiatives and projects play in the infrastructure. The VRP initiated another project the objective of which is to provide guidance to optimize design of future vehicles by focusing on improved recyclability, resource optimization, and reduction of the amount of shredder residue by recovering reusable materials.

A website was launched in March of 2006 to provide for better communication and networking with stakeholders and other research teams: http://www.es.anl.gov/Energy_systems/CRADA_Team_Link/Index.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. During this reporting period, the website was updated with more information including access to additional information sources.

The CRADA team held a media event at ANL 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 “Market Driven Technology Development for Sustainable End-of-Life Vehicle Recycling: A Perspective from the United States,” 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, the ARA and 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. Another review of the projects and ongoing efforts of the CRADA team was held on February 27, 2008. We also conducted a workshop on September 24 and 25, 2008 to develop a roadmap for recycling lightweighting metals (Al, Mg, and Ti).

Publications

1. Automotive Recycling in the United States: Energy Conservation and Environmental Benefits, J. A. Pomykala Jr., B. J. Jody, J. S. Spangenberg, and E. J. Daniels, *The Journal of Metals*, Vol. 59, No. 11, Nov. 2007, pp. 41–45.
2. *The R&D of the FreedomCAR Materials Program*, J. A. Carpenter 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.
3. *Market Driven Technology Development for Sustainable End-of-Life Vehicle Recycling: A Perspective from the United States*, E. J. Daniels, B. J. Jody, J. A. Pomykala Jr., and J. S. Spangenberg, presented at the 6th International Automobile Recycling Congress, Amsterdam, Netherlands, March 15–17, 2006.
4. *Industry and Government Collaboration to Facilitate Sustainable End-of-Life Vehicle Recycling*, E. J. Daniels, 2005 ASME International Mechanical Engineering Congress & Exposition, BRTD-4: Sustainability Applications in Product Design and Manufacture, Orlando, Florida, November 5–11, 2005

5. *Market Driven Automotive Recycling in North America*, C. Duranceau, presented at the Institute of Scrap Recycling Industries Shredder Meeting, Dallas, TX (Oct. 30, 2004).
6. *Sustainable End-of-Life Vehicle Recycling: R&D Collaboration between Industry and the U.S. DOE*, E. J. Daniels, J. A. Carpenter Jr., C. Duranceau, M. Fisher, C. Wheeler, and G. Winslow, JOM, The Mineral, Metals & Materials Society, vol 56, no 8, pp. 28–32 (Aug. 2004).
7. *Market Driven Automotive Recycling in North America*, C. Duranceau, USCAR, J. Carpenter, U.S. DOE, M. Fisher, American Plastics Council, keynote at the 2004 International Car Recycling Workshop, May 19, 2004, Washington D.C.
8. *Automotive Materials Recycling: A Status Report of U.S. DOE and Industry Collaboration*, E. J. Daniels, Ecomaterials and Ecoprocesses, Proc. of the International Symposium on Ecomaterials and Ecoprocesses, August 24–27, 2003, Vancouver, BC, Canada, pp. 389–402.
9. *Effects of Transportation on the Ecosystem*, J. A. Carpenter Jr., Ecomaterials and Ecoprocesses, Proc. of the International Symposium on Ecomaterials and Ecoprocesses, August 24–27, 2003, Vancouver, BC, Canada, pp. 13–22.
10. *Automotive Technology: Looking Forward*, R. Sullivan, 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.
11. *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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Contractor: Argonne National Laboratory (ANL)

Contract No.: DE-AC02-06CH11357

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 associated with various end-of-life recycling technologies.
- Conduct reference case end-of-life recyclability studies.

Accomplishments During This Reporting Period (10/1/07–9/30/08)

- Work on this project was deferred in the later part of fiscal year (FY) 2008 in favor of supporting the shredder who is building the 20 ton/hr validation plant. Accomplishments are:
 - Updated the data base of recycle technologies.
 - Compiled data for updating the document reviewing technologies for recycling shredder residue.
 - Complete the life cycle analysis model for the automotive recycling infrastructure

Prior Accomplishments

FY 2007

- Updated the data base 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.

FY 2006

- Completed compilation of recycle bibliography, structured as a pull-down PDF file, posted on US End-of-Life (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 future years will be on:
 - Updating the data base of recycle technologies.
 - Updating the document reviewing technologies and the state-of-the-art for recycling automotive materials with emphasis on lightweighting metals [Al, magnesium (Mg), and titanium (Ti)].
 - Conduct life-cycle and sustainability studies of lightweighting automotive materials.

Introduction

The objectives of this project are to (1) benchmark the automotive materials recycling industry and (2) 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 research and development (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 the 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 and 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. Society of Plastics Engineers:
 - (International) Automobile Recycling Congress (ARC) from 1998 to 2000
 - Global Plastics Environmental Conference (GPEC) from 2002–2007
4. Other conference proceedings:
 - International Automobile Recycling Congress from 2001 to 2006
 - The Minerals, Metals, and Materials Society (TMS) Fourth International Symposium of Recycling of Metals and Engineered Materials, 2000.
 - Ecomaterials and Ecoprocesses, The Conference of Metallurgists, COM 2003

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

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.

Recycling Technologies: State-of-the-Art

The final document has been published and has been posted on the U.S. 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 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 such as mercury and polybrominated diphenyl ethers (PBDEs) can impede materials recycling. Nickel, lithium, and cobalt, which will be used in electric batteries for hybrid and plug-in vehicles, could also impact materials recycling and will have to be recycled themselves to prevent sharp increases in their prices.

Life-Cycle Studies

The objective is to use life-cycle analysis to assess the environmental impacts of various separation 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 Bilanzierung) software, was contracted to perform these analyses. Four analyses have been completed for: (1) Salyp NV's mechanical separation process, (2) CWT's thermal conversion process, (3) ANL's mechanical and froth-flotation process, and (4) automotive recycling infrastructure analysis.

