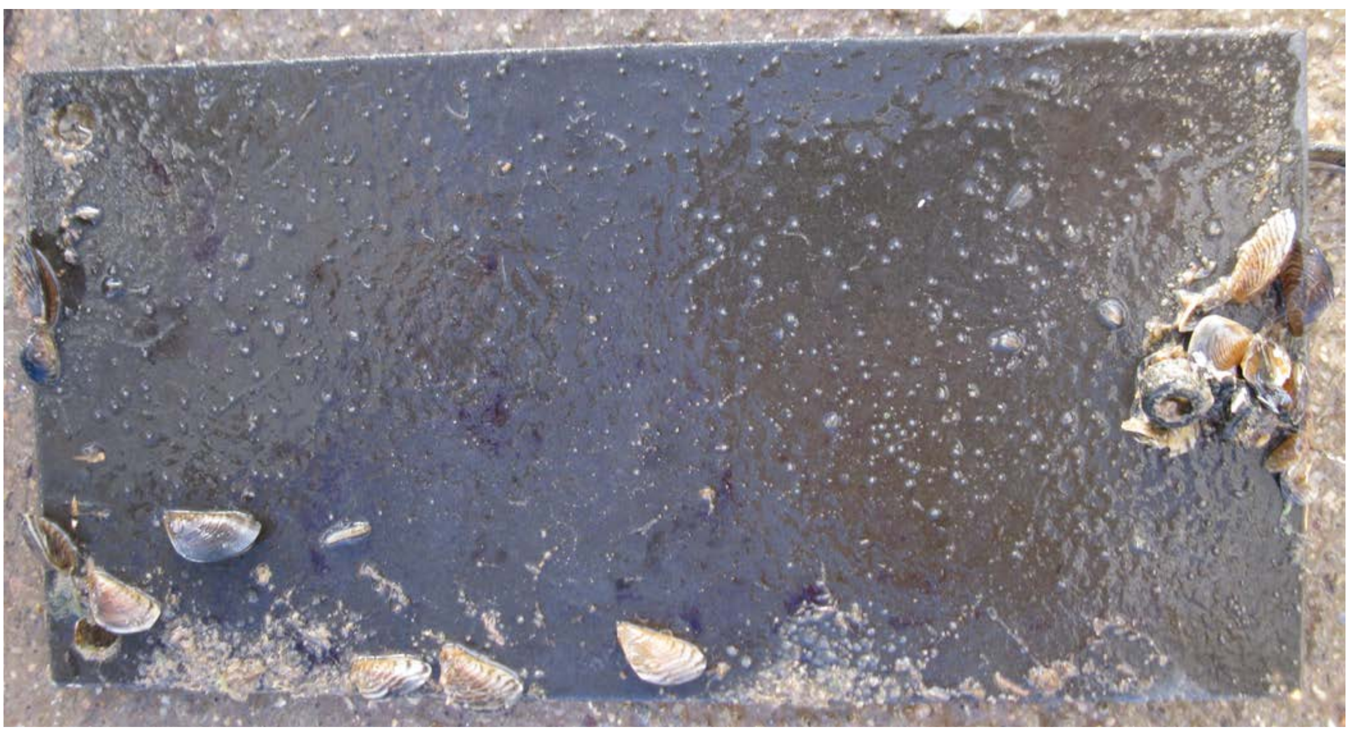
RECLAMATION

Managing Water in the West

Final Report ST-2016-0809-01
Technical Memorandum No. 8540-2016-02

Durable Silicone Foul Release Coatings CRADA – Final Report

Research and Development Office
Science and Technology Program



U.S. Department of the Interior
Bureau of Reclamation
Technical Service Center
Denver, Colorado

March 2016

MISSION STATEMENTS

The U.S. Department of the Interior protects America's natural resources and heritage, honors our cultures and tribal communities, and supplies the energy to power our future.

The mission of the Bureau of Reclamation is to manage, develop, and protect water and related resources in an environmentally and economically sound manner in the interest of the American public.

On the cover: Steel plate coated with an experimental foul release coating supplied by Fujifilm Hunt Smart Surfaces, LLC, after 12 months of immersion in mussel-infested water.

Disclaimer:

Information in this report may not be used for advertising or promotional purposes. The enclosed data and findings should not be construed as an endorsement of any product or firm by the Bureau of Reclamation (Reclamation), U.S. Department of the Interior, or the Federal Government. The products evaluated in this report were evaluated in environmental conditions and for purposes specific to Reclamation's mission. Most of these products were originally developed for the marine environment and not necessarily for use in freshwater. The data should be viewed as site specific and not necessarily applicable to all freshwater exposure conditions. Reclamation gives no warranties or guarantees, expressed or implied, for the products evaluated in this report, including merchantability or fitness for a particular purpose.

REPORT DOCUMENTATION PAGE	*Form Approved* *OMB No. 0704-0188*

The public reporting burden for this collection of information is estimated to average 1 hour per response, including the time for reviewing instructions, searching existing data sources, gathering and maintaining the data needed, and completing and reviewing the collection of information. Send comments regarding this burden estimate or any other aspect of this collection of information, including suggestions for reducing the burden, to Department of Defense, Washington Headquarters Services, Directorate for Information Operations and Reports (0704-0188), 1215 Jefferson Davis Highway, Suite 1204, Arlington, VA 22202-4302. Respondents should be aware that notwithstanding any other provision of law, no person shall be subject to any penalty for failing to comply with a collection of information if it does not display a currently valid OMB control number.

PLEASE DO NOT RETURN YOUR FORM TO THE ABOVE ADDRESS.

