PARTRAC

US Navy - ESTCP

Project Number: ER-201214

Demonstration of fluorescent magnetic particles for linking sources to sediments at DoD sites

Partrac Tracer Material 'White Paper'

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DOCUMENT CONTROL

Version History					
Version	Date	Prepared by	Reviewed by	Approved by	Approved as
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n/a	Original version
V02	Inclusion of ecotoxicology test data

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1. INTRODUCTION

1.1 Background

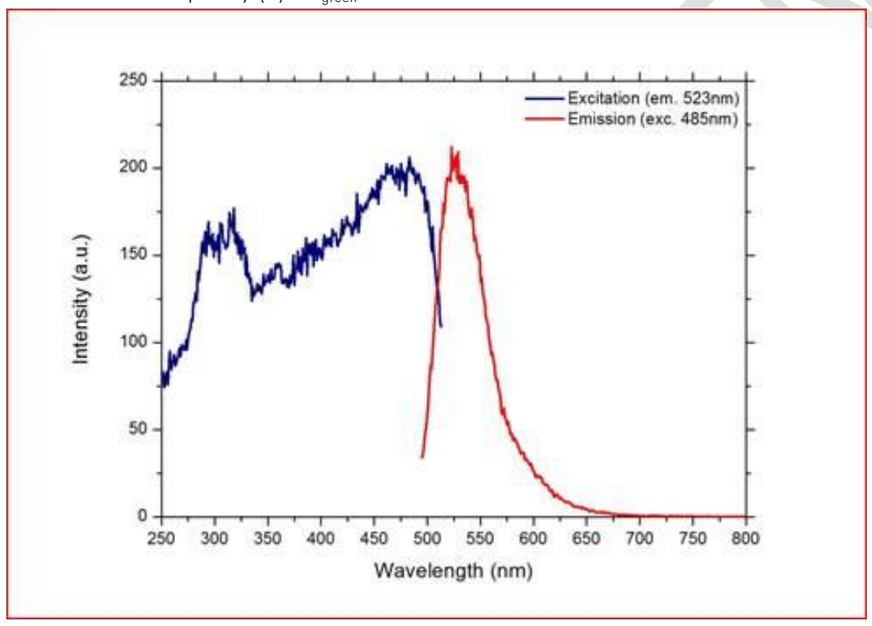
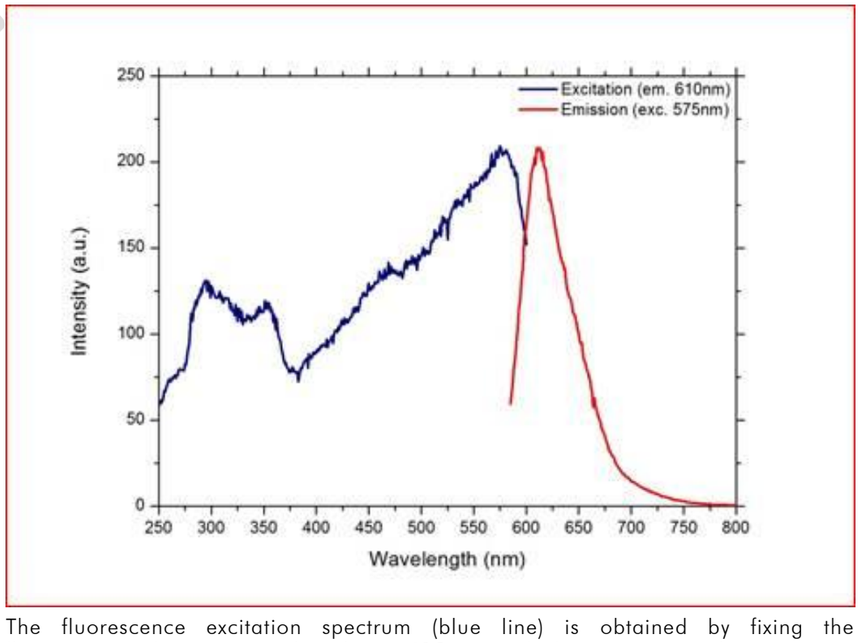
Within the Action Items that resulted following the November 2012 SERDP/ESTCP In-Progress Review of the ESTCP funded project 'Demonstration of Fluorescent Magnetic Particles for Linking Sources to Sediments at DoD Sites' (Project Number: ER-201214) Action Item 3 was a request to provide ESTCP with white paper that discusses the toxicity and biodegradation pathways of the dyes used in the demonstration. In addition, provide the MS/DS for the dye compounds that have some estimate of toxicity to aquatic organisms. Finally, provide an estimate of the dye mass at the initial point of release.