The CRADA team is planning to conduct a study where late-model vehicles will be shredded in a new shredder that is being built at this time. Older vehicles will then be shredded followed by shredding a normal mix of materials. This study is expected to take place on October 4th, 2008 and data analysis will be completed in February, 2009. Data generated from this study will be used to update the life-cycle study of current automotive recycling operations.

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 analysis, including all energy, water, and material inputs, plus data on emissions to air and water, wastes, and products produced. The four 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 CO₂ 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 were 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 CO₂ 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 CO₂ 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 CO₂ 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 the 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 analyses concluded that 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.

A life-cycle analysis model for the automotive recycling infrastructure was also completed. This model will facilitate the analysis of various automotive recycling technologies and compare their performance.

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.

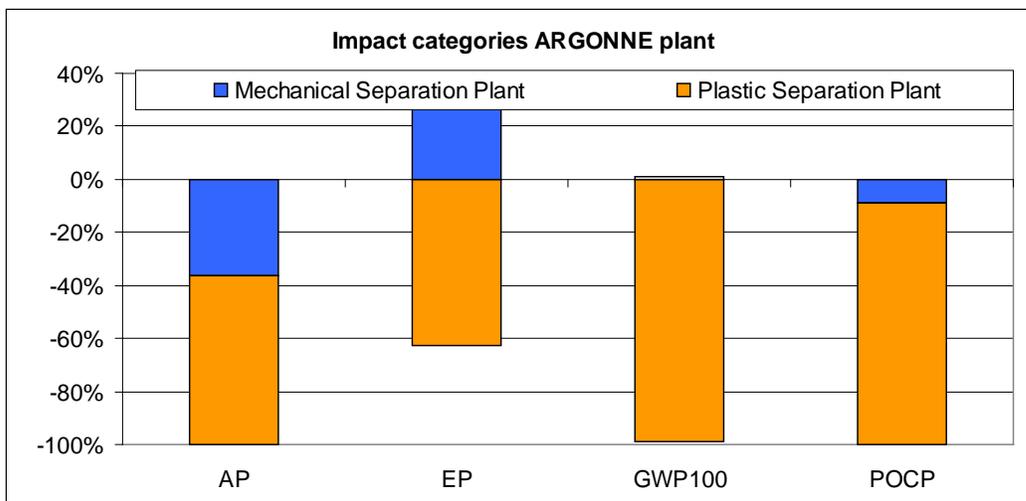


Figure 1. Impact Categories of the ANL Plant. (AP is acidification potential, EP is eutrophication (depletion of oxygen in water) potential, GWP is global-warming potential and POCP is photochemical ozone creation potential). [Y axis indicates increase (+) or decrease (-) in the impact of the different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

^aComparison is done here only with the froth-flotation process because both ANL’s froth-flotation process and CWT’s process require mechanical separation of the inorganic materials.

The materials breakdown is summarized in Table 4. In comparison, the materials composition of a production Ford Taurus is summarized in Table 5.

Table 4. 2004 Toyota Prius materials breakdown.

Materials	Mass (lb)	%
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)	%
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

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; 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. 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 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.

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

Calculation Method	As Produced (%)	Aluminum 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

^aIf 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.

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 lightweighting automotive materials if recycling mandates are to be met and to ensure that lightweighting materials are not excluded because of the inability to recycle them.

It is interesting to point out that even if the lightweighting metals that replace steel and iron are recycled at the same rate as the present rate of recycling steel and iron, the overall recyclability rate of the obsolete vehicle will decrease because the relative weight of the metals in the vehicle will decrease. Therefore, in order to maintain high vehicle recycling rates it is important that non-metallic materials that are presently not recycled, such as polymers, be recycled.

Publications

1. *A Life Cycle Look at Making Oil From End-of-Life Vehicles*, C. S. Wheeler, N. L. Simon, M. Binder, G. R. Winslow, and C. M. Duranceau, 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*, M. Binder, N. L. Simon, C. M. Duranceau, C. S. Wheeler, and G. R. Winslow, 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*, C. S. Wheeler, N. L. Simon, C. M. Duranceau, G. R. Winslow, and M. Binder, SAE Paper 2005-01-0847.
4. *United States National Life Cycle Inventory Database Project, A Status Report*, J. L. Sullivan, C. S. Wheeler, and N. L. Simon, SAE Paper 2005-01-0852.

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

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Contractor: Argonne National Laboratory (ANL)

Contract No.: DE-AC02-06CH11357

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 (10/1/07–9/30/08)

- Tests were conducted on ANL's two-stage process using a 5 lb reactor.
- Conducted swipe tests on auto parts made with polyolefins recovered from shredder residue.
- ECO₂ conducted tests using their proprietary process.
- Midland Compounding/Energy Anew conducted vacuum de-volatilization tests in a twin-screw extruder to explore removal of PCBs.
- Tests were conducted on a proprietary process developed by an independent organization.

Prior Accomplishments

FY 2007

- Additional bench-scale testing of ANL's two-stage cleaning process was conducted. The testing consistently produced plastics from the polypropylene (PP)/polyethylene (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/h). Quotes for the major system components were obtained.
- Hosted and participated in forums and seminars on SOCs including perfluorooctanoic acid (PFOA)/perfluorooctanesulfonates (PFOS), brominated fire retardants, and emerging lead- and mercury-free technologies.

FY 2006

- ANL developed a two-stage cleaning process which, in bench-scale tests, consistently produced plastics from the PP/PE product with <2 ppm PCBs.
- Completed tests in the commercial solvent washing equipment using proprietary, solvent-based solutions and in CO₂.
- Cooperated with the Bromine Science and Environmental Forum (BSEF) in preparing the BSEF brochure entitled "Deca-BDE Flame Retardant."