1. REPORT DATE *(DD-MM-YYYY)*	**2. REPORT TYPE**	**3. DATES COVERED** *(From - To)*
01/2016	Research	

4. TITLE AND SUBTITLE	
Durable Silicone Foul Release Coatings CRADA – Final Report	**5a. CONTRACT NUMBER** 16XR0680A1-RY1541ZQ201420809
	5b. GRANT NUMBER
	5c. PROGRAM ELEMENT NUMBER 1541 (S&T)
6. AUTHOR(S) Allen Skaja, Ph.D., Coatings Specialist Bureau of Reclamation, Technical Service Center Materials and Corrosion Laboratory askaja@usbr.gov; 303-445-2396	**5d. PROJECT NUMBER** 0809 **5e. TASK NUMBER** **5f. WORK UNIT NUMBER** 86-68540
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) Allen Skaja, Ph.D., Coatings Specialist Bureau of Reclamation, Technical Service Center Materials and Corrosion Laboratory	**8. PERFORMING ORGANIZATION REPORT NUMBER** 8540-2016-02
9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES) Research and Development Office U.S. Department of the Interior, Bureau of Reclamation PO Box 25007, Denver, CO 80225-0007	**10. SPONSOR/MONITOR'S ACRONYM(S)** R&D: Research and Development Office BOR/USBR: Bureau of Reclamation DOI: Department of the Interior **11. SPONSOR/MONITOR'S REPORT NUMBER(S)** ST-2015-0809-01

12. DISTRIBUTION/AVAILABILITY STATEMENT
Final report can be downloaded from the Bureau of Reclamation's Web site: https://www.usbr.gov/research/

13. SUPPLEMENTARY NOTE

14. ABSTRACT

The Bureau of Reclamation and Fujifilm Hunt Smart Surfaces, LLC, entered into Cooperative Research and Development Agreement (CRADA) 13-CR-8-1006 on March 5, 2014. The goal of the CRADA was to develop a durable foul release coating system that would resist damage and aquatic invasive mussel attachment. At the time, there were no durable foul release coatings commercially available with easy release properties for fouling control. Polydimethyl siloxane (PDMS) was the only polymer that provided nonfouling or easy release characteristics. This report explains the approaches taken under the CRADA and the test results.

15. SUBJECT TERMS
Invasive Mussel Control, Quagga Mussels, Zebra Mussels, Foul Release Coatings, Fouling Prevention

16. SECURITY CLASSIFICATION OF: Unclassified			**17. LIMITATION OF ABSTRACT**	**18. NUMBER OF PAGES**	**19a. NAME OF RESPONSIBLE PERSON** Allen Skaja
a. REPORT U	**b. ABSTRACT** U	**a. THIS PAGE** U	U	37	**19b. TELEPHONE NUMBER** *(Include area code)* 303-445-2125

Standard Form 298 (Rev. 8/98)
Prescribed by ANSI Std. Z39.18

Peer Review Documentation

Project and Document Information

Project Name:	Durable Silicone Foul Release Coatings CRADA	**WOID:**	Z0809
Document:	Durable Silicone Foul Release Coatings CRADA – Final Report		
Document Author(s):	Allen Skaja	**Document date:**	March 2016
Peer Reviewer (Reclamation):	David Tordonato, Bobbi Jo Merten		

Review Certification

Peer Reviewer: I have reviewed the assigned items/sections(s) noted for the above document and believe them to be in accordance with the project requirements, standards of the profession, and Bureau of Reclamation policy.

Reviewer: Dave Tordonato (Signature) Date reviewed: 3/24/2016

Reviewer: (Signature) Date reviewed: 4/18/2016

BUREAU OF RECLAMATION
Technical Service Center, Denver, Colorado
Materials and Corrosion Laboratory, 86-68540

Final Report ST- 2016-0809-01
Technical Memorandum No. 8540-2016-02

Durable Silicone Foul Release Coatings CRADA – Final Report

Allen D. Skaja	4-18-16
Prepared by: Allen D. Skaja, Ph.D., PCS Chemist, Materials and Corrosion Laboratory, 86-68540	Date
Dave Tordonato	4/18/2016
Checked by: David S. Tordonato, Ph.D., P.E. Materials Engineer, Materials and Corrosion Laboratory, 86-68540	Date
BJM	4/18/2016
Technical Approval by: Bobbi Jo Merten, Ph.D. Chemist, Materials and Corrosion Laboratory, 86-68540	Date
WFK	4/18/16
Peer Review: William F. Kepler, Ph.D., P.E. Manager, Materials and Corrosion Laboratory, 86-68540	Date

ACKNOWLEDGMENTS

We would like to thank the Bureau of Reclamation Research and Development Office's Science and Technology Program for providing funding for this research over the past 3 years. We would also like to thank the Bureau of Reclamation, Lower Colorado Region, Lower Colorado Dams Office, and the Parker Dam staff for all of their in-kind contributions, access, and support.

ACRONYMS AND ABBREVIATIONS

ASTM	American Society for Testing and Materials
CRADA	Cooperative Research and Development Agreement
Fuji	Fujifilm Hunt Smart Surfaces, LLC
g	gram(s)
g/mol	grams per mole
m^2/g	meters squared per gram
MTAS	methyl triacetoxy silane
PDMS	polydimethyl siloxane
pli	per liner inch
psi	pounds per square inch
Reclamation	Bureau of Reclamation

Symbols

>	greater than
%	percent
wt. %	weight percent

Contents

Page

Tables

Page

Figures

Page

EXECUTIVE SUMMARY

In an attempt to combat zebra and quagga mussels, a Cooperative Research and Development Agreement (CRADA) was initiated between the Bureau of Reclamation (Reclamation) and Fujifilm Hunt Smart Surfaces, LLC (Fuji). CRADA 13-CR-8-1006 was signed on March 5, 2014, with the goal of developing a durable foul release coating system that would resist damage and aquatic invasive mussel attachment.

On December 10, 2015, Fuji issued Reclamation a notification of CRADA discontinuance, stating that the company elected to discontinue its Smart Surfaces fouling release coatings business. Despite a shortened time period of performance, valuable information was gathered that advanced the goal of developing a durable foul release coating.

In one study, the CRADA partners investigated the thickness of silicone topcoat required to prevent fouling. Five different dry film thicknesses of Fuji's commercial foul release coating were applied, ranging from 18 to 97 microns. All thicknesses prevented fouling. The results of this study showed that a very thin film of silicone prevents mussel attachment.

Fuji's initial formulations were an attempt to improve coating durability by reinforcing the silicone polymer with greater volumes of fumed silica. The fumed silica significantly increased the viscosity, necessitating additional solvent to produce viscosities for spray application. Fuji then experimented with the reinforcing filler, Celtix, in an effort to maintain the solution's viscosity. Following preliminary screening, Fuji provided Reclamation with four experimental formulations thought to have increased abrasion resistance. However, Reclamation's brush abrasion test indicated a material loss of 0.300–0.580 gram, which is a decrease in performance compared to Fuji's commercial product. Field testing showed many juvenile mussels and a few adult mussels attached to the surface in flowing water (dynamic) conditions. In static conditions, many adult mussels attached to the surface.

