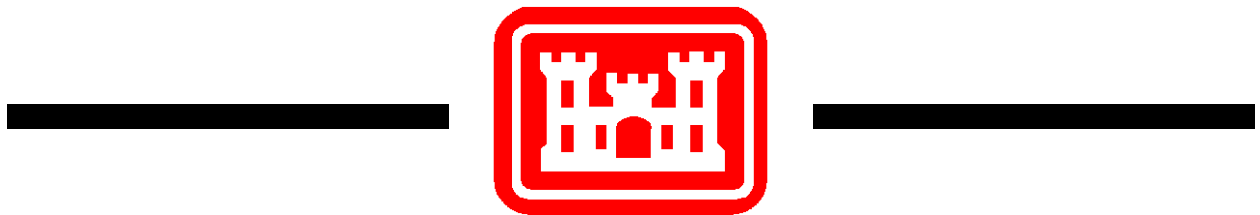


PUBLIC WORKS TECHNICAL BULLETIN PWTB 200-1-145
30 NOVEMBER 2014

**RECYCLE OF SOLVENT CLEANERS FOR
TACTICAL AND TRANSPORTATION VEHICLES**



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FACILITIES ENGINEERING
ENVIRONMENTAL

RECYCLE OF SOLVENT CLEANERS FOR TACTICAL
AND TRANSPORTATION VEHICLES

1. Purpose.

a. The purpose of this Public Works Technical Bulletin (PWTB) is to outline ways of extending the life of cleaning solvents by filtering dirt, oils, and tar from the cleaning fluid and fortifying it with the addition of depleted active ingredients or fresh concentrated solvent.

b. All PWTBs are available electronically at the National Institute of Building Sciences' Whole Building Design Guide webpage, which is accessible through this link:

http://www.wbdg.org/ccb/browse_cat.php?o=31&c=215

2. Applicability.

a. This PWTB applies to all U.S. Army Public Works activities and facilities having surface cleaning activities for tactical and transportation vehicles.

b. Note that the contents of this report are not to be used for advertising, publication, or promotional purposes. Citation of trade names does not constitute an official endorsement or approval of the use of such commercial products. All product names and trademarks cited are the property of their respective owners. The findings of this report are not to be construed as an official Department of the Army position unless so designated by other authorized documents.

3. References.

a. Army Regulation (AR) 200-1, *Environmental Protection and Enhancement*, 28 August 2007.

b. ASTM D6361 / D6361M - 98(2010)e1, *Standard Guide for Selecting Cleaning Agents and Processes*, 01 June 2010.

c. MIL-PRF-680B, *Solvent Selection Guide*, 26 October 2006.

d. U.S. Environmental Protection Agency (USEPA) Method 1664 (USEPA 2013), Revision A: N-Hexane Extractable Material (HEM; Oil and Grease) and Silica Gel Treated N-Hexane Extractable Material (SGT-HEM; Non-Polar Material) By Extraction and Gravimetry.

4. Discussion.

a. AR 200-1 requires that Army installations comply with Federal environmental regulations, including air emission restrictions established by the Clean Air Act.

b. The Resource Conservation and Recovery Act requires the U.S. Environmental Protection Agency (USEPA) to promulgate regulations regarding the storage, processing, and disposal of solid and hazardous wastes. In 1984 the Act was augmented by the Hazardous and Solid Waste Amendments, which included provisions that encourage the recycling and reuse of hazardous wastes.

c. Congress established the Waste Minimization and Pollution Prevention (WMPP) program to demonstrate promising off-the-shelf environmental technologies at Army installations. Funding for the WMPP program ended in fiscal year 2005 (FY05). During its 12-year tenure, this program evaluated and demonstrated many commercially available environmentally friendly cleaners in the laboratory.

d. ASTM D6361/D6361-98(2010)e1 is a general guide that is used in developing cleaning requirements for manufacturing, maintenance, or overhaul specifications. This guide was designed to be application specific for individual cleaning tasks, and to assure the design engineer that cleaning agents and processes selected by the industrial or manufacturing engineer will be compatible with both the part material and with subsequent processes. Industrial or manufacturing engineers use the guide to customize their selection of cleaning products based on: the materials of the part being cleaned; the cleanliness required

for the subsequent processes; and environmental, cost, and health and safety concerns.

e. MIL-PRF-680B is a U.S. Department of Defense standard used to describe products that meet specific performance and manufacturing standards for equipment and chemicals.

f. U.S. Army vehicle maintenance facilities attempt to select environmentally friendly surface cleaners that are free of Hazardous Air Pollutants (HAPs), that contain the minimum possible amounts of Volatile Organic Compounds (VOCs), and that eliminate greenhouse gas emissions. They also consider the costs associated with the cleaners and the amount of waste generated from the cleaning process. This PWTB identifies and provides recommendations on extending the life of the cleaning tanks by reusing the solvents, thus reducing both purchase costs and costs associated with waste disposal.

g. Appendix A to this PWTB describes potential options for extending the life of the surface cleaning baths for removing tar and asphalt from tactical and transportation vehicles.

h. Appendix B contains a list of technical references cited in Appendix A.

i. Appendix C contains a list of acronyms and abbreviations used in this PWTB.

