Final Progress Report

1. DOE Award # DE-FG02-06ER84584

Vertec Biosolvents, Inc.

2. BIOSOLVENTS FOR COATINGS, RESINS AND BIOBASED MATERIALS

P.I./ Project Director: Dr. Rathin Datta

3. Date: August 2009

Executive Summary

The project has substantially achieved and in some aspects exceeded its goals and objectives. The details are provided in the following sections.

The primary accomplishments:

With close collaboration with several industrial coatings manufacturers several solvent blends were developed tested and optimized.

These were then piloted in the commercial company's reactors and systems.

Three were successfully tested in commercial applications and two of these - Methotate replacement and a specialty ketone replacement were sold in commercial quantities in 2009. Further sales are anticipated in 2010 and the following years.

One patent application was developed and submitted to the US patent office.

The laboratory refurbishment and equipment and personnel costs were kept within the budget.

4. Comparison of Actual Accomplishments with Goals and Objectives

The project has substantially achieved and in some aspects exceeded its goals and objectives. Details provided below.

1. Optimize the biosolvent blends with three specific types of polymers of the commercial partners in the bench scale.

This task has been completed and actually exceeded with very good results. Details provided in section 5.

2. Refurbish laboratory space and procure and install two small reactors with appropriate agitation, heating, cooling and exhaust handling capability.

The laboratory space remodeling has been accomplished. This included all the contractual work that included architectural, mechanical, electrical work. The Downers Grove authorities approved all the permitting and the final inspection. This laboratory is now fully functional for the bench scale formulation, tests and making batches. The two small reactors will be procured and installed later. Vertec's industrial supporters (Akzo and Hexion) had the appropriate reactors to make the polymer batches according to their specifications. Hence the test batches were made by them and provided to Vertec. This enabled fast progress of the key Task 1.

3. Optimize the processes in these reactors and provide adequate samples for tests.

Fast progress in this task was made because Vertec's industrial supporters (Akzo and Hexion) had the appropriate reactors to make the polymer batches according to their specifications. Hence the test batches were made by them and provided to Vertec. Further optimization for one of the specialty acrylate polymer was also accomplished.

4. In conjunction with the commercial partners, conduct short as well as longer term tests as specified by various protocols.

Short-term tests for the specialty acrylate polymer are underway at Akzo. The initial results are encouraging (see detail in section 5). Longer-term tests are conducted and will be continued by the commercial companies.

5. Select the top two candidates for potential commercialization and make several pilot-scale runs in conjunction with the commercial partners.

Three pilot scale runs were conducted with solvent replacement blends for methotate, isophorone and acrylate polymers with the commercial partners with the solvent blends that were developed and optimized by Vertec. Some of the properties and comparative test data are summarized in Appendix 1.

6. Complete several pre-commercial demonstration runs in conjunction with the commercial partners and establish a commercial supply relationship.

This was successfully completed for two replacement blends for methotate and a ketone (isophorone) and initial commercial sales began in 2009 with expected continuation for 2010.

7. Continue with the development of new applications of the low odor biosolvent blends that were serendipitously discovered.

Excellent progress has been made. Several solvents that are considered toxic and HAP (hazardous air pollutant) such as methyl ethyl ketone (MEK), methyl isobutyl ketone (MIBK), Xylenes and Toluene and other specialty solvents have been targeted for replacement applications in formulations. We have developed physical and chemical models for matching key properties of these solvents with our bio-solvent blends and laboratory tests for estimating their polymer dissolution properties. Based on these several application data sheets have been prepared and several major industrial companies are testing these blends for replacement of these specific solvents in their formulations. The initial results are very encouraging. Some of the technical details are provided in section 5 and the Appendix 1.

5. Discussion of Accomplishments

Summary of technical review meeting of the project is attached below. Some of the proprietary information has not been included. This shows more of the details in progress of Tasks 1, 2 and 3.