Reclamation experimented with many different formulations, but the mechanical properties were not significantly improved compared to commercial foul release coatings. The formulations with field results prevented mussel fouling.

The formulation entitled "Experiment 4" delivered the greatest improvement in mechanical properties by using a blend of polymer molecular weights and increasing the fumed silica content to 27 percent by weight of the cured coating. The results indicated that the poly (ethoxy silicate) curing agent provided better tensile strength, but the methyl triacetoxy silane curing agent provided better tear resistance. The majority of the experimental formulations exceeded Federal volatile organic compound regulations and, therefore, cannot be commercially sold or distributed in the United States for industrial maintenance coating applications.

It was not determined which mechanical property contributes most to improved abrasion resistance; tensile strength, elongation, and tear strength were measured for all samples.

INTRODUCTION

Preliminary Research

The Materials and Corrosion Laboratory staff evaluated foul release coatings at Parker Dam since May 2008. The Parker Dam facility consists of a large forebay area created by a trashrack bridge structure that spans the length of the forebay opening (figure 1). More than 100 coatings in flowing conditions downstream from the trashrack structure (yellow line) and also in quasi-static flow conditions on the upstream face of the dam (red line) were evaluated. Several of the commercial coatings evaluated prevented mussel attachment but were susceptible to damage; this presented a durability concern for application at Bureau of Reclamation (Reclamation) facilities.

Figure 1.—Aerial view of Parker Dam field test site.

One aspect of commercial foul release coating design is the slow release of silicone oils from the film to form an oil layer on the coating surface. The theory for marine environments is that fouling organisms, including mussels, cannot attach firmly to this protective layer. To evaluate the theory's applicability to invasive mussel attachment in freshwater, a commercial product was tested side-by-side with a silicone oil-free version of the same product in May 2012. The theory did not stand, and the silicone oil-free formulation continues to prevent mussel colonization following 3-1/2 years of field exposure, shown on figure 2.

Figure 2.—Fujifilm Hunt Smart Surfaces, LLC, oil free formulation at 178 weeks of exposure.

The inconsistency between marine and freshwater fouling requirements prompted efforts to better understand the coating properties required to prevent mussel fouling in freshwater environments. Pseudo-barnacle adhesion testing was conducted in accordance with American Society for Testing and Materials (ASTM) D5618 on commercially available foul release coatings. This laboratory screening method measures the shear force required to remove a circular dolly fixed to the coating surface with an adhesive. A lower force to remove the dolly generally resulted in better field performance. Table 1 shows both datasets for a comparison of many of the commercial products tested. The results are shaded green, yellow, and red to indicate good, fair, and poor performance, respectively.

Another preliminary study involved field evaluation of liquid applied silicone gasket materials. These gasket materials had improved mechanical properties compared to the silicone foul release coatings. Some of the silicone gasket materials prevented mussel attachment, while other materials did not. Although specific formulations were unknown, the results suggested that different combinations of fillers, polymers, and curing agents can improve mechanical properties while preventing mussel attachment.

Table 1.—Pseudo-barnacle adhesion tests on commercial coatings

Type	Commercial coatings	Pseudo-barnacle adhesion (psi)[1]	Field performance (mussel fouling)
Foul release	Fuji Optimized	7.3	No mussels
	Fuji Original	2	No mussels
	Fuji Oil Free	8.9	No mussels
	Bioclean	14.1	No mussels
	Sigmaglide 890	9.2	No mussels
	Intersleek 970	20.6	Few
	Intersleek 1425	3.3	No mussels
	Hempasil X3	8.4	No mussels
	Jotun Sealion Repulse	3.6	No mussels
	Nusil 9707	76	No mussels
	SEI Coat	30.9	Many
	AS&M Black	104.4	Many
	AS&M White	67.4	Many
Durable foul release	Seaspeed V5	> 390	Many
	Du Slip	> 390	Many
	SEI Chemical	> 390	Many
	Duromar 2510FR	166	Many
	Nusil R1082	229	Many, easily cleaned
	Jotun Sealion Resilient	122.7	Many, self-cleaning
	Tesla	> 390	Many

[1] Pounds per square inch.

Cooperative Research and Development Agreement Development

Reclamation initiated a Cooperative Research and Development Agreement (CRADA) conversation with Fujifilm Hunt Smart Surfaces, LLC (Fuji), in February 2013 to develop a more durable nonfouling coating system for freshwater applications. CRADA 13-CR-8-1006 was signed on March 5, 2014. This CRADA brought together the joint interests and complementary capabilities necessary to effectively achieve research results that neither party was producing on its own.

Reclamation's contributions:

- Research expertise from a materials engineering, chemistry, and biology perspective
- Use of Reclamation's Parker Dam field test site
- Use of Materials and Corrosion Laboratory equipment

Fuji's contributions:

- Research staff with extensive experience in formulating foul release coatings
- Specialized research and production facilities
- Extensive coating scale-up and manufacturing know-how

The following coating performance goals were identified under the CRADA:

1. Mussels will not colonize on the coating surface during a 1-year test period in the Colorado River. Colonization is defined as a group of mussels attached to a coating sample under normal water flows at the Parker Dam test site.

2. A coating will adhere to a corrosion-resistant primer or tie coat (i.e., no delamination).

3. Coating weight loss resulting from the Reclamation abrasion tests will not exceed:

   Target goal: 0.10 gram (g). Abrasion tests on currently available foul release coatings result in a weight loss of 0.50 g or more.

   Stretch goal: 0.05 g. Abrasion tests on a conventional epoxy coating result in a weight loss of 0.035 g.

4. A coating will meet NSF/ANSI Standard 61 for coatings used in water pipes with diameters equal to 54 inches or greater. Ideally, the coating will meet NSF/ANSI Standard 61 for pipes as small as 3 inches in diameter.

This report provides the final results and conclusions obtained during the performance period of the CRADA.