5. Points of Contact.


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FOR THE COMMANDER:



for JAMES C. DALTON, P.E., SES
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Appendix A

RECYCLE OF SOLVENT CLEANERS FOR REMOVING TAR AND ASPHALT FROM TACTICAL AND TRANSPORTATION VEHICLES - REDUCTION OF SOLVENT COSTS AND WASTE MINIMIZATION

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INTRODUCTION

Background

Ground vehicles that come into the maintenance facility (especially bitumen spreaders) commonly have asphalt ("road tar") on their surfaces, which must be removed before the vehicle can be washed or painted. The recent focus at Army maintenance facilities is to begin using surface cleaners that are free of Hazardous Air Pollutants (HAPs) and that contain minimal amounts of Volatile Organic Compounds (VOCs), thereby eliminating greenhouse gas emissions. These facilities also have an interest in reducing overall energy costs associated with surface cleaning operations. Requirements include the use of solvents compatible with wastewater treatment plants that handle phosphate type solutions.

An earlier ERDC-CERL study (Baird et al. 2009) identified Citrus King, an environmentally friendly aqueous cleaner, as one of the most suitable cleaners for removing tar and asphalt from oil spreaders and other components of the Army transport vehicles. The use of the Citrus King as a cleaner for tar and asphalt was demonstrated at the Red River Army Depot (RRAD), where it was given very good results in removing road tar from military vehicles. While the solvent is expensive (about \$20/gal), its effective cost can be reduced (and its environmental desirability increased) if its useful life is extended by cleaning and reuse.

The life of the cleaner from a tar removal bath can be extended by: (1) removing the accumulated/emulsified contaminants from the cleaner bath in situ, or (2) by removing the condemned bath contents (saturated with dirt, tar, and asphalt compounds) to a separate container, then processing it to recover the solvent cleaner for reuse. Processing could include filtering tar and other dirt out of the solvent, then fortifying the remaining solution with the depleted emulsifying agents. This would reduce the overall amount of fresh cleaning solvent a vehicle cleaning facility would use, and also reduce the amounts of spent cleaner (tar and dirt laden solvent) requiring disposal.

Current Cleaning Procedure

PWTB 200-1-110 (HQUSACE 2011) reviewed 47 commercial solvents and identified about 10 solvents for laboratory testing with well defined protocols. These solvents were ranked based on their ability to remove the tar from stainless steel coupons and to

dissolve tar. Two of these solvents were demonstrated at RRAD on bitumen spreaders in a 10x8x6-ft tank. Currently RRAD alone has about 28 tar/bitumen spreading tanks to be cleaned and refurbished. These vehicles are sent to various Army depots from paving operations both inside and outside the continental United States. This maintenance often requires replacement of the tar frozen distributors costing about \$5,000 for the parts alone for each of the vehicle.

Current Effort

This work began with a survey of current literature describing commercially available solvent recycle and reuse options. One survey of DOD facilities (Rhee et al. 1995) listed desired general properties of cleaning solvents and provided specific guidance to identify cleaners for application to surfaces of tactical and transport vehicles. Trivedi et al. (2004) presented an excellent review of general cleaner requirements and green cleaning alternatives for weapon systems and machine parts for the DoD. PWTB 200-1-110 (HQUSACE 2011) and Baird, et al. (2009) gave a detailed guidance for solvent selection.

This effort focused specifically on methods that would reduce the cost of solvents by minimizing the total solvent requirement and the amount of spent solvent to be disposed. Bench-scale tests evaluated the use of de-emulsifiers to settle the tar and dirt from the spent solvent, of the use of filtration to separate tar and dirt from the spent solvent, and of fortifying the cleaned solvent with fresh solvent to extend its useful life. The fortified solvent was also evaluated for performance in a laboratory environment. The results of this evaluation may be used to provide a cost effective, environmentally friendly, and sustainable process for tar and asphalt removal. The remainder of this PWTB documents and recommends specific applications for removing tar and asphalt from ground vehicle surfaces.

Benefits of Implementation

The processes outlined in this PWTB will enable installations to select environmentally friendly cleaners and cost-effective, sustainable processes to remove tar and asphalt from transportation vehicles. This document provides guidance to extend the life of the cleaner bath, which:

1. Will reduce the amount of spent cleaner waste to be disposed. Reducing the waste is a sound practice as this will lower the

environmental exposure and also the costs associated with handling and disposal.

2. Will extend the useful life of the cleaner bath, which will reduce the net amount of new cleaner to be purchased, reduce the costs associated with acquisition time, and potentially simplify the logistics associated with the materials handling.
3. Will reduce liabilities associated with VOC and HAP emissions, handling, and disposal.
4. Will help reduce the overall energy costs associated with the surface cleaning operation, including those energy (and logistical) costs associated with storage, solvent recovery, and residuals disposal.
5. May also reduce operating costs due to increased productivity by reducing the frequency of cleaning and refilling the bath.

Requirements for Cleaners

The recycled cleaner should meet the same three basic criteria that are applicable to fresh cleaner:

1. Cleaning Effectiveness. This is a quantitative measurement of how much tar or asphalt the recycled/refurbished solvent can remove. Simply stated, the best solvent removes the most tar or asphalt.
2. Corrosiveness. This refers to the solvent's ability to damage or rust the metal. Very corrosive solvents can cause surface rust immediately after cleaning. Checking the pH of the solvent can give an indication of its corrosiveness. If the pH is either too high or too low, it can easily corrode the metal, rendering it useless.
3. Cost. Army maintenance facilities use large quantities of solvents to clean vehicles and other equipment, and have a great interest in minimizing solvent costs. Overall cost can be reduced by the ability to recycle and reuse the solvent after its initial use, up to the break-even cost associated with the costs in training, equipment, and downtime required to recycle the solvent.