The following document has been compiled in response to the Action item request and contains the all of the toxicological information that is available for the tracer material.

2. MANUFACTURED TRACER MATERIAL SPECIFICATIONS

Table 1 displays the average specifications of the manufactured tracer material expected to be deployed during the ESTCP Study

Table 1 Actual specification of manufactured tracer material

Specific metrics	Green material	Red material
Particle Labeling	Fluorescent color (green) and magnetic signature	Fluorescent color (red) and magnetic signature
Quantity used in Study (kg)	To be determined in the Demonstration plan development	To be determined in the Demonstration plan development
Particle Size (d_{50})	0.04-0.05 mm	0.04-0.05 mm
Constituents (% of total)	Natural Sand/Silt grains (silicon dioxide, or SiO_2) = 80% Dye Pigment (8%) Magnetite (5%) Polyester Resin Binding/Coating agent (7%)	Natural Sand/Silt grains (silicon dioxide, or SiO_2) = 80% Dye Pigment (8%) Magnetite (5%) Polyester Resin Binding/Coating agent (7%)
Specific Gravity	To be determined expected to be ~1500 kg m^{-3}	To be determined expected to be ~1500 kg m^{-3}
Bulk Density (kg m^{-3}) dry weight	To be determined expected to be ~2600 kg m-3	To be determined expected to be ~2600 kg m^{-3}
Spectral properties of fluorescent dye	Shade: Brilliant BSR-CH227 Chartreuse Peak emission frequency (λ)is λ_{green}=~530 nm  The fluorescence excitation spectrum (blue line) is obtained by fixing the fluorescence detector wavelength at 523nm and then scanning the excitation wavelengths. Inversely, the fluorescence emission spectrum (red line) was obtained by fixing the excitation wavelength at 485nm and then scanning the emission wavelengths.	Shade: Brilliant BSR-RD213 Red Peak emission frequency (λ)is λ_{red}=~610 nm  The fluorescence excitation spectrum (blue line) is obtained by fixing the fluorescence detector wavelength at 610nm and then scanning the excitation wavelengths. Inversely, the fluorescence emission spectrum (red line) was obtained by fixing the excitation wavelength at 575nm and then scanning the emission wavelengths.

3. SAFETY PROCEDURES FOR MATERIAL USE AND TRACER EMPLACEMENT

3.1 Material Safety

The material that is manufactured and is natural quartz sand and/or silt that has had a fine layer of a magnetic and fluorescent coloured pigment coating applied to each of the individual particles using a polyester resin binding agent. The material and coatings are non-toxic and benign when placed into an aquatic environment such as intended for the present study.

3.1.1 Sand / Silt

The tracer product contains 80% by volume of naturally occurring sand and/or silt (dependent on the size distribution of the material required). The sand/silt is predominantly composed of naturally occurring silicon dioxide (SiO_2). The sand/silt is inert and non-toxic.

3.1.2 Dye pigments

The tracer product contains 8% by volume of fluorescent dye pigment that is either Brilliant BSR-CH227 Chartreuse Green or Brilliant BSR-RD213 Red.

Brilliant™ the manufacturers of the dye pigments indicate within their technical literature for the BSR fluorescent coloured pigments used in the tracer manufacture that toxicity tests conducted through independent laboratories have found Brilliant Group BSR Series Fluorescent Pigments to be "essentially non-toxic." The literature describes that the pigments have the following specifications:

- insoluble in water,
- 100% solid by weight
- 3 to 5 microns average particle size
- Non Hazardous, and as such they contain no reportable hazardous ingredients.

The Material Safety Data Sheet (MSDS), produced by Brilliant™ for the dye pigments in June 2012, supplied by the manufacturer Brilliant shows that with regard to regulatory information the following apply:

U.S. FEDERAL REGULATIONS

TSCA (TOXIC SUBSTANCE CONTROL ACT): ALL COMPONENTS OF THIS PRODUCT ARE INCLUDED ON THE TSCA INVENTORY IN COMPLIANCE WITH THE TOXIC SUBSTANCES CONTROL ACT, 15 U.S.C. 2601 ET. SEQ.