Cooperative Research and Development Agreement Research Plan

Reclamation and Fuji developed a collaborative research plan during a meeting in April 2014. The goal of the first study was to investigate variations in silicone topcoat thicknesses and determine if there was a lower limit in which fouling prevention degraded. Subsequent studies were conducted to pursue targeted mechanical properties. Based on preliminary field research and literature reviews, the CRADA partners identified the following approaches to creating a durable foul release coating:

1. Mechanical properties of silicones are improved by incorporating reinforcing fillers such as fumed silica. The filler's enhancement becomes stronger with increased concentration and decreased particle size. This experiment was also conducted in an attempt to maximize tear resistance; 30–35 percent (%) fumed silica is the desired pigment loading for this experiment (Brassard, 2010).

2. Increasing the number of covalent bonds between the material's silicone polymer and fumed silica particles increases its mechanical properties. This experiment was conducted in an attempt to increase covalent bonds in the polymer-pigment matrix using silicate curing agents to increase toughness in the resulting coating. Tri-functional methyl triacetoxy silane is a very common silicate curing agent. It can form silsesquioxanes (ring structures) when used in excess that aid in reinforcing coating materials.

3. The final approach was to reduce the thickness of the topcoat's silicone polymer layer to evaluate the effect of bulk modulus of the primer or tie coat on coating toughness.

The goal of the research was to develop a silicone polymer coating system that had greater resistance to scraping or impact damage; this exposure is typical of Reclamation's coated equipment. Baseline data provide a means to determine if experimental formulation properties are more desirable than commercially available coating systems. Tensile strength (ASTM D2370) and tear resistance (ASTM D624) are two tests that can be performed quickly to screen coatings for durability and toughness. Baseline mechanical testing was performed on three commercially available coatings: Sigmaglide 890, Fuji Duplex,[1] and NuSil R1082. The latter is a silicone coating with significantly increased toughness compared to other foul release coatings. Field results indicated that this coating allows mussels to attach, but it is easily wiped clean of mussels. These baseline test results are shown in table 2, reported as pounds per square inch (psi), per liner inch (pli), and percent (%).

[1] 2013 formulation.

Table 2.—Tensile strength and tear resistance of commercially available coatings. Baseline for developing new materials.

Product	Ultimate tensile (psi)	Elongation (%)	Tear strength (pli)
Sigmaglide 890	250	165	8
Fuji Duplex	145	148	9
Nusil R1082	1,421	950	126

EXPERIMENTAL

Dry Film Thickness Study

The first experiment executed under the CRADA was to evaluate Fuji's commercial product at varying surface coat film thicknesses. The goal was to understand the impact of silicone topcoat thickness on its ability to prevent mussel attachment. Fuji provided two replicates per sample set with the surface coat applied at the following thicknesses: 18, 29, 53, 63, and 97 microns. These samples were installed at the Parker Dam field test site on August 20, 2014.

Fuji's Formulations

Fuji screened more than 20 experimental formulations; the specific formulations were not made available to Reclamation. In their experiments, Fuji maintained a favorable solution viscosity for spray applications as they attempted to improve the dry film's mechanical properties. Typically, hydrophobic fumed silica is used to reinforce silicone polymers; however, this significantly increased the viscosity, requiring more solvent to achieve a sprayable viscosity (Brassard, 2010). Many formulations with the desired toughness were formulated, but balancing sprayable viscosities was very difficult to achieve without incorporating a high solvent content. Fuji next experimented with Celtix, a natural diatomaceous earth material, in an attempt to reinforce the polymer while minimizing the viscosity increase.

Fuji provided four formulations (T1, T3, T5, and T9) to Reclamation in March 2015 for lab and field evaluation; the formulations are listed in table 3. These samples were installed at Parker Dam for field testing on May 19, 2015. All formulations used a silanol terminated polydimethyl siloxane (PDMS) polymer resin. Formulation T1 used a resin with a molecular weight of 49,000 grams per mole (g/mol) and an ethyl silicate curing agent. Formulation T3 was the same formulation as T1, but it incorporated a quaternary ammonium salt in the polymer backbone as an antimicrobial agent. Formulation T5 used a

Table 3.—Fuji's experimental formulations, all shown in weight percent

ID	PDMS polymer	Celtix	Naphtha + pigment	Ethyl silicate	Quaternary ammonium
T1	36	22	40	2	0
T3	36	22	38	2	2
T5	36	22	40	2	0
T9	Commercial proprietary formulation				

slightly lower molecular weight resin, 44,000 g/mol, compared to T1 to decrease the viscosity of the formulation. Formulation T9 was the commercial foul release formulation.

Reclamation's Formulations

Reclamation's formulations aimed to identify coatings with increased durability while providing adequate fouling prevention. Reclamation did not target a sprayable solution viscosity, and the coatings were brush applied.

The formulation ingredients were purchased from Gelest, Inc., or Evonik, Inc., including a number of hydroxyl-terminated PDMS resins. Gelest DMS-S21, DMS-S27, DMS-S31, DMS-S35, DMS-S42, DMS-S45, and DMS-S51 have molecular weights 4,200, 18,000, 26,000, 49,000, 77,000, 110,000, and 140,000 g/mol, respectively. These polymers were crosslinked with three different curing agents: Gelest SIM 6519.0 methyl triacetoxy silane (MTAS), Gelest PSI-021 poly (diethoxy silicate), and Evonik Silbond 40 poly (diethoxy silicate).

The formulations were reinforced at varying levels with hydrophobic fumed silica treated with hexamethyldisilazane; Gelest SIS 6962.0 and Evonik Aerosil R 8200 fumed silica products were used. The primary difference was that Aerosil R 8200 has a surface area of 160 meters squared per gram (m^2/g) compared to Gelest SIS 6962.0, which has a surface area of 200 m^2/g. The average particle size is 20 nanometers for Gelest SIS 6962.0 and 25 nanometers for Aerosil R 8200.

The catalyst for the formulations was Gelest SND3260 dibutyltin diluarate. Naphtha, purchased from Superior Oil, was added to reduce viscosity and improve film formation.