FY 2005

- 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.

FY 2004

- 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

- Determine the scalability and economics of ANL's two-stage process.
- Develop a conceptual design and perform cost analysis of the ANL process.
- Incorporate the process into the overall process design for recovering materials from shredder residue.
- Continue testing at ECO₂ using their proprietary process.

- ANL's two-stage process has successfully produced recovered plastics, at least PP/PE materials, with less than 2 ppm PCBs in multiple tests. Operational problems including sticking of the plastics to the walls of the reactor have been encountered in the larger-scale tests. Granulation of the desorbed plastics is also required. Further evaluation is necessary to investigate the scalability of the process and to establish its final flow diagram and economics.

Introduction

The objective of this project is to develop techniques and/or technology to identify, quantify, 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 impact the overall costs of recovering recyclable materials. In some cases, their presence at parts-per-million levels, 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 regionally. 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 technologies 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 used in automobiles could also become SOCs in the near future. Examples include some of the brominated fire retardants.

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.

The criteria for the 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.

Development and Testing of a Two-Stage Process at ANL

The work done so far on cleaning of polymers recovered from shredder residue suggests that different washing methods and solutions appear to be able 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. In addition to added cost, prolonged washing, particularly in organic solvents, is further complicated by the absorption of the solvent by the plastics which may alter the properties and the value of the plastics.

This behavior of PCBs suggests that the PCBs on the plastics are removed by more than one mechanism. Some of the PCBs are in the oils and dirt that are on the plastics and some are adsorbed on the plastics and do not desorb easily during washing. This hypothesis was tested in the lab. Washing tests were conducted 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 with or without 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 first at the bench scale at ANL and then in a 5 lb reactor that processed 5 lb of plastics per test. It has repeatedly reduced the PCBs concentration in PP/PE samples to less than 2 ppm. For example, at sufficient residence times and temperature conditions that do not cause the plastics to oxidize, the concentration of PCBs on the recovered polyolefins was reduced from about 32 ppm to as low as 0.58 ppm. A preliminary design for an apparatus to test this process at a larger scale (15–25 lb/h) was developed. Commercially-available equipment that could be adopted with modification to this process was identified. Quotes for some of the system components have been obtained. The design was changed after discussions with ANL's safety committee. It was then decided to run larger batch tests (5 lb per test). The apparatus, Figure 1, was designed and built and five tests were conducted. After completing the running of the first test, it was discovered that some of the polymers dissolved/melted and, when the solvent was removed and the reactor cooled, the polymers stuck to the reactor walls. Samples of the processed polymers were sent for PCBs analysis and for properties analysis. It appears that this occurred due to localized heating in parts of the reactor.

This problem was resolved by modifying the experimental procedure including having better control of the heating system as well as better circulation of the solvent through the plastics. Four more tests were conducted without any of the material sticking to the reactor wall. The washed-and-rinsed samples were then transferred to a plastics drying oven at an elevated temperature to



Figure 1. Experimental apparatus for testing the ANL two-stage process.

desorb the residual PCBs from the plastics. Samples of the plastics after washing and rinsing as well as after desorption were sent for analysis. Samples were also sent for evaluating the mechanical properties of the cleaned plastics in order to determine if the cleaning process caused any degradation of the properties of the plastics. Results are pending.

Swipe tests were conducted on two auto parts made with recycled PP/PE recovered from shredder residue using acetone. The objective was to see if PCBs can leach from such parts. The procedure for the swipe tests involved first marking the area on which the test is to be performed and determining its surface area. The surface area was 1 ¾ in. × 2 ¾ in. on one object and 2 in. × 3 in. on the other object. The surface areas were then cleaned with soap and water and allowed to air-dry completely. A hexane wipe cloth was then removed from one of the jars that was provided and was used to generously wipe the denoted surface area. The hexane wipe cloth was then placed back in a jar and the jar was closed. The hexane wipe cloths were then sent for analysis. Analysis of the acetone-containing swipe cloth showed no detectable PCBs on the wipe cloths.

Testing of a New Proprietary Process

An independent organization tested a proprietary process that they developed for cleaning the plastics. Testing of the process was conducted and processed samples were analyzed. The results

were unsatisfactory and no further testing is planned.

Testing of a Catalyzed Ozonation/Ultra Violet (O₃/UV) Process

Preliminary testing of a catalyzed O₃/UV process was conducted to determine if the PCBs can be selectively degraded. The initial results were not encouraging.

Testing of a Biodegradable Solvent Process

Tests using a biodegradable solvent were conducted to determine if this solvent can remove the PCBs. It was successful in reducing the concentration to about 10 ppm but further reduction to below 2 ppm did not appear likely. Testing using this solvent was discontinued because its effectiveness degrades when water is present.

Testing of a Devolatilization Process in a Vacuum Extruder

Midland Compounding/Energy Anew conducted a series of vacuum devolatilization tests on polyolefin samples recovered from shredder residue using a 30-mm twin-screw vacuum extruder. An experimental design was developed to test the impact of three variables on the PCBs removal process: screw speed (high, 300 revolutions per minute (rpm) and low, 100 rpm), feed rate (high, 20 lb/h and low, 10 lb/h), and temperature (high, 325°C and low, 250°C). A mid-point at 200 rpm, 15 lb/h, and 287°C with and without vacuum was also tested. The apparatus is shown in Figure 2. The results are summarized in Table 1. The results indicate that devolatilization alone was not sufficient to remove PCBs from the polyolefin blend. The IZOD property of the treated polymers did not change.