Laboratory Evaluation of Cleaner Recycling Options

Literature Review

An Ohio Environmental Protection Agency (1995) fact sheet summarizes ways to extend the life of aqueous cleaning solutions. Potential options suitable for recycling dirty cleaners include distillation and membrane filtration. Both of these recycling techniques involve emptying the dirty cleaner out of the cleaner bath, then processing and reusing the cleaner.

Distillation, which is an energy intensive process, is suitable only for highly volatile organic solvents. Since new distillation equipment for onsite recovery can be expensive, distillation may not be cost effective if a new setup is to be installed.

Setting up a membrane filtration process close to the bath is also an energy intensive, pressure-driven process. However, depending on the cost of the recovered cleaner and its quality, a low pressure membrane or cartridge filtration process may be feasible. Simple cartridge filtration to remove the dirt, tar, and oils may be a desirable option for extending the life of the bath. This type of cartridge filtration unit can be set up next to the cleaner bath to filter out floating oils, tar, and materials that have settled to the bottom of the bath. After filtration, a small amount of fresh solvent can be added to the tank to keep the bath active and efficient.

Other approaches may include: (1) membrane filtration followed by fortifying the filtrate with active ingredients (emulsifiers or surfactants), (2) simple decanting, or (3) gravity separation of the dirt and oils.

For tar-contaminated solvents, an evaluation of sedimentation or decanting processes in combination with addition of ionic and non-ionic compounds, or surfactants (as emulsion breaking techniques) shall also be explored. Protocols detailed in USEPA Method 1664 state that emulsion-breaking techniques must be used when emulsions consist of more than one-third of the solvent layer. The following section describes a laboratory method to determine the best ionic or non-ionic surfactants that can be used to separate specific solvents from tar-solvent solutions.

Laboratory Method for Determination of Surfactants for Destabilizing Tar-Solvent Emulsion

The development of an environmentally benign technology/protocol for tar solvent recycling is an important factor in meeting the sustainability goal of the Army bases. This section explores the use of surfactants, both ionic and non-ionic, to break emulsions of oil and water. Surfactants are expected to increase the aqueous solubility of solvents and the dissolution of hydrocarbons based pollutants such as tar.

The theory behind using a surfactant to break emulsions is based on the basic molecular structure of any surfactant molecule. The head of the molecule is hydrophilic and the tail is hydrophobic. In combination, these opposing properties lower the surface tension of the solvent in which the targeted material is dissolved. Lowering interfacial tension between the tar and the solvent makes it much easier to suspend the tar in the solvent, allowing for a clean separation. The hydrophilic groups are attracted to the polar solvent and keep the surfactant from being separated from the polar solvent.

When high concentrations of surfactant are introduced into an emulsion, hydrophobic groups gather and form a surfactant "micelle" (Figure A-1). This surfactant micelle can solubilize hydrophobic organic compounds by partitioning hydrophobic organic compounds between the micelle cores of the surfactant and the solvent.

There are two major types of surfactants, ionic and non-ionic. Ionic surfactants are molecules that separate into cations and anions in solution, where one of the ions breaks the emulsion. Anionic surfactants have negatively charged hydrophilic ends and are typically used to remove dirt from solutions or surfaces. Cationic surfactants have positively charged hydrophilic ends and are commonly found in fabric softeners. Since these surfactants have a charge and attract oppositely charged molecules, their ability to disperse oil in water is weakened. Non-ionic surfactants do not have a charge since the molecule does not separate once it enters solution, and therefore are better for removing oil and tar. Typically, non-ionic surfactants are less pH sensitive than ionic surfactants and the adsorption of non-ionic surfactants increases with temperature due to a decrease in surfactant solubility.

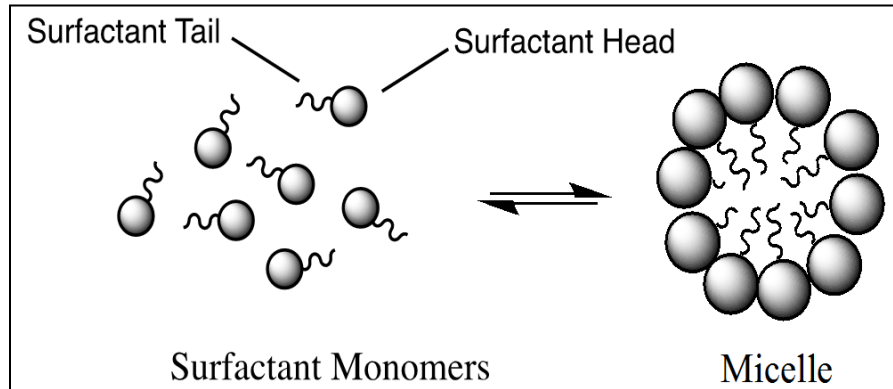


Figure A-1. Schematic of surfactant monomers forming a surfactant micelle.

Experimental Details

Materials

Table A-1 lists materials used in this study and their properties.

Tar Solution Preparation

A 500 ml solution of solvent (Citrus King) and tar was prepared by dissolving tar and solvent in 20:1 volumetric ratio. The solvent used was 100% Citrus King as received from the vendor, or as a mixed solution consisting of either 75:25 or 50:50 wt/wt ratio of solvent to water. The solution was allowed to sit for up to 48 hours to completely dissolve.

Surfactant Testing

Ten (10) ml of the tar solution was dispensed into each test tube along with 1 wt%, 5 wt%, or 10 wt% of each surfactant, creating 15 different solutions along with a control solution with no surfactant. The vials were stirred manually, covered with aluminum foil, and allowed to sit for 72 hours.