Experimental Formulations with Field Results

The Reclamation formulations described in table 4 have field test results. The formulations varied the molecular weight of the silicone polymer, the percentage of fumed silica, and the type of curing agent in the formulations. Gelest fumed

Table 4.—Experiment 1 formulations shown in weight percent

ID	DMS-S31	DMS-S45	DMS-S51	Naphtha	SND3260	Fumed silica	SIM-6519	PSI-021
S31 MTAS 0	77.5			11.6	0.1		10.8	
S31 MTAS 5	72.3			12	0.1	5.3	10.3	
S31 MTAS 10	68.0			13.6	0.1	9.3	9.0	
S31 PSI-021 5	66.8			20	0.1	5.0		8.1
S45 MTAS 0		52		44.4	0.1		3.5	
S45 MTAS 5		46.7		46.7	0.1	3.4	3.1	
S45 MTAS 10		45.2		45.2	0.1	6.5	3.0	
S45 PSI-021 5		40		56	0.1	2.9		1.0
S51 MTAS 0			40.7	57	0.1		2.1	
S51 MTAS 5			40	54.6	0.1	3.2	2.1	
S51 MTAS 10			27.8	66.6	0.1	4.0	1.5	
S51 PSI-021 5			28.6	68.6	0.1	2.1		0.6

silica was the reinforcing pigment used, which was known to have a strong effect of increasing a coating's mechanical strength and imparting toughness (Brassard, 2010). It was also known that increasing the molecular weight of the silicone polymer increases the mechanical properties and abrasion resistance (Brassard, 2010). The majority of these formulations exceed the Federal volatile organic compounds regulation for industrial maintenance coatings at 450 grams per liter, which may limit their viability for commercialization.

Laboratory Studies Only

Reclamation also evaluated other formulations but did not initiate field testing prior to terminating the CRADA. Experiment 2 involved investigating different hydrophobic fumed silica, keeping the hydroxyl-terminated PDMS molecular weight constant. The PDMS used was Gelest DMS-S31. Two different fumed silicas were used: Evonik Aerosil R 8200 and Gelest SIS 6962.0. Table 5 shows these formulations.

Table 5.—Experiment 2 formulations shown in weight percent

ID	DMS-S31	Naphtha	SND3260	Fumed silica	PSI-021
5% Aero 8200	69.0	20.7	0.3	5.0	5.0
10% Aero 8200	65.8	19.7	0.3	9.5	4.7
20% Aero 8200	53.6	26.8	0.2	15.5	3.9
20% Gelest silica	53.6	26.8	0.2	15.5	3.9

Experiment 3 involved investigating the use of multiple polymer molecular weights. Table 6 shows these formulations. It was hypothesized that one could increase the tear resistance by using multiple molecular weight polymers, and the chains would break at different elongation lengths. For this study, 15 weight percent (wt. %) of Gelest SIS 6962.0 fumed silica was constant; it varied the ratios and molecular weights of the polymer.

Table 6.—Experiment 3 formulations shown in weight percent

ID	DMS-S27	DMS-S31	DMS-S35	DMS-S45	Naphtha	Fumed silica	SND3260	PSI-021
A Varied MW ratios	0	31.9	0	19.2	38.3	9.5	0.2	1.0
B Varied MW ratios	0	31.9	9.5	9.5	38.3	9.5	0.2	1.1
C Varied MW ratios	6.4	31.9	6.4	6.4	38.3	9.5	0.2	1.0
D Varied MW ratios	9.5	31.9	0	9.5	38.3	9.5	0.2	1.1
E Varied MW ratios	9.5	31.9	9.5	0	38.3	9.5	0.2	1.1
F Varied MW ratios	0.0	31.9	19.2	0	38.3	9.5	0.2	1.0
G Varied MW ratios	19.2	31.9	0	0	38.3	9.5	0.2	1.0
H Varied MW ratios	0	31.9	6.4	12.7	38.3	9.5	0.2	1.0

Experiment 4 involved investigating multiple molecular weights of hydroxyl-terminated PDMS but kept the ratios of each molecular weight constant. For this study, fumed silica content increased to 27 wt. %. Both Gelest SIS 6962.0 and Aerosil R 8200 were used for direct comparison. Two curing agents were used in this study. Fuji suggested Silbond 40 because it is believed to be similar to Gelest PSI-021. The other curing agent was Gelest SIM 6519.0. Table 7 shows these formulations.

Table 7.—Experiment 4 formulations with 27 wt. % fumed silica, shown in weight percent

ID	DMS-S15	DMS-S21	DMS-S31	DMS-S27	DMS-S35	DMS-S42	DMS-S45	Naphtha	Fumed silica	SND3260	SIM-6519	Silbond 40
Gelest Si Silbond 40	4.3	4.3	4.3	4.3	4.3	4.3	4.3	53.2	12.8	0.1	0.0	3.8
Gelest Si MTAS	4.3	4.3	4.3	4.3	4.3	4.3	4.3	53.2	12.8	0.1	3.8	0.0
Gelest Si Silbond 40/MTAS	4.3	4.3	4.3	4.3	4.3	4.3	4.3	53.2	12.8	0.1	1.9	1.9
8200 Si Silbond 40	4.3	4.3	4.3	4.3	4.3	4.3	4.3	53.2	12.8	0.1	0.0	3.8
8200 Si MTAS	4.3	4.3	4.3	4.3	4.3	4.3	4.3	53.2	12.8	0.1	3.8	0.0
8200 Si Silbond 40/MTAS	4.3	4.3	4.3	4.3	4.3	4.3	4.3	53.2	12.8	0.1	1.9	1.9

Test Procedures

Linear Abrasion

The mechanical abrasion testing procedure was performed using a reciprocating Linear Taber Abraser test machine (Model 5750) equipped with a silicon carbide abrasive end brush 1/2-inch diameter with a 0.035-inch bristle diameter manufactured by Weiler. A 3 x 6 x 1/8-inch panel was submerged in 10 ounces of deionized water in an acrylic tub and held in place by two C-clamps. A weight (1,000 g) was placed on a splined shaft connected to the brush to control the normal force exerted on the coated surface; the total weight of the splined shaft and weight was 1,380 g. The brush was cycled 1,500 times at a speed of 75 cycles per minute, creating a 3.5-inch wear track on the coating. The test panel was removed from the solution and allowed to dry overnight. The coating was weighed to the nearest 0.001 g to determine material loss due to abrasion. The test was terminated at 1,500 cycles or at breakthrough to the epoxy coating, whichever occurred first.