Additional tests were conducted at Midland Compounding where high-pressure water or water/glycol/NaOH were injected to help dechlorinate and steam strip the polymer after the samples were first homogenized by extrusion and post-blending. The extruder was sectioned into three zones: high-pressure liquid injection, atmospheric vent, and then a vacuum vent. The



Figure 2. Extruder with die and vacuum knockout pot used in the devolatilization testing.

Table 1. PCBs and Impact Test Results from Vacuum Devolatilization Testing

Sample No.	Temp. °C	Screw RPM	Feed Rate lb/h	PCB 1242 ppm
1	325	300	20	18.2
2	325	300	10	13.6
3	325	100	20	16.9
4	325	100	10	16.2
5	287	200	15	12.4
6 ^a	287	200	15	12.6
7	250	300	20	17.9
8	250	300	10	19.7
9	250	100	20	17.7
10	250	100	10	15.4
11 ^b	287	200	15	21.4
12 ^c	-	-	-	22.2

^aNo vacuum.

^bWater at feed.

^cRaw feed.

samples were then run through the extruder under different conditions. Samples were sent for analysis and for property evaluation. The results are pending.

Testing at ECO₂ Plastics

A contract was also awarded to ECO₂ to conduct tests using their proprietary process. Phases 1–3 of testing have been completed and further testing is planned.

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 CO₂ only. The washing failed to remove the PCBs. itec Environmental Group (name changed to ECO₂) reduced PCB levels in the plastics from 11 ppm to 2.8 ppm via solvent washing; no CO₂ 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 CO₂. 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 TPI and tested a specially-designed machine that has a design capacity of about 300 lb/h 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 2, and the spiked samples were

Table 2. Effect of phthalates on PCBs analysis

Weight-Percent of Phthalates added	PCBs Concentration (ppm) by GC-ECD	PCBs Concentrations (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

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.

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.

Two of the labs identified Aroclor 1242 as the only PCB present. The third found 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 3. 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. Environmental Protection Agency (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 4). 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 h, and another three 120-g samples were extracted with hexane for 24 h. Similarly, three 120-g samples were extracted with toluene for 8 h, and another three 120-g samples were extracted with toluene for 24 h. All extractions were carried

Table 3. PCBs analysis using GC-ECD and GC/MS [extraction using hexane at 2,000 pounds per square inch absolute and 100°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 4. 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

out while maintaining the siphoning time at 8–10-min 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.

Determination of Extraction Time

Three additional 120-g samples were extracted with hexane for 4 h 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 h 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-g samples extracted for 24 h discussed above, six 60-g samples and six 30-g samples were processed and sampled in the same manner as before (24-h extraction time and same siphoning intervals) by using hexane. The results indicate that a 30-g sample size 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 h and two of the 120-g samples that were extracted with hexane for 8 h were also quantified by using the European method. The results were essentially identical within analytical errors (Table 5).

Table 5. Comparison of the U.S. EPA and the European quantification methods

Extract Time (h)	PCBs (ppm)	
	EPA method	European method
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

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.

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, nickel, and emerging lead- and mercury-free technologies.

Publications

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

D. Post-Shred Materials Recovery Technology Development

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Changing World Technologies is cost-sharing on the evaluation of its thermal depolymerization process. The Polyurethanes Recycle and Recovery Council is also participating and cost-sharing on the evaluation of the Troy Polymers, Inc. process for converting polyurethane foam to polyols.

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Contractor: Argonne National Laboratory (ANL)

Contract No.: DE-AC02-06CH11357

Objective

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

Approach

- Characterize shredder residue 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 shredder residue to recover materials for market evaluation and to provide "control" samples for testing of alternative technologies.
- 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 (10/1/07–9/30/08)

Validation Plant Activities

- Agreement has been reached with a shredder to build a 20-ton-per-hour validation plant to confirm process yields, process economics, and to confirm qualities of the recovered polymers

Controlled Auto-Shredding Project

- An agreement has been reached with a company that recently completed building a new shredding facility to separately shred into four categories of vehicles. The four categories are: (1) late model domestic—2002–2007 model-year vehicles from Chrysler, Ford and General Motors, (2) late-model transplants—2002–2007 model-year vehicles from foreign companies which are manufactured in the United States, (3) pre-2000 normal end-of-life vehicles (ELVs) which are typical of what is shredded currently, and (4) imported federalized vehicles that are not designed for U.S. end users, but retrofitted to qualify for sale in the U.S. The resulting shredder residues will be processed to determine if differences in the residues exist. Changes in the levels of substances of concern (SOCs) such as polychlorinated biphenyls (PCBs) will be evaluated. Preparation for the test including sourcing of the vehicles to be shredded, arrangement for conducting PCB testing of the shredded material and arrangements for the shipping of the shredder residue to ANL and to Energy Anew has been completed. Shredding of the vehicles which was originally scheduled to take place in July, 2008 took place October 4–5, 2008.

Mechanical Separation of Shredder Residue

- Completed processing of the last 20,000 pounds of shredder residue, out of a 30,000 pound batch, received from the shredder who is building a plant to validate the ANL technology.

Shredding of Auto Parts Containing Nano Materials

- USCAR, ANL, and the National Institute for Occupational Health and Safety (NIOSH) developed an experimental design. A “Request for Proposals” was prepared and released by USCAR for a third party to conduct analysis of nano particles in the dust generated during the shredding of the auto parts. Professor Peter C. Raynor, from the University of Minnesota, School of Public Health, was selected to do the analysis. Contract negotiations between USCAR and the university are underway.

Froth-Flotation Separation of the Plastics

- ANL designed, built, and tested modifications to the flotation process which reduced waste water production by over 50% by minimizing the need for salts and dewatering techniques.