CERCLA (COMPREHENSIVE ENVIRONMENTAL RESPONSE COMPENSATION, AND LIABILITY ACT): THIS IS NOT A REGULATED MATERIAL UNDER 40 CFR 117.302. NOTIFICATION OF SPILLS IS NOT REQUIRED.

SARA TITLE III (SUPERFUND AMENDMENTS AND REAUTHORIZATION ACT): THIS PRODUCT CONTAINS NO KNOWN CHEMICALS CONTAINED ON THE LIST OF TOXIC CHEMICALS SUBJECT TO THE REPORTING REQUIREMENTS OF SECTION 313 OF THE EMERGENCY PLANNING & COMMUNITY RIGHT TO KNOW ACT OF 1986 & OF 40 CFR372.

311/312 HAZARD CATEGORIES: NONE KNOWN

313 REPORTABLE INGREDIENTS: NONE KNOWN

STATE REGULATIONS: CALIFORNIA PROPOSITION 65: WARNING: THIS PRODUCT CONTAINS A CHEMICAL KNOWN TO THE STATE OF CALIFORNIA TO CAUSE CANCER.

INTERNATIONAL REGULATIONS:

CANADA DSL: ALL COMPONENTS OF THIS PRODUCT ARE INCLUDED ON THE DOMESTIC SUBSTANCES LIST.

EEC EINECS: ALL COMPONENTS OF THIS PRODUCT ARE INCLUDED ON THE EUROPEAN INVENTORY OF EXISTING CHEMICAL SUBSTANCES (EINECS) IN COMPLIANCE WITH COUNCIL DIRECTIVE 67/548/EEC AND ITS AMENDMENTS.

A copy of the Material Safety Data Sheet (MSDS) are attached to this document.

3.1.3 Magnetite

The tracer product contains 5% by volume of naturally occurring magnetite. It is sourced from Minelco and won from Minelco group mines are located in Kiruna and Malmberget in the northern part of Sweden.

Magnetite, Fe_3O_4, is a black ferri-magnetic naturally occurring iron oxide and a member of the spinel group. Magnetite is inert and non-toxic.

3.1.4 Polyester Resin Binding/Coating Agent

The tracer product contains 7% by volume of a polyester resin that is used as a binding agent for the magnetite and dye pigment for coating of the individual sand/silt particles. This polyester material is the same as found within standard industrial water purification systems.

4. DYE MASS AT THE INITIAL POINT OF RELEASE

As is apparent from Table 1 the mass of the dye pigment at the initial point of release will be 8% of the total mass of the amount of tracer material released. For example in 100 kilogrammes (kg) of tracer material released the dye mass would be 8 kg. For comparison to the toxicity results in the next section, if 100 kg of tracer material is released from an outfall, a concentration of 0.1 milligram/Liter (mg/L) would be expected in a well mixed box of 10 meters per side (a volume of 1 million Liters). If the tracer is not immediately homogenously mixed it might be expected to be at higher concentrations so the toxicity tests were run over a range of concentrations from 0.1 up to 10 mg/L.

5. TOXICITY TESTING RESULTS

Toxicity testing was initiated following concerns about potential toxicity issues related to the typical use of the tracer product. Standard EPA elutriate testing was done with adult mysid shrimp (mortality endpoint) and echinoderm and mussel embryos (normal development endpoints). The tracer material was added to an excess of clean fresh water to "wet" the tracer material to ensure complete transfer of the tracer into liter beakers to produce a slurry. The excess fresh water was decanted off and the remaining slurry was brought up to volume using filtered San Diego Bay salt water (approximately 30 parts per thousand salt). These tests used a range of tracer concentrations that would be expected to be present in typical tracer experiments (0.1, 1.0, and 10 mg tracer per Liter of seawater). For elutriate tests, the different concentrations of tracer were kept agitated in seawater on a shaker table for 30 minutes and then allowed to settle for 1 hour. Samples of the overlying seawater were then siphoned off, with the samples split into filtered and unfiltered splits to look for any effects of fine particles that might remain in the overlying water. Results are presented in Tables 1 – 3 below.