Pseudo-Barnacle Adhesion Test

The pseudo-barnacle adhesion test is a testing procedure used to rank coatings to determine the release properties. The tests were performed in accordance with ASTM D5618.

Tensile Properties

Tensile strength, elongation at break, and elastic modulus were measured following ASTM D2370. The sample size was 0.5 inch width and 2 inch gauge length. All samples were run at an extension rate of 2 inches per minute.

Tear Resistance

Tear resistance was measured in accordance with ASTM D624. Die cut C was used for all samples. The samples were run at an extension of 20 inches per minute.

RESULTS AND DISCUSSION

Dry Film Thickness Study

All variations in silicone thicknesses allowed algae to attach to the surface but were free of mussels after 69 weeks exposure in dynamic conditions. This suggests that only a thin layer of silicone is required on a coating's surface to provide nonfouling behavior in these test site conditions. Figure 3 shows a panel with an 18-micron surface coat.

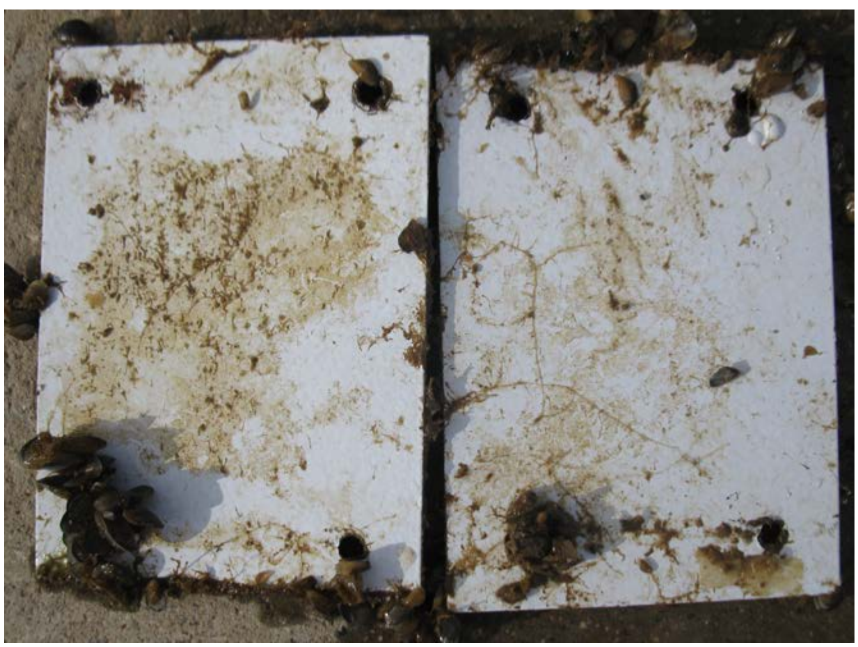


Figure 3.—Fuji commercial product with 18-micron surface coat, 69 weeks exposure.

Fuji's Laboratory Testing

Laboratory test results of Fuji's formulations are provided in table 8. Additional information was provided but without corresponding formulation recipes. The maximum mechanical properties obtained were ultimate tensile strength of 870 psi, elongation of 148%, and tear strength of 61 pli.

Table 8.—Mechanical properties of Fuji's formulations

Formulation name	Pseudo-barnacle adhesion (psi)	Mechanical properties: Ultimate tensile strength (psi)	Mechanical properties: Elongation (%)	Mechanical properties: Tear strength (pli)	Abrasion weight loss (g)
T1	131	595	59	51	0.580
T3	135	522	50	48	0.447
T5	120	580	53	50	0.428
Fuji's commercial product	2	145	148	9.0	0.300

Abrasion testing produced significant damage on the commercial formulation, but it resisted breakthrough to the epoxy coating for more than 1,500 cycles (figure 4). Formulations T1, T3, and T5 abraded to the epoxy layer within 1,500 cycles. Figure 5 shows T3 as an example of the wear, following completion of the test.

Figure 4.—Fuji commercial product, brush abrasion test after 1,500 cycles and 4,500 cycles.

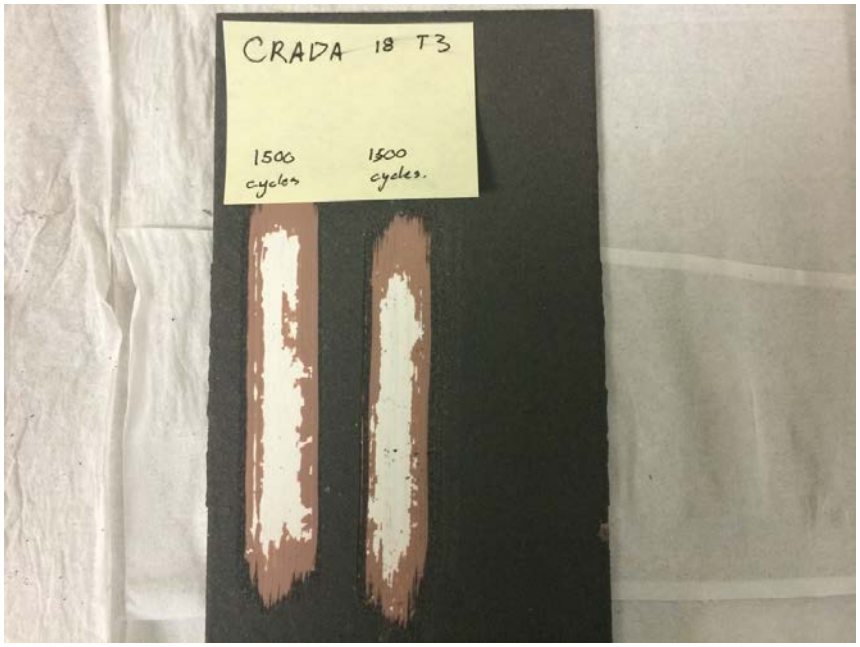


Figure 5.—Fuji experimental product CRADA T3, brush abrasion test after 1,500 cycles.

Fuji Film's Field Results

The field performance of the Fuji experimental formulations is inconclusive due to test duration. Many juvenile mussels and a few adults were adhered to the coating surface in dynamic conditions, as seen on figure 6. In static water, a greater number of adult mussels were present, as seen on figure 7. This could mean that the coatings are self-cleaning in flowing water conditions. These coatings would need to be tested longer to determine their long-term performance.