Recycling of Hybrid-Vehicle Batteries

- OnTo Technology, sponsored by the VRP, conducted tests on recycling two battery chemistries—nickel-metal hydride (NiMH) and lithium ion—using small batteries. The process starts by discharging and dismantling of the battery followed by CO₂ extraction of the electrolyte. The battery is then shredded at room temperature and the metals are separated. The remaining material is then pulverized and the plastics are separated from the pulverized material. The powder containing the Li is then recovered. Copper and cobalt are then recovered from the residual material by an electrowinning process. Early results show great promise. Recovered materials have been analyzed and tested in new batteries with good results. The method has the potential to cost-effectively remove more materials of value including the lithium and electrolyte fractions than current technologies. OnTo is continuing their research to look at additional battery chemistries.

Energy Anew Process for Recycling Shredder Residue Fines

- Energy Anew has constructed a mobile cyclonic system to process shredder fines with a throughput capability of approximately 3 tons per hour.

Changing World Technologies (CWT) Thermal Conversion Process

- CWT completed the large-scale test using actual shredder residue material. The results confirmed the results of the earlier small-scale tests.

Prior Accomplishments

FY 2007

- ANL was approached by two shredders who expressed interest in building a large-scale validation plant based on the ANL technology for recovering materials from shredder residue.

Mechanical Separation of Shredder Residue

- Completed testing of 10,000 pounds of shredder residue out of a 30,000 pound batch from the shredder who is building the 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, nonferrous 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/h plastics-separation sink/float module.
- Defined process conditions for upgrading the polycarbonate (PC)—acrylonitrile-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 sorters and near infra-red (NIR) systems for plastics, wood and rubber separation.

MBA's Plastics Separation Test

- Tests were 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 awarded a contract to OnTo Technology of Bend, Oregon to develop technology to recycle batteries used in hybrid and electric vehicles. Initial testing results were promising.

FY 2006

Mechanical Separation of Shredder Residue

- Ran one 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 shredder residue and of the fractions generated at ANL.
- Conducted preliminary investigation of costs and performance of commercial color sorters, electrostatic separators, and infra-red (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 Inc.'s (TPI's) process for conversion of urethane foam to polyol initiators. Over 1,200 lb of foam were used, and produced over 100 gal of polyol initiators.
- Conducted pilot-scale testing of CWT's process for converting shredder residue to fuels. Pre-processed shredder residue 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 Shredder Residue

- Ran four 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 shredder residue 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 "heavies" plastic fraction.
- Developed a wet process for removal of wood and rubber from the recovered polyolefin fraction.
- Recovered 5,000 lb of polyethylene (PE)/polypropylene (PP) essentially free of wood and rubber.

Other Accomplishments

- Conducted bench-scale tests and in a five-gallon reactor of TPI's, of a glycolysis process for conversion of urethane foam to polyol initiators.
- Conducted bench and pilot-scale testing of CWT's thermal depolymerization process for converting shredder residue 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 shredder residues and the second module includes the unit operations required to recover selected plastics from the mixed plastics concentrates.

FY 2004**Mechanical Separation of Shredder Residue**

- Completed construction, shakedown, and start-up of the bulk separation facility.
- Ran six 5-ton trial campaigns in the 4th quarter.

Froth-Flotation Process for Recovering Plastics

- Completed construction and shakedown of the pilot plant with electronics plastics.
- Ran a shakedown campaign with shredder-residue 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**Validation Plant**

- Support the shredder in the design, construction, and operation of the validation plant.
- Analyze data from the validation plant.
- Evaluate the quality of recovered materials.
- Determine the economics of the process based on the full-scale validation-plant operation.

Mechanical Separation of Shredder Residue

- Process shredder residue generated by controlled shredding of selected items (autos only).
- Shred automotive nano composites to determine potential release of nano particles.
- Investigate necessary process modifications, if any, for recovering new lightweighting materials and their impact on recycling of other materials.

Froth-Flotation Process for Recovering Plastics

- Conduct process improvement studies to increase value and reduce cost.

Other Future Plans

- Evaluate new, novel processes for recycling shredder residue including its conversion to fuels and chemicals.
 - Develop and evaluate processes for recycling automotive lightweighting materials including lightweighting metals (aluminum, magnesium, and titanium).
-

Introduction

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, TPI, VW-SiCon, CWT, Energy Anew, and ANL mechanical separation processes. Process improvement research is ongoing on the ANL flotation process. Work is also continuing with OnTo on battery recycling.

Validation Plant

Two shredders have expressed interest in building a plant on their sites to validate the results obtained in ANL's pilot plant. Agreement has been reached with a shredder to build a 20 ton/h validation plant to confirm process yields, process economics, and to confirm qualities of the recovered polymers. Before the agreement was signed, 15 tons of shredder residues from three of the plants of this shredder were processed in our pilot-plant to determine if a business case exists. The material was first separated in the mechanical-separation plant to recover the polymer concentrate and then the polymer concentrate was processed in the flotation plant. Discussions followed to explore licensing opportunities and an

agreement has been reached. Process flow diagrams have been finalized. The building for housing the flotation part of the process and the critical parts of the mechanical process has been undergoing renovation and utilities have been brought to it. Specification of the equipment for the mechanical-separation system is complete and some of the equipment for the mechanical system and for material handling has been ordered. Placement of purchase orders for the equipment is underway and is expected to be completed by mid-November 2008. Specifications of the equipment for the flotation system are essentially complete. Placement of purchase orders for the equipment is expected to be completed by mid-December 2008. Specifications of the equipment for the polyolefins upgrade system are complete. Placement of purchase orders for the equipment is expected to be completed by end of January 2009.

Characterization of Shredder Residue

Over 130 tons of shredder residues from eight facilities were processed in ANL's mechanical-separation plant. Table 1 shows the average composition of the different fractions produced. Large variations in the concentration of fines, metals, rubber and wood and less variation in the composition of the plastics fraction were observed.