Analysis

Large quantities (>100 ml) of supernatant solution were extracted from batches of solution prepared as above. The supernatant (decanted or filtered) solution was evaluated for cleaning capabilities using tar-coated metal coupons.

Table A-1. Study materials and their properties.

Category	Name	Properties	Supplier
Tar	WRI 956-10-B		Western Research Institute
Solvent	Citrus King		Citrus Depot
Surfactant	NaCl	ionic	Acros
Surfactant	CaCl ₂	ionic	Fischer Scientific
Surfactant	Brij S 100	ionic	Sigma-Aldrich
Surfactant	Tropaeolin 000 No. 2	ionic	Sigma-Aldrich
Surfactant	Lignosulfonic acid	ionic	Sigma-Aldrich

Tar-coated coupons were prepared. The amount of tar was obtained by weighing the coupons before coating with tar and weighing the dried coupon with tar. The dried coupon was then placed in 100 mL of extracted solvent. The time till the coupons were clean was recorded and the percent of tar removed was determined.

Results and Discussion of Tar Removal by the Extracted Solvent

The pure solvent was originally tested. When tar was dissolved, a black solution was formed. The surfactants were added and allowed to sit for several days. The emulsion could not be broken no matter how much surfactant was added. Microfiltration was also attempted, but was could not separate the tar from the solvent. Figure A-2 shows the results of the surfactant testing. Since it did not separate the pure solvent/tar solution, this method was not tested further.

During initial testing with Citrus King, it was noted that Citrus King could be combined with water to reduce the amount of solvent used, while still retaining cleaning ability. When combined with water, the product formed a milky orange solution, which when combined with the tar, formed a grainy black-brown solution. When surfactants NaCl and CaCl₂ were added, there was an immediate partial disintegration of the emulsion, which was proportional to the amount of water in the original solution. The time required for formation of two distinct layers is termed the "emulsion breakdown time." This time varies depending on the concentration of tar, dirt, the free solvent, surfactant, water and temperature

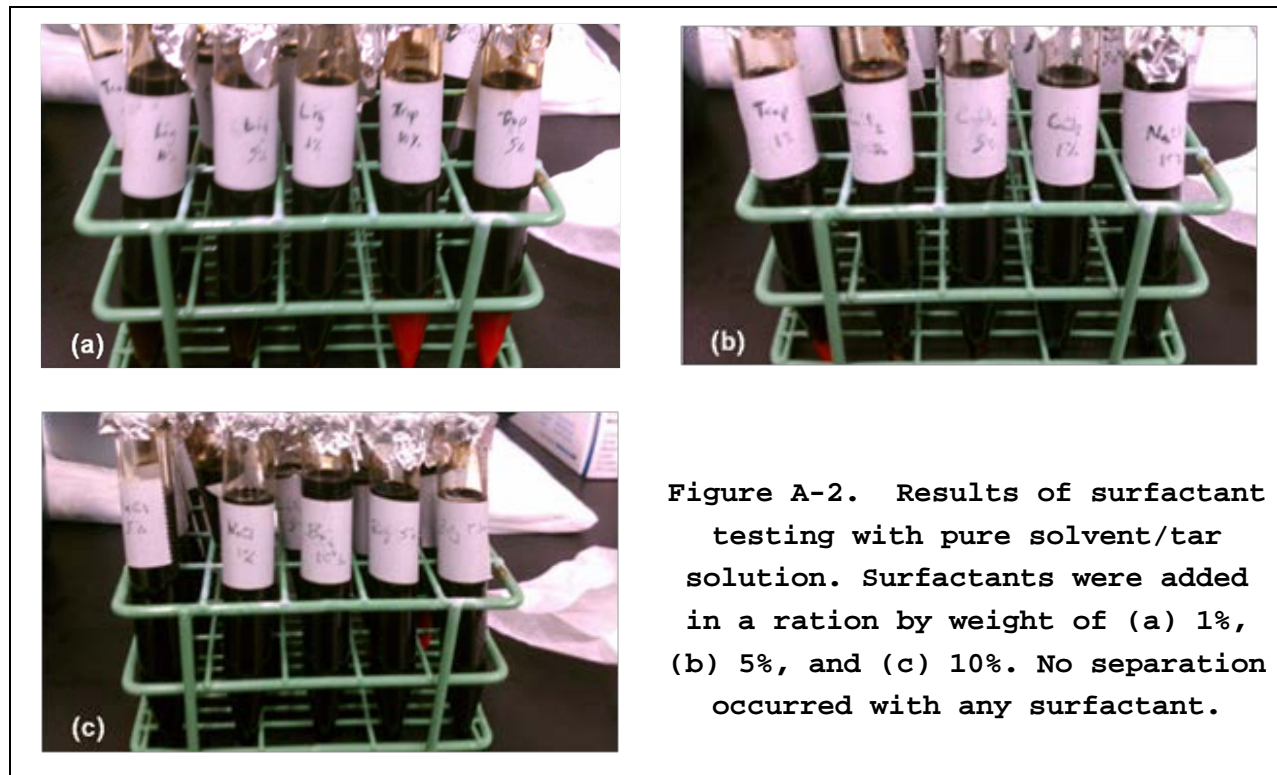


Figure A-2. Results of surfactant testing with pure solvent/tar solution. Surfactants were added in a ration by weight of (a) 1%, (b) 5%, and (c) 10%. No separation occurred with any surfactant.

There was no change in the separation between 4 and 48 hours, suggesting that after immediate separation, there is no additional separation. These two surfactants did not separate the solvent from the solution, only the water. This is most likely due to the ionic potential of these salts. The ions are small enough to break the solvent from the solution, but also the water from the solvent. The solvent then recombines with the tar at a high rate such that separation is impossible.

Lignosulfonic acid gave slow separation, taking about 48 hours to separate. It also turned the solvent slightly red (Figure A-3). This solution showed limited cleaning capabilities because the acid chemically bonded with the solvent.

Tropaeolin was able to separate a small amount of solvent from the tar-solvent emulsion. The solvent extracted using Tropaeolin could be fully reused to clean additional tar with a cleaning ability similar to the solvents original ability.

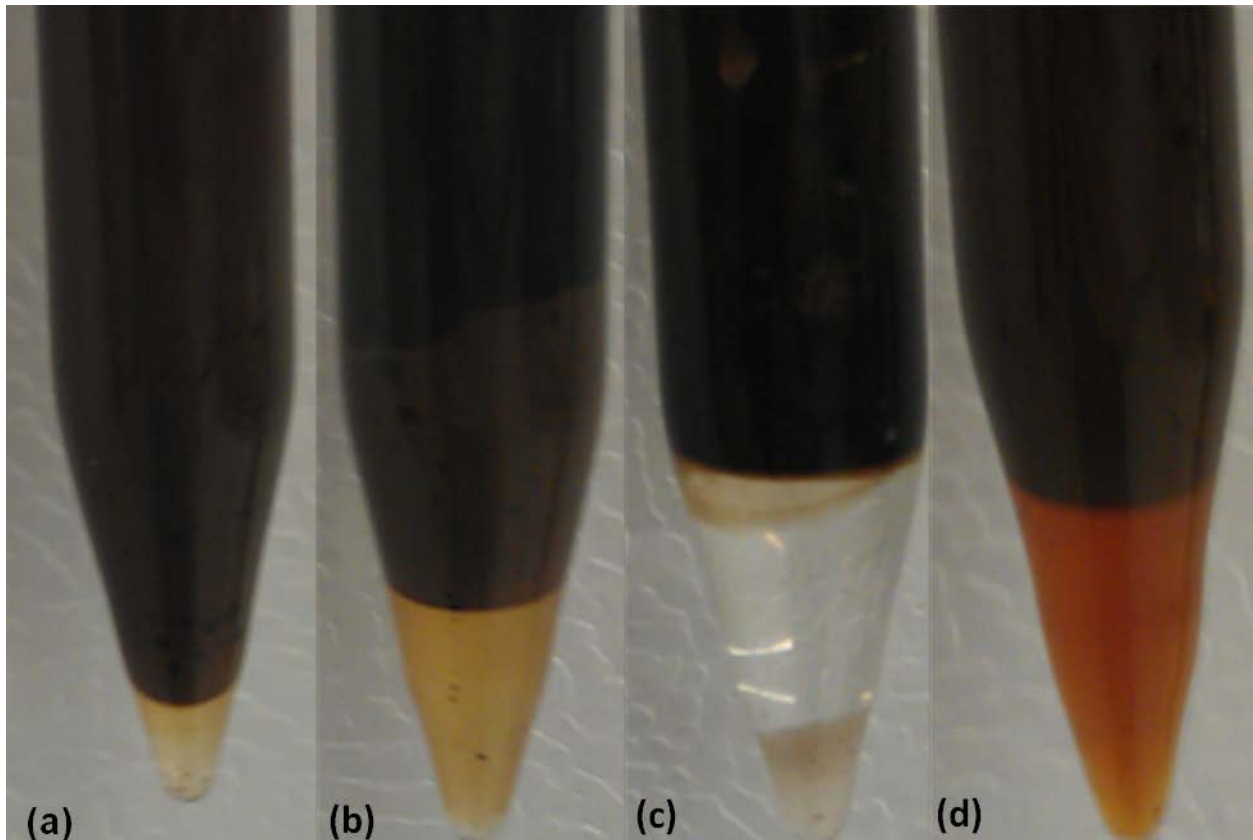


Figure A-3. Solvent separation due to 5% by wt of (a) Tropaeolin, (b) Brij S100, (c) NaCl, and (d) Lignosulfonic Acid added to the tar-solvent emulsion

The last surfactant used was Brij S100. Brij had the highest emulsion breaking ability of the surfactants tested. It yielded a solvent that was fully reusable for tar and asphalt cleaning. Figure A-3 clearly shows the different emulsion breaking ability of each tested surfactant. While lignosulfonic acid and NaCl showed the greatest separation ability, they did not produce a solvent that could be reused. Brij S 100 and Tropaeolin were able to produce a completely reusable solvent, but did not produce much of the desired solvent.

Finally, the tar-solvent emulsion and the tar-solvent-water emulsion were allowed to sit untouched for roughly 3 months to test the ability of both types of emulsion to separate on their own. After 3 months, the tar-solvent emulsion without any water showed no separation. The emulsion with water however, did separate after roughly 3 months (Figure A-4a). The solution was agitated to ensure complete separation of tar and solvent. The resultant mixture of 50% water and 50% Citrus King (Figure A-4b) showed a lighter yellow-orange solvent that smelled slightly like sour oranges. The tar sank to the bottom of the solution as thick, viscous liquid ("ooze"). The solvent was removed and the tar solution that remained was set aside.

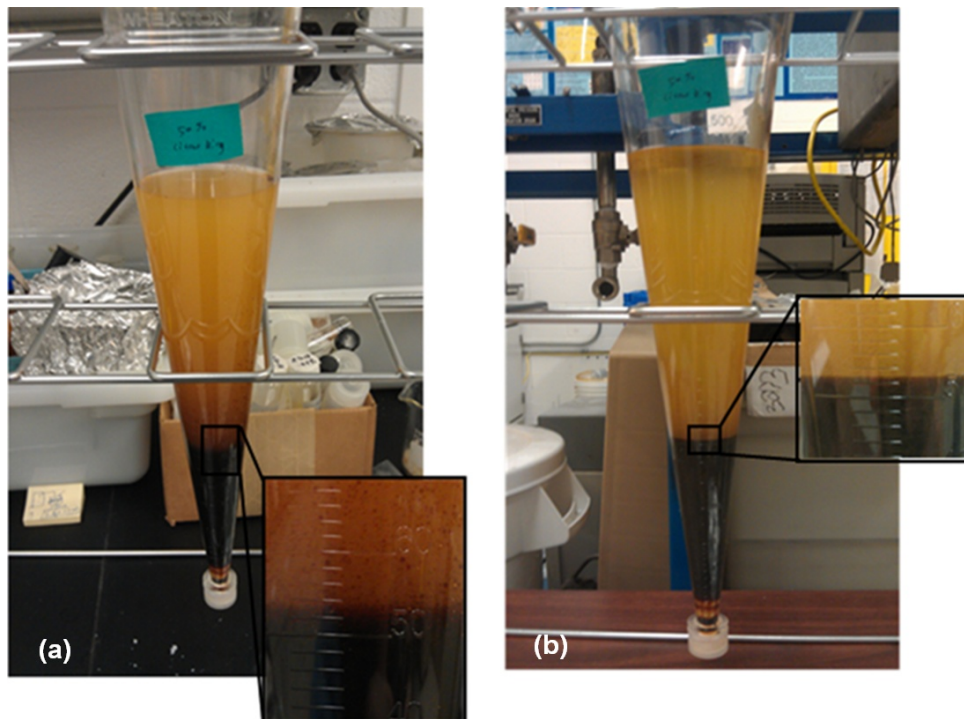


Figure A-4. 50% Citrus King, 50% water after being allowed to settle for 3 months. The solution was agitated (a) and then allowed to sit for 24 hours (b) to see if the solution would remain separated.

The solvent was then put through cleaning tests; Table A-2 lists (and Figure A-5 shows) the results.

Table A-2. Results of cleaning tests of the recycled 50% Citrus King 50% water solution

Mass of Added Tar (g)	Tar Removed (g)	% Tar Removed	Cleaning Time (h:mm)
0.9812	0.9869	100.58%	3:00
1.0582	0.9265	87.55%	3:00
1.0106	0.9481	93.82%	3:00
Average Percent removed: 93.98%			

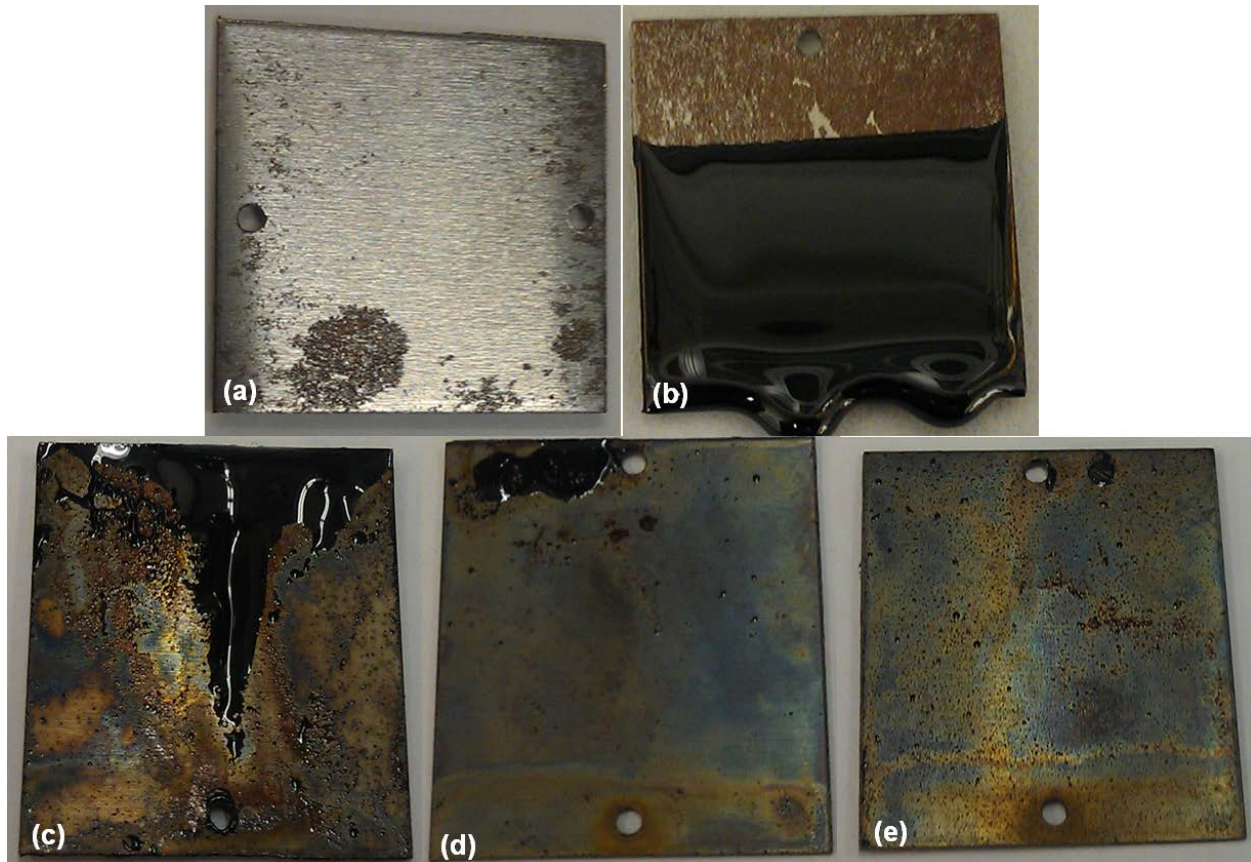


Figure A-5. Results of cleaning tests using the recycled 50% Citrus King 50% where (a) is an unused metal coupon, (b) is a tar coated coupon, and (c-e) are cleaned metal coupons using recycled 50% Citrus King 50% water solution for 3 hours at 298 K.

The recycled solution was tested for its ability to clean freshly prepared coupons. After 3 hours, the coupons did not appear to be visually free of tar. The tar floated to the top of the solvent making it difficult to fully clean the metal coupon.

There was significant rusting in each of the three samples, due to the coupon having been immersed in the solvent for too long. However, the rusting problem can be overcome by using a deeper container and higher solvent level, removing the tar as it floated to the top, leading to a shorter immersion time and overall better cleaning ability.

An identical separation test was performed over a period of 3 months using a 75% Citrus King 25% water tar emulsion (Figure A-6). After 3 months, the solution separated into an orange lower layer, and a black upper layer. The orange layer was similar in appearance to the unused solvent, though slightly lighter in color. The black layer was slightly more viscous than the orange layer.

The solvent was then put through cleaning tests, Table A-3 lists (and Figure A-7 shows) the results.

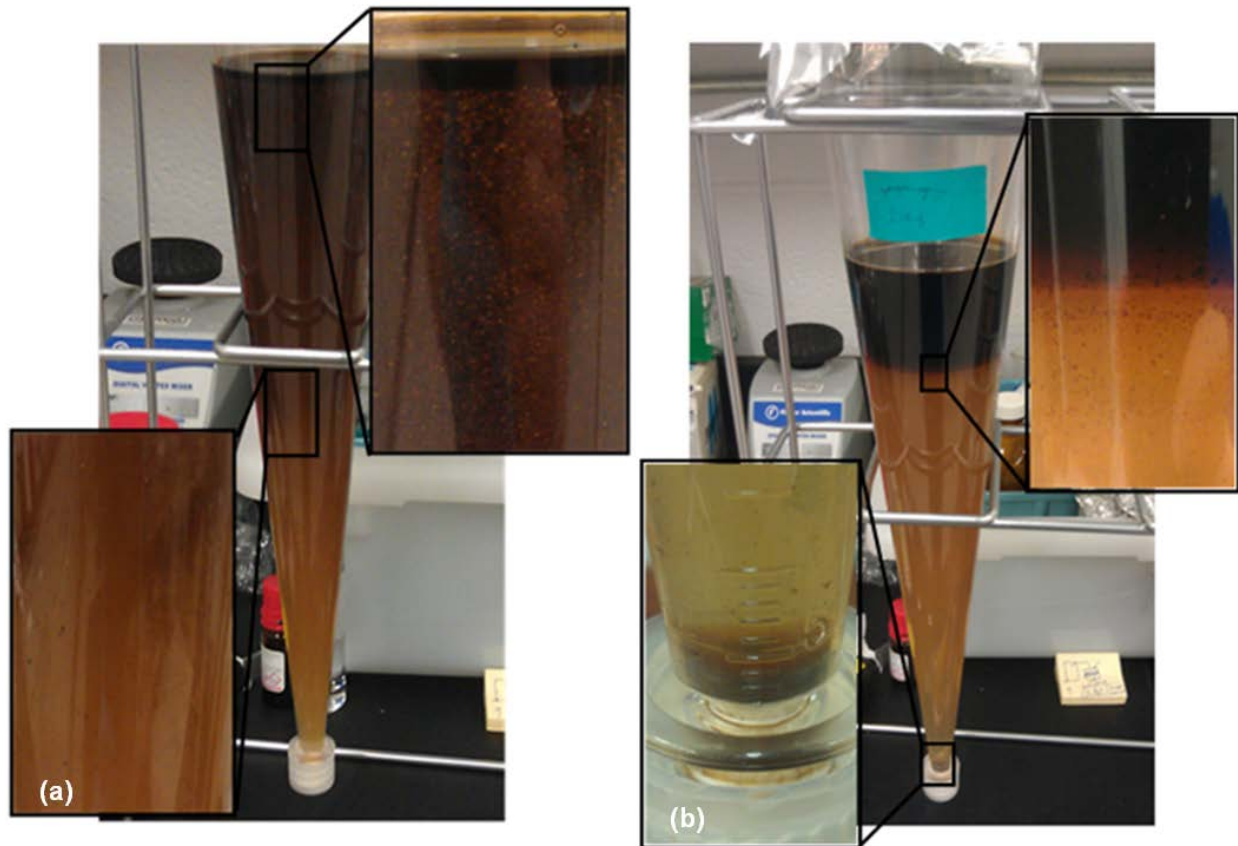


Figure A-6. 75% Citrus King, 25% water after being allowed to settle for 3 months. The solution was agitated (a) and then allowed to sit for 24 hours (b) to see if the solution would remain separated.

Table A-3. Results of cleaning tests of the recycled 75% Citrus King 25% water solution

Mass of Added Tar (g)	Tar Removed (g)	% Tar Removed	Cleaning Time (h:mm)
0.3401	0.3410	100.27%	1:15
0.4553	0.4562	100.20%	1:15
0.3850	0.3864	100.37%	1:15
Average Percent removed: 100.28%			

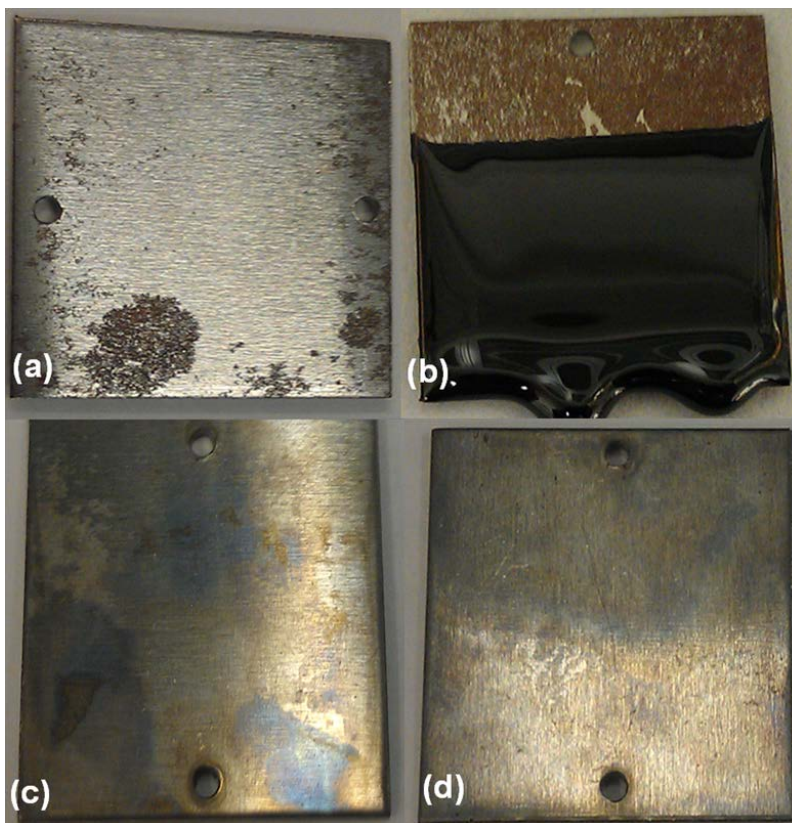


Figure A-7. Results of cleaning tests using the recycled solvent where (a) is an unused metal coupon, (b) is a tar coated coupon and (c)-(d) are cleaned metal coupons using recycled 75% Citrus King 25% water solution for 1 hr, 15 minutes at 298 K.

The recycled solution was tested for its ability to clean freshly prepared coupons. The solvent was able to fully clean the metal coupons within 1 hr, 15 minutes. The solvent was able to remove all of the tar and rust from the surface of the metal coupon, leaving it shiny and smooth (Figure A-7).

Conclusion

This study demonstrated the ability to recycle solvents from tar-solvent emulsions. The cleaning ability of the recycled solvents was then measured by quantitative cleaning ability.

Based on these preliminary findings, three recommendations can be made regarding the use of solvent that is initially mixed with water:

1. Use Brij S 100 up to a 5% by weight solution to incite separation.
2. Use Tropaeolin up to a 5% by weight solution to incite separation.
3. Allow the emulsion to rest for an extended period of time, approximately 3 months, such that it separates on its own

It is recommended that the initial solvent be mixed with water; otherwise, the tar-solvent emulsion will not separate. Allowing the solvent to rest, while being a very slow process, yields the most recycled solvent. If a faster separation is require, Brij S 100 yields the most reusable solvent in the shortest period of time.

Appendix B

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Appendix C

ACRONYMS AND ABBREVIATIONS

<u>Term</u>	<u>Definition</u>
APAI	Asphalt Paving Association of Iowa
AR	Army Regulation
ASTM	American Society for Testing and Materials
CDRL	Contract Data Requirements List
CEERD	U.S. Army Corps of Engineers, Engineer Research and Development Center
CERL	Construction Engineering Research Laboratory
CONUS	Continental United States
DA	Department of the Army
DEQ	Department of Environmental Quality
DoD	U.S. Department of Defense
EPA	Environmental Protection Agency
ERDC	Engineer Research and Development Center
HAP	Hazardous Air Pollutant
HEM	N-Hexane Extractable Material
HQUSACE	Headquarters, U.S. Army Corps of Engineers
MSDS	Material Safety Data Sheet
NAVAIR	U.S. Navy Naval Air Systems Command
NEWMOA	Northeast Waste Management Official's Association
NIC	National Information Center
ODS	Ozone-Depleting Substance
OPS	Operations
POC	Point of Contact
PWTB	Public Works Technical Bulletin
RRAD	Red River Army Depot
SGT	Silica Gel Treated
U.S.	United States
USEPA	U.S. Environmental Protection Agency
VOC	volatile organic compound
WAPA	Washington Asphalt Pavement
WHO	World Health Organization
WMPP	Waste Minimization and Pollution Prevention
WRI	Western Research Institute

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