Figure 6.—Fuji's T1 formulation after 30 weeks of dynamic water exposure.

Figure 7.—Fuji's T1 formulation after 30 weeks in static water exposure.

Reclamation's Laboratory Results

Table 9 shows the laboratory results for the first experiment, evaluating pseudo-barnacle adhesion, mechanical properties, and mechanical abrasion. The colored cells represent desired (green) to undesirable (red) results. Tensile and tear strength increased with increasing fumed silica content and molecular weight as expected. Future studies will be conducted in an attempt to further increase tensile and tear strength by increasing the fumed silica content.

Table 9.—Experiment 1 pseudo-barnacle, mechanical property, and mechanical abrasion test results

ID	Pseudo-barnacle adhesion (psi)	Mechanical properties			Abrasion weight loss (g)
		Ultimate tensile strength (psi)	Elongation (%)	Tear strength (pli)	
S31 MTAS 0	11.5	35.8	85	4.6	0.395
S31 MTAS 5	8	270.9	175	8.4	0.388
S31 MTAS 10	8.1	434.2	N/A	8.7	0.117
S31 PSI-021 5	10.7	420.7	N/A	4.5	0.227
S45 MTAS 0	12.5	44.0	259	6.5	0.33
S45 MTAS 5	23.9	105.2	459	15	0.293
S45 MTAS 10	22	203.8	786	17.5	0.222
S45 PSI-021 5	7.5	118.8	287	12.8	0.084
S51 MTAS 0	16.1	34.8	205	9.5	0.319
S51 MTAS 5	26.7	54.7	315	12.8	0.235
S51 MTAS 10	35.5	175.7	660	25.3	0.239
S51 PSI-021 5	12.8	66.0	171	16.8	0.084

Two formulations met the goal of the CRADA to have less than 0.1 g weight loss in the mechanical abrasion test. Unfortunately, these were very high molecular weight polymers that required an extreme amount of solvent to reduce the viscosity to the point required to apply as a coating film. In general, the results show that the poly (diethoxy silicate) (PSI-021) curing agent provides better abrasion resistance than the MTAS.

Table 10 shows the laboratory test results for Experiment 2. Pseudo-barnacle adhesion increases with fumed silica content. The mechanical properties were not an improvement over the commercially available foul release coatings, which could be due to using similar molecular weight polymers that are used in the foul release coatings. Abrasion resistance was not measured due to the mechanical properties not being significantly different from the commercial products.

Table 10.—Experiment 2 results showing the effect of hydrophobic fumed silica

Formulation	Pseudo-barnacle adhesion (psi)	Ultimate tensile strength (psi)	Elongation (%)	Tear strength (pli)
5% Aero 8200	16	120	156	4.6
10% Aero 8200	27.6	196	125	7.4
20% Aero 8200	50.8	290	121	10.8
20% Gelest silica	33.4	232	400	9.1

Table 11 shows the laboratory results for Experiment 3. The majority of the formulations in this set had higher pseudo-barnacle adhesion compared to commercial products. The mechanical properties were comparable to the commercial products Sigmaglide 890 and Fuji Optimized. The tear resistance was slightly increased when higher molecular weights were used.

Table 11.—Experiment 3 results for multiple molecular weights

15% gelest silica	Pseudo-barnacle adhesion (psi)	Ultimate tensile strength (psi)	Elongation (%)	Tear strength (pli)
A	17.4	261	181	10.3
B	42.6	273	173	9.1
C	25.2	245	145	8.6
D	31.0	290	158	6.9
E	19.4	232	124	9.7
F	22.9	247	147	9.1
G	18.3	157	75	6.9
H	24.7	268	175	8.6

Table 12 shows the laboratory results for Experiment 4. All formulations in this set had higher pseudo-barnacle adhesion compared to commercial products. Tensile and tear strength increased compared to the Sigmaglide 890 and Fuji Optimized. However, they were still lower than Nusil R1082. Silbond 40 provided the highest tensile strength, lowest elongation, and slightly higher tear resistance compared to commercial foul release coatings. The MTAS curing agent provided the highest tear strength; by blending the two curing agents, it provided high tensile and tear strengths.

Table 12.—Experiment 4 results for 27 wt% fumed silica and varied curing agents

Formulation	Pseudo-barnacle adhesion (psi)	Ultimate tensile strength (psi)	Elongation (%)	Tear strength (pli)
Gelest Si Silbond 40	51.9	648	60	13.9
Gelest Si MTAS	120.2	262	141	15.3
Gelest Si Silbond 40/MTAS	46.7	589	71	14.2
8200 Si Silbond 40	46.4	645	76	15.3
8200 Si MTAS	50	261	44.5	17.4
8200 Si Silbond 40/MTAS	54.5	503	70	16.1

Fuji indicated during conversations that their formulations had better tensile and tear strength than Reclamation's formulations. From this point forward, Fuji would formulate new coatings while Reclamation provided input, conducted abrasion testing, and perform field testing. Once the field test results were obtained in December 2015, Fuji and Reclamation would have further discussions on the next steps.

Reclamation's Field Results

The Experiment 1 formulations prevented mussel attachment during testing at Parker Dam. Figure 8 shows the field results of formulation S51 MTAS 10, which is representative of all formulations. These formulations were not highly reinforced but provided excellent field performance. This particular formulation had higher elongation properties compared to commercial foul release coatings.

Figure 8.—S51 MTAS 10 at 82 weeks of exposure.

CONCLUSIONS

The Fuji film thickness study indicated that all silicone thicknesses down to 18 microns prevented mussel attachment. This further supports the hypothesis that mussels do not attach to the thinnest surfaces of silicone. Therefore, it may be possible to formulate a tougher tie coat to increase the system's bulk modulus.

The experimental formulations provided by Fuji were incorporated into field testing in May 2015. The field results were inconclusive; however, adult mussels were attached to the surface in static exposure, and many juvenile mussels attached to the surface in dynamic conditions. The laboratory test results showed lower abrasion resistance properties than the commercially available system. Several formulations abraded to the epoxy primer within 1,500 cycles.