The weight percent (wt %) of the polymer concentrate recovered from eight runs totaling 80,000 lb of shredder residue from a given source conducted over a 6-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.

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

	Shredder Residue	Oversized Heavies	Oversized Foam rich	Fines ^a	Ferrous Rich	Nonferrous Rich	Lights	Polymer Concentrate
Weight (lb)	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
High Impact Polystyrene (HIPS)	261	0	0	0	4	8	15	234
Nylon	379	0	0	0	4	9	19	347
Polyvinyl Chloride (PVC)	512	0	0	0	0	0	0	511
Polyoxyphenylene (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
Polyurethane (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 ^a	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

^aFines are material smaller than 0.25 inch in size and also contain some polymers and metals.

ANL Pilot-Plant

The 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 shredder residue to yield a polymer concentrate, ferrous and nonferrous concentrates, a fines fraction, and other fractions. The plant achieved over 90% recovery of the plastics targeted for

recovery as a polymer concentrate and over 95% recovery of the metals in the shredder residue.

The polymer concentrate included high and varying amounts of wood and rubber. Wood was about 1–4 wt %.

Separation of Wood and Rubber

Trials using commercial air aspirators, air classifiers, gravity tables, and mineral jigs to remove wood and/or rubber from the polymer concentrate did not yield satisfactory results. Trials using wet methods 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/h of polymer concentrate. The process separated over

75% of the rubber and produced a rubber fraction containing less than 10% of nonrubber material.

Froth-Flotation Pilot-Plant

This facility includes six continuous stages for the separation of targeted plastics. 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. Over 30,000 lb of polymer concentrate from shredder residue has been processed in this facility. The recovered fractions are described in the following sections.

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 lb of typical shredder residue.

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 ^a
Weight (lb)	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	59	10	0	0	0	1	5	42
Total	10,044	1,736	141	108	689	856	1,203	5,311

^aRubber and metals are to be recovered from these streams.

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. It is also rich in filled nylons.

Rubber

Recovered rubber fraction was upgraded by the ANL dry rubber separation process to over 90% rubber. The other 10% were mostly plastics.

In summary, the process has recovered the unfilled polyolefins, 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.

A 5,000 lb/h flotation module has been built and tested using over 10,000 lb of mixed polymers. Modifications to the tank and to the operating procedures resulted in over 90% reduction in the amount of chemicals used and in the amount of waste water generated.

Development of a Process Flow Sheet

A general process conceptual design for a 20 ton/h mechanical-separation plant has been developed. The flow sheets for the validation plant have been finalized. The process steps for the general conceptual design include (1) a device to separate large metals and rocks, (2) a screen to separate large pieces of fabrics, foam and tire rubber, (3) a shredder, (4) a screen or a trommel to separate

"fines," (5) a magnet and eddy current to separate metals, (6) a granulator, and (7) an air classifier to remove "lights" from the granulated material. Tests using shredder residue were conducted to evaluate the performance and cost of various equipment used in the design. ANL conducted analysis of the sensitivity of the cost of producing the polymer concentrate to yield per ton of shredder residue, value of the by-products (ferrous, nonferrous, etc.), cost of utilities, number of operating shifts, cost-of-capital, etc. The cost can be as low as \$0.02 per pound of polymer concentrate for a plant operating three shifts per day and the polymer concentrate is about 45% of shredder residue to over \$0.075 per pound for a plant operating one shift and the polymer concentrate is only 15%. The value of the recovered metals has a significant impact on the overall cost. The value of the metals depends on their concentration in shredder residue and on the composition of the metal fraction produced. Evaluation of Color and Infrared Sorters ANL 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 (full color and IR), (5) NRT (chip full spectrum and IR), and (6) PROTEC (chip full spectrum). Based on the results of these tests, it appears that at this time these systems cannot recover marketable polymer products from shredder residue nor can they separate wood and rubber from the recovered polymer fractions. ANL's process for the separation of wood and rubber from plastics appears to be 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.

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 2–6 mm range was between 30 and 50 wt % of the weight of the fines fraction. It contained up to 50% by weight 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, and a mixed plastic and rubber concentrate and recover the

residual metals as a metal concentrate. About 20% of the plastics had specific gravity < 1.

The ferrous material, including oxides, in the fines has been reduced significantly compared to years ago. Shredders are using more-efficient metal-separation equipment and less rust forms on later-model vehicles.

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 shredder residue contained less rubber and wood than the U.S. residue. MBA processed about 40,000 lb of Salyp's polymer concentrate and recovered five materials: polyolefin "A" and "B," filled PP, ABS, and HIPS. The total yield 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-good applications.

CWT

CWT processed a shredder-residue 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%), and heavy-hydrocarbon oils (15%), and 3% as gas.

PCBs in the input shredder residue was 21.8 parts per million (ppm) and in the products were below the detection limit. This indicated that the PCBs degrade during the process. The char contained 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 and 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) and in the char (87 ppm). None was found in the output liquid products.

The analysis also indicates that sulfur compounds degraded in the process. A test using about 1,000 lb of a pre-processed organic fraction derived from about 2,000 lb of shredder residue has been completed. The results confirmed the ability of the CWT process to produce a 18,000 BTU/lb diesel oil, a 12,000 BTU/lb solid carbon, and a 5,000 Btu/lb fuel gas with the largest fraction being methane.

CWT also completed a large scale (2,000 lb) test using actual shredder-residue material. The results confirmed the results of the earlier small-scale tests.