Results for all concentration ranges show no statistical differences from seawater controls. For the more sensitive tests with embryo development, both filtered and unfiltered samples were run to look for any differences. There are reports that fine particles remaining in suspension in samples after 1 hour of settling might cause artifacts leading to less normal development with the echinoderm development test. But in the case of both the echinoderm and mussel larval development tests run here, the filtered and unfiltered samples showed no differences.

Table 1 Experimental ecotoxicological data (standard EPA elutriate testing) for tests on echinoderms.

Treatment ID		Rep	Number Counted	Number Normal	% Normal	Mean % Normal	p-Value
Echinoderm Development Test (10-11/29/13)							
Filtered	1 Control	A	100	97	97	97.5	-
		B	100	94	94		
		C	100	99	99		
		D	100	100	100		
	2 (0.1mg/kg)	A	100	92	92	96.3	0.591
		B	100	98	98		
		C	100	95	95		
		D	100	100	100		
	3 (1mg/kg)	A	100	99	99	98.5	0.531
		B	100	100	100		
		C	100	98	98		
		D	100	97	97		
	4 (10mg/kg)	A	100	94	94	95.5	0.240
		B	100	96	96		
		C	100	95	95		
		D	100	97	97		
Echinoderm Development Test (10-11/29/13)							
Un-filtered	1 Control	A	100	98	98	99.0	-
		B	100	99	99		
		C	100	100	100		
		D	100	99	99		
	2 (0.1mg/kg)	A	100	96	96	98.0	0.329
		B	100	100	100		
		C	100	98	98		
		D	100	98	98		
	3 (1mg/kg)	A	100	95	95	97.3	0.133
		B	100	99	99		
		C	100	97	97		
		D	100	98	98		
	4 (10mg/kg)	A	100	98	98	98.8	0.624
		B	100	99	99		
		C	100	99	99		
		D	100	99	99		

Table 2 Experimental ecotoxicological data (standard EPA elutriate testing) for tests on mysids.

Treatment ID	Number Exposed	96 Hour Survival	% Survival	Mean % Survival
Mysid 96-h Acute Survival Test (06/19/13)				
1	5	5	100	100
	5	5	100	
2	5	5	100	100
	5	5	100	
3	5	5	100	100
	5	5	100	
4	5	5	100	100
	5	5	100	

Table 3 Experimental ecotoxicological data (standard EPA elutriate testing) for tests on bivalves.

Treatment ID		Rep	Number Counted	Number Normal	Number Abnormal	Adjusted # (if #norm > initial# of larvae)	% Normal	Mean % Normal	p-Value	% Comb norm	Mean % Norm Alive	p-value
Bivalve Survival & Development Test (10/29/2013)												
Filtered	1	A	208	165	43	165.0	79	78.9	-	76	73.4	-
		B	202	161	41	161.0	80			74		
		C	206	165	41	165.0	80			76		
		D	190	145	45	145.0	76			67		
	2	A	175	133	42	133.0	76	81.1	0.314	61	75.6	0.712
		B	216	177	39	177.0	82			82		
		C	194	163	31	163.0	84			75		
		D	221	182	39	182.0	82			84		
	3	A	197	166	31	166.0	84	85.0	0.002	77	77.0	0.206
		B	205	171	34	171.0	83			79		
		C	186	162	24	162.0	87			75		
		D	197	168	29	168.0	85			78		
	4	A	169	127	42	127.0	75	80.8	0.537	59	67.7	0.352
		B	169	132	37	132.0	78			61		
		C	199	175	24	175.0	88			81		
		D	185	152	33	152.0	82			70		

Un-filtered	1	A	194	162	32	162.0	84	85.2	-	75	74.5	-
		B	175	147	28	147.0	84			68		
		C	198	175	23	175.0	88			81		
		D	190	161	29	161.0	85			74		
	2	A	216	180	36	180.0	83	83.4	0.296	83	77.9	0.348
		B	204	164	40	164.0	80			76		
		C	204	172	32	172.0	84			79		
		D	186	159	27	159.0	85			73		
	3	A	214	185	29	185.0	86	84.0	0.437	85	76.1	0.723
		B	196	162	34	162.0	83			75		
		C	180	149	31	149.0	83			69		
		D	194	163	31	163.0	84			75		
	4	A	198	167	31	167.0	84	84.2	0.622	77	76.3	0.665
		B	214	172	42	172.0	80			79		
		C	174	146	28	146.0	84			67		
		D	200	176	24	176.0	88			81		

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