Slide 1. BIOSOLVENTS FOR COATINGS

Akzo - Vertec- Hexion
Meeting 4/9/08
Downers Grove, IL

Slide 2. Polymer Diluents – Viscosity and Phase Behavior

- Four polymers received from Hexion (Feb 2008)
 - Acrylate (Akzo 2A176)
 - Three polyesters – Polymac 220-1935; 220-1959; 66-6613
- Scoped potential biosolvent blends – potential properties, ready availability, economics etc.
 - Viscosity, solubility parameters, evaporation rates etc.
 - Readily available biosolvents (*or those that can be made from bioderived feedstocks –if in large demand*)
 - Manufacturing processes not too complicated

Slide 3. Polymer Diluents – Viscosity and Phase Behavior ^{contd.} Comparative Solvents

- Petroleum Solvents – MIBK, Xylenes, Butyl Cellosolve, AR-150
- Biosolvents – Ethyl lactate, Ethanol with n-Hexanol and n-Butyl Acetate

- Developed and conducted comparative tests
 - Single solvents and blends
 - Viscosity – Brookfield, done in-house
 - Phase behavior – RT, 40C, 4C and –20C

Slide 4. Other Properties

- **Phase Behavior –**
 - Four temperatures – 40C, RT, 4C and –20C
 - No differences observed between bio- and petro- blends
- **Paint formulation tests**
 - Results from Phase 1 project were very good
 - Similar positive results are expected for these biosolvents

Slide 5. Summary

- **Very Positive Results**
 - Viscosity reduction – polyacrylates:
 - all of the biosolvents and blends give as good or better results
 - Viscosity reduction – polyesters:
 - Much better than the typical aromatics – Xylene or AR-150
 - Viscosity reduction feasible with lower amount of biosolvent usage
 - Phase Behavior – Did not see any difference
- **Paint formulation tests**
 - Results from Phase 1 project were very good
 - Similar positive results are expected for these biosolvents

For Task 7, we have developed physical and chemical models for matching key properties of these solvents with our bio-solvent blends and laboratory tests for estimating their polymer dissolution properties. Several major industrial companies are testing these blends for replacement of these specific solvents in their formulations. The initial results are very encouraging as can be seen from a recent comment sent by a major industrial coatings company, which is now proceeding to pilot scale tests.

May 2, 2008

Jim and Kevin,

I have excellent results to report regarding the use of ELSOL XTR and ELSOL KTR1 in our "green" primer formulation. The solubility was excellent, and we were able to achieve the exact spray characteristics and drying rate that we need by using a 50:50 blend of the two solvents.

My question is this. Could you supply a single solvent to us that represents a 50:50 blend of the XTR and KTR1? If so, I would like to obtain a 5 gallon quantity so we can get some material out for field trials ASAP. If not, could you supply a couple of gallons each of the XTR and KTR1 so we can make up our own blend.

We are also evaluating an acrylic coating based on these solvents, and will probably wind up with a 50:50 blend for this as well. Great job on coming up with these formulations! Looks like they will provide just what we were looking for!

Thanks, and Best Regards,

Gordon

6. Cost Status

The costs are within budget. See details in attached Financial Status Report SF-269.

7. Schedule Status

The performance schedule from the project narrative and management plan is shown below.

The project has substantially achieved and in some aspects exceeded its tasks. Details have been provided in section 4 above.

8. Any Changes in Approach or Aims

None to date. One positive outcome of the industrial collaboration with Akzo and Hexion is that the need for the reactors has been reduced. Thus the capital for the reactors is usefully deployed to other project costs.

Furthermore, two reactor vessels with distillation stills were available at the Argonne National Laboratory and these were provided to Vertec on loan to be used in its laboratory for solvent formulation and reaction tests. Hence the cost of the purchase of reactors was usefully deployed for other project costs.

9. Actual or Anticipated problems

None anticipated.

10. Absence or Changes in Key Personnel

Mr. Kevin Monti was hired to join this project in December, 2007. He has over 10 years of experience in the coatings industry. The PI and other key personnel remain the same.

11.. Products or Technology Transfer:

- A. Publications – none
- B. Website – provides information on ELSOL products that came out as a result of the proprietary solvent blends developed under this grant www.vertecbiosolvents.com
- C. Networks Collaborators – Beyond AKZO and Hexion there are several industrial supporters that are testing the biosolvents in their formulations. One of them, Becker Specialty Chemicals of Elk Grove, Illinois has moved forward with commercial testing and some initial purchase.
- D. Technologies/Techniques – summarized above in sections 4 and 5
- E. Patent Applications – “Composition of Lactate Esters with Alcohols with Low Odor and Enhanced Performance” U.S. Patent Application # 11/781,450, filed July 23, 2007.
- F. Other Products –See Appendix 1

- G. Initial commercialization of two of the solvent blends has been successful and has led to sales in 2009 and is anticipated for increased sales in 2010. The Matrix for Jobs and Revenue data requested by the DOE SBIR project manager has been provided and is attached in Appendix 2.