Energy Anew Recycling of Fines

Energy Anew conducted tests on 300-lb samples of fines (< 1 in.). Shredder residue was screened using a 7/8-in. screen and the material that passed the screen was processed to yield organic- and inorganic-rich fractions. Energy Anew constructed a mobile cyclonic system to process shredder fines with a throughput capability of about 3 ton/h. The system successfully sorts the fine fraction into a nonferrous metal concentrate, polyolefin concentrate, styrenic plastic concentrate and an organic fluff fraction. The process can be used in combination with coarse size-reduction systems to produce enriched-plastic products from a wide variety of raw materials including automotive shredder residue and shredded appliance, electronic and packaging scrap. Nonferrous concentrates of greater than 75% metal content have been demonstrated with use of additional down-stream grinding and sorting. Economic analysis of the process shows that recovery of plastic concentrates, metals and sand from windshield glass may be viable.

TPI Glycolysis Process

The process converted foam from shredder residue to polyol initiators at yields of 88% and 72%, for clean and dirty foam, respectively. Activated carbon reduced the concentration of PCBs in the products to < 2 ppm. Over 100 gal. of polyol initiator were produced from about 1,200 lb of foam.

Twenty gallons of the polyol initiator were propoxylated and were tested for making rigid foams. They required less or no catalyst and had better flame resistance than the foams made with

virgin polyols. Initial economic analysis indicated that the process is potentially economical.

OnTo Technology

OnTo Technology, under contract from USCAR, conducted research on recycling two kinds of advanced battery chemistries—NiMH and lithium ion—to determine the technical feasibility of recycling these types of automotive batteries. The process starts by discharging and dismantling of the battery followed by CO₂ extraction of the electrolyte. The battery is then shredded at room temperature.

Ferrous and nonferrous metals are then separated by magnets and eddy currents, respectively. The remaining material is then pulverized and the plastics are separated from the pulverized material. The powder containing the Li is then recovered. Copper and cobalt are then recovered from the residual material by an electrowinning process. Early results show promise.

Recovered materials have been analyzed and tested in new batteries with good results. The method has the potential to cost-effectively remove more materials of value including the lithium and electrolyte fractions than current technologies. OnTo is continuing their research to look at additional battery chemistries.

Co-Combustion of Plastics-Rich Material with Biomass

The ACC-PD co-funded a study with Plastics Europe to study the synergies of co-combustion of plastics-rich fuels and biomass. The study concluded that the benefits include fast drying and ignition of the biomass, higher furnace temperature and improved combustion efficiency. The study noted that chlorine and metallic aluminum contents in the fuel mixture should be compatible with the boiler type.

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E. Development of High Speed Multispectral Imaging for Sorting Automotive Plastics

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Contract No.: DE-FG02-06ER84559 Small Business Innovation Research (SBIR) and Small Business Technology Transfer Program

Objective

- Develop a cost-effective technology for the identification of end-of-life automotive plastics and their sortation according to polymer type.

Approach

- Design a primary optical system for imaging the infrared (IR) characteristics of automotive polymers.
- Design a high-speed, IR filtering system which integrates with the optical system and provides the mechanism for isolating a polymer signature.
- Design a secondary optical system which transfers the filter response to a multi-sensor detection array.
- Develop software for high-speed control of the filtering system and detection array utilizing a real-time operating system (RTOS).
- Develop software to identify the unique signatures of various polymers and subsequently segregate these pieces into desired groupings.
- Develop software for system-level configuration and control.
- Integrate and test sortation system.

Accomplishments During this Reporting Period (10/01/2007–09/30/2008)

Commercialization Activity

- NRT has realized a Ph III sale of an advanced real-time computing module based upon the Phase II technology under development.
- Development of the computing module is underway according to specifications received from the Ph III client.
- Delivery of the advanced computing module to the Phase III client is expected in spring 2009.

Primary Optical System

- Determined specifications for an IR lighting system—an innovative approach is being investigated that can optimize resolution and enhance sensitivity of the detection system.

- Mechanical design of a high-luminance IR lighting system is in process as an adjunct to the innovative approach discussed above.
- Began design of the primary optical system using an optical design software package.

IR Filtering System

- Have selected a high-speed, reconfigurable IR filters that meets design parameters.
- Have constructed an optical model of a reconfigurable IR filter for the purpose of simulation and testing.
- Using a Phase I prototype reconfigurable IR filter for testing and evaluation.

Secondary Optical System

- Design underway of the secondary optical system using an optical design software package.
- Design underway of mechanical components to integrate the primary optical, IR filtering, and secondary optical systems and IR detection array.

Detection Array and Data Acquisition Hardware

- Determined specifications for a multi-sensor infrared detection array.
- Determined specifications for detector acquisition hardware.
- Acquired a high-speed, Camera Link-based camera which incorporates the desired detection array.
- Acquired a Camera Link-based frame grabber which enables communication with and data acquisition from the IR detection array.
- Tests underway using QNX real-time system data acquisition rates and camera control with camera parameter setup through a Windows-based system.

Real-Time-Operating-System Software for Filter and Detector Control

- Evaluated various aspects of several RTOSs and selected QNX for this project.
- Ordered, received, and activated QNX developments tools – these are essential for programming and interacting with QNX.
- Ordered, received, and put into service a single-board computer (SBC), chassis and other ancillary hardware for hosting QNX and our proprietary software.
- Performed configuration design of RTOS and subsystems.
- Software development is underway of the RTOS and subsystems, including that needed for the Phase III advanced computing module.

Identification and Sortation Software

- Investigating potential algorithms for identifying polymers.

System Configuration and Control Software

- Have performed detailed examination of QNX to understand the interactions among its internal components.
- Development underway of communications between proprietary software components.
- Primary hardware and software structures defined—driver developments underway.

Investigate Complications from Coated Polymers

- Obtained samples of painted polymers from Argonne National Laboratory.
- Sent samples to a laboratory for analysis using a laser-induced breakdown spectroscopy (LIBS) technology which automatically penetrates surface coatings to expose underlying material.
- Testing performed on the samples using a double pulse laser and LIBS analyses to analyze underlying polymer materials. Test report received from laboratory performing tests and results analyzed.

Prior Accomplishments**Determined Desirable Polymers Present in Automobile Shredder Residue**

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

Constructed a Bench-Scale Unit to Determine and Demonstrate Feasibility

- 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 fiscal year (FY) 2009 will continue to focus on this SBIR Phase II project. A summary of Phase II plans for this period is provided below.

Design, Fabricate, and Test Commercial Computing Module

- Design and testing to be performed under this program.
- Component costs and manufacture costs to be provided by NRT external to this program.

Design of Primary Optical System

- Finish design of the primary optical system.
- Fabricate mechanical components.
- Assemble optical and mechanical components and test.

Design of IR Filtering System

- Determine optimal physical configuration of the reconfigurable filter through simulations with optical model.
- Order a reconfigurable filter with the desired configuration.
- Verify proper operation of the filtering system.

Design of Secondary Optical System

- Finish design of the secondary optical system.
- Finish design of mechanical components to integrate the primary optical, IR filtering, and secondary optical systems and IR camera.
- Assemble integrated systems.
- Verify intended performance of the integrated systems.

Develop Real-Time-Operating-System Software for Filter and Detector Control

- Finish development of filter and detector control.
- Test controlling software.

Develop Identification and Sortation Software

- Develop various algorithms for identification.
- Test algorithms for speed, efficiency, and accuracy with simulated data.
- Test algorithms with acquired data.
- Develop controlling software for grouping materials.

Develop System Configuration and Control Software

- Finish interface protocols for communication between proprietary software components.
- Implement system control and configuration modules.
- Test system control and configuration software.

Test Integrated Sortation System

- Assemble components into a small but commercial-scale prototype sorting system.
- Evaluate overall performance of the assembled system.
- Evaluate sortation performance of the system by sorting shredder residue.

Introduction

The objective of this project is to develop a cost-effective technology for the identification of end-of-life automotive plastics and their sortation according to polymer type. This technology will be capable of simultaneously acquiring polymer spectra from multiple spatial locations, yet operate at high speeds when compared to current state-of-the-art detection systems.

The prospective sortation system may be considered as composed of three sub-systems: an optical filtering unit, a real-time processing unit and a control unit. Specifications, simulations, and mechanical designs for the optical filtering unit have begun. Development of the real-time

components has progressed significantly, where many of the subtasks (e.g., acquiring hardware and toolsets) are completed. Likewise, significant progress toward implementing the control unit has been made due to investigating the underlying operating system, charting an implementation strategy, and testing several concepts with software applications.

Technological developments in this program have enabled the commercial sale to an NRT customer of a computing module based upon the systems being developed under this SBIR program. The unit that has been ordered by our customer will incorporate certain aspects of the RTOS developments described more fully in this report.

Optical and Filtering Unit

The initial processing segment of the intended sortation system is composed of a complex optical and filtering unit which images the IR characteristics of automotive polymers. It is intended that this unit continuously image the spectral signature of pieces which are larger than 0.25" and lie within a 24" processing width. An inherently low-intensity polymer signature coupled with short acquisition times (required for high-speed and high-volume sorting) requires complex and efficient imaging optics. The tight optical constraints of a high-speed, reconfigurable IR filter further complicate the optical design.

The manufacturer of a reconfigurable IR filter is assisting in the development of an optically valid model of a filter for use in simulations.

Incorporating this model into an optical design software package enables evaluation of several possible configurations before a prototype is constructed. It is estimated that 40% of the effort for developing this unit has been completed.

Real-Time Components

The real-time components may be considered as the hardware and software necessary to effect the high-speed sortation. The hardware components consist of a single-board computer (SBC) with a multi-core processor, high-speed line-scan camera, frame-grabber, and digital input/output (I/O) controller. The software components consist of an RTOS, hardware drivers, polymer identification algorithms, and material removal routines.

An early step in planning for this project was to produce a compilation which functionally described the various components of the intended system and chart their interactions. Once completed, the specifications for individual components were further refined so that hardware selection could occur. As a result, much of the hardware has been ordered and received.

The foundation for all of our real-time software components is QNX. This commercially available RTOS seems to be the best solution for the project and some extended period of effort has been directed towards familiarization with and the understanding of its intricacies. We have acquired

the development tools for QNX, installed the operating system on the SBC, explored the various aspects of the system, and are now in the process of constructing code specific to our project.

User Interface and Control Software

The control unit may be considered both as a system supervisor and as a means for personnel to adjust sortation parameters. In its supervisory role, the control unit provides a mechanism for programmatically controlling external hardware (e.g., conveyors, lights, alarms) as well as invoking the sortation process. The control unit also provides a set of applications, collectively termed a "user interface" (UI), which allow an operator to readily manipulate sortation parameters.

The response latency criterion for the control unit software is considerably less stringent than for that of the sortation software. This relaxed requirement permits us to implement the software on a non-RTOS, such as Windows XP. Isolating the non-real-time control from the real-time sorting process provides two advantages. First, the sorting process is more stable since any critical failures in the non-real-time software has a much lower probability of corrupting the real-time processes. Second, since the UI applications may execute on a computer with a network connection to the sorting module the sorting system may be controlled from any remote location or simultaneously monitored from various locations.

Defining the base structures for UI applications and protocols for control software is a significant task. We are well into this endeavor and reached the point of implementing and testing several of these concepts in Windows XP.

Conclusion

The research on this Phase II project is generally progressing as intended. No outstanding difficulties have been encountered.

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