The experimental formulations prepared by Reclamation provided minimal improvement in tensile and tear strength compared to commercially available foul release coatings. The best properties required 27 wt. % fumed silica, which significantly increased the viscosity, requiring a large amount of solvent to achieve a sprayable viscosity. The poly (ethoxy silicate) curing agent provided greater tensile strength than the MTAS curing agent; however, the latter gave better tear strength.

REFERENCES

1. Brassard, David. 2010. The Silicone Elastomer Handbook. Silicone Solutions.

Laboratory Experimental Data of Field Test Samples -- BR & Fujifilm CREDA: #13-CR-8-1006

Formulas

	Field Code T1 [wt, %]	Field Code T3 [wt, %]	Field Code T5 [wt, %]	Field Code T7 [wt, %]	Field Code T9 [wt, %]
PDMS - DMS-S35 (Gelest)	36	36			
PDMS - DMS-S33 (Gelest)			36	36	
Diatomaceous Earth (World Mineral)	22	22	22	22	
Silicon Carbide - SIS6569.0 (Gelest)	3	3	3	3	
Organosilane Antimicrobials - HM4100 (Gelest)		2		2	
Black Pigments - Silcopas Black 220 (Gayson)	3	3	3	3	
Ethyl Polysilicate (Silbond)	2	2	2	2	
Di-n- Butyldiauryl Tin (Gelest)	0	0	0	0	
Solid Content [we, %]	65	67	65	67	
Naphtha (Ashland)	35	33	35	33	
Note - Reference or Test	Test	Test	Test	Not Tested	Commercial Sher-Release Surface Coat - Reference

Mechanical Strength

Y. Modulus, [N/mm^2]	39.31	30.73	34.44	42.44	0.45
Toughness, [N.mm]	445	360	447	294	267
Tensile Strength, [N/mm^2]	4.1	3.6	4.0	3.4	1.0
Elongation, [%]	59	50	53	42	148
Tear Strength,[lb/in]	51	48	50	47	8.9
Scratech Resistance (ASTM G-171 Modified), [g] Force to cut through 12 mils of TC & SC	1057	1111	1150	1046	389

Surface Property

Contact Angle, [Degree] (ASTM-D7334-08 & D5725-99-R03)	134	134	132	135	107

Foul Release Performance Estimate

Pseudo-Barnacle Adhesion Strength (ASTM D5179-02), [psi]	131.3	134.7	120.3	120.8	1.6

(Data for Final Report – ST-2016-0809-01_022916)

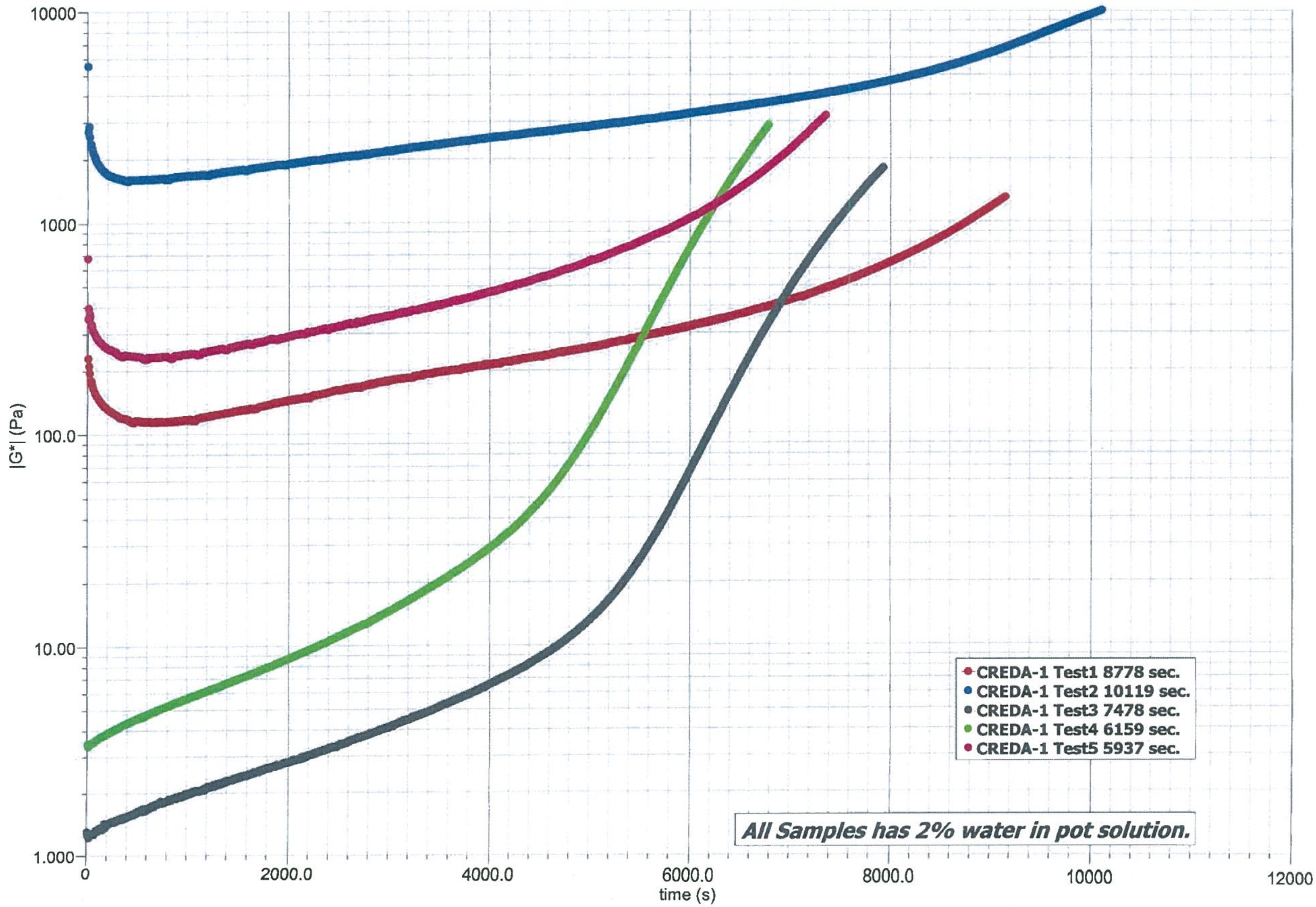
CREDA-1

Effect of Adding Water to Pot Solution

	Modulus	