APPENDIX 1

SOLVENT REPLACEMENT PROPERTY AND DATA SHEETS

VertecBio ELSOL™ - KTR1

Patent pending blend of biobased ethyl lactate and ethyl alcohol

- High Solvating Power of [Ethyl Lactate Without the Odor](#)
 - Greatly Increased Solvating Power vs. Ethyl Alcohol (Ethanol) Alone
 - Dries Three Times Faster Than Ethyl Lactate Alone
 - Ideal Wipe Solvent – [100% Biobased Replacement for MEK](#)
 - Better Cleaning Power and Reduced Solvent Usage vs. MEK
 - Precision Cleaning Solvent, Dries Completely and Leaves No Residue
 - High Performance, Versatile Solvent Blend for Formulations
 - Excellent Solvency for Paint, Coating and Ink Formulations
 - Moderate Vapor Pressure and High Loading Capacity
 - 100% Biodegradable to Carbon Dioxide and Water
 - Sustainable Chemistry – No Impact on Global Warming (“Carbon Neutral”)
 - U.S. EPA Approved 4B Inert Solvent for Agricultural Formulations
 - Custom Blends Available for Maximum Performance
-
- EPA Approved SNAP Solvent – No Ozone Depleting Chemicals
 - No Environmentally Hazardous Ingredients

GENERAL PHYSICAL PROPERTIES **VERTECBIO ELSOL-EtOH 50**

Flash Point ..59 F ASTM D3278 (Setaflas)
Vapor Pressure.....44 mmHg @ 68
PH of Water Dispersion.....
Specific Gravity.....0.7

Vertec BioSolvents ELSOL™-KTR1 Performance Sheet

Suggested Replacement for MEK

Introduction

Methyl ethyl ketone's largest use is in industrial coatings applications and industrial cleaning and surface preparation applications. For coatings formulators looking to reduce their use of hazardous petroleum solvents, Vertec BioSolvents KTR1 blend is an ideal replacement for MEK in a wide array of coatings formulations and cleaning applications.

Vertec BioSolvents suggests a replacement blend with renewable, carbon neutral biobased solvents. These biobased solvents are derived from corn, soybeans, citrus fruits and other renewable feedstocks, and have a reduced toxicity profile.

Reformulation Solvents

Hazardous Solvents	MEK
BioBased Replacements	ELSOL-KTR1

The solvent blends shown are only suggested starting points for developing alternative systems. All blends should be thoroughly evaluated to determine suitability for specific applications

	Relative	Flash	Hansen Solubility Parameters			Total
	Evaporation Rate	Point(°F)	Dispersion	Polar	H Bonding	
	~3	~30	7.7	3.4	6.1	10.4
	4.03	26	7.8	4.4	2.5	9.3

Reformulation Solvents Typical Properties

Environmental	Uses	Comments
Non Hap	Paint, coatings	Renewable, carbon neutral
Non SARA reportable	cleaners, adhesives	Reduced toxicity profile

Conclusion

Formulators and applicators are looking for sustainable technologies that offer alternatives to hazardous solvents. This can be seen in many industries including paint and coatings, adhesives and inks. Formulators are not only feeling the pressures at the federal and state level, but abroad as well. The suggested replacements using the sustainable, carbon neutral, biobased

Vertec BioSolvents ELSOL™-KTR2 (Experimental A&B) Performance Sheet

Suggested Replacement for MIBK

Introduction

MIBK's largest use is in industrial coatings application, industrial cleaning and surface preparation applications. For coatings formulators looking to replace HAPS (Hazardous Air Pollutant Solvents) with a biobased, sustainable, carbon neutral HAPS free alternative, Vertec BioSolvents **KTR2** blends are an ideal replacement for MIBK in a wide array of coatings formulations.

Vertec BioSolvents suggests a replacement blend with renewable, carbon neutral biobased solvents. These biobased solvents are derived from corn, soybeans, citrus fruits and other renewable feedstocks, and have a reduced toxicity profile.

Reformulation Solvents

Hazardous Solvents	MIBK
BioBased Replacements	ELSOL-KTR2A or KTR2B

The solvent blends shown are only suggested starting points for developing alternative systems. All blends should be thoroughly evaluated to determine suitability for specific applications

Relative Evaporation Rate	Flash Point (°F)	Hansen Solubility Parameters			
		Dispersion	Polar	H Bonding	Total
2.4	~78	7.7	3.4	5.7	10.4
3	~60	7.7	3.3	5.7	10.4
1.7	56	7.5	3.0	2.0	8.3

Reformulation Solvents Typical Properties

Environmental	Uses	Comments
Non Hap	Paint, coatings	Renewable, carbon neutral
Non SARA reportable	cleaners, adhesives	Reduced toxicity profile

Conclusion

Formulators and applicators are looking for sustainable technologies that offer an alternative to hazardous solvents. This can be seen in many industries including paint and coatings, adhesives and inks. Formulators are not only feeling the pressures at the federal and state level, but abroad as well. The suggested replacements using the sustainable, carbon neutral,

Vertec BioSolvents ELSOL™-XTR Performance Sheet

Suggested Replacement for Toluene and Xylene

Introduction

Toluene's and xylene's largest uses are in industrial coatings applications, primarily in coil, extrusion, wood furniture and fixtures, containers and closures, automotive finishes and machinery. Formulators have been seeking an alternative to these hazardous solvents due to employee exposure concerns and increased legislative pressure geared toward reducing the use of these solvents.

Vertec BioSolvents suggests a replacement blend with renewable, carbon neutral biobased solvents. These biobased solvents are derived from corn, soybeans, citrus fruits and other renewable feedstocks, and have reduced toxicity profiles.

Reformulation Solvents

Hazardous Solvents	Toluene, Xylene
BioBased Replacement	ELSOL-XTR

The solvent blend shown is only a suggested starting point for developing alternative systems. All blends should be thoroughly evaluated to determine suitability for specific applications

Relative Evaporation Rate	Flash Point(°F)	Hansen Solubility Parameters			
		Dispersion	Polar	H Bonding	Total
~1	~68	7.7	2.9	5.4	9.8
2	40	8.8	0.7	1	8.9
0.77	85	9.8	0.9	1.2	9.9

Reformulation Solvents Typical Properties

Environmental	Uses	Comments
Non Hap	Paint, coatings	Renewable, carbon neutral
Non SARA reportable	cleaners, adhesives	Reduced toxicity profile

Conclusion

Formulators and applicators are looking for alternatives to hazardous solvents as more regulations are enforced. This can be seen in many industries including paint and coatings, adhesives and inks. Formulators are not only feeling the pressures at the federal and state level, but abroad as well. The suggested replacements using the

Vertec BioSolvents ELSOL™ -MTR5 Performance Sheet

Suggested Replacement for Methotate®

Introduction

Methotate's largest use is in industrial coatings applications and industrial cleaning and surface preparation applications. For coatings formulators looking to replace petroleum based solvents with a biobased, sustainable, carbon neutral alternative, Vertec BioSolvents blend is an ideal replacement for Methotate in a wide array of coatings formulations and cleaning applications.

Vertec BioSolvents suggests a replacement blend with renewable, carbon neutral biobased solvents. These biobased solvents are derived from corn, soybeans, citrus fruits and other renewable feedstocks, and have a reduced toxicity profile.

Reformulation Solvents

Hazardous Solvents	Methotate
BioBased Replacements	ELSOL-MTR5

The solvent blends shown are only suggested starting points for developing alternative systems. All blends should be thoroughly evaluated to determine suitability for specific applications

Relative Evaporation Rate	Flash Point(°F)	Hansen Solubility Parameters Dispersion	Polar	H Bonding	Total
0.24	106	7.8	3.7	6.3	10.7
0.19	133	7.4	2.3	4.7	9.1

Reformulation Solvents Typical Properties

Environmental	Uses	Comments
Non Hap	Paint, coatings	Renewable, carbon neutral
Non SARA reportable	cleaners, adhesives	Reduced toxicity profile

Conclusion

Formulators and applicators are looking for alternatives to hazardous solvents as more regulations are enforced. This can be seen in many industries including paint and coatings, adhesives and inks. Formulators are not only feeling the pressures at the federal and state level, but abroad as

APPENDIX 2

SBIR Jobs and Revenue Data Request

SBIR Jobs and Revenue Data Request Form			
Question			
1	Company Name	Vertec Biosolvents, Inc	
2	Year Established	2001	
3	Year of Company's First SBIR Award	2006 Phase 1, 2007-09 Phase2	
4	Number of Employees in the Year of Company's First SBIR Award	4 FTE	
5	Number of Employees Today	6 FTE	
6	Annual Revenue 2009 (\$)*	\$1.5 MM	
<i>* 2009 was a recession year + but the SBIR project enabled commercial development which has led to major growth of sales in the "Biosolvents for Coatings"</i>			
Annual Revenue 2010 (\$) estimated		\$3.5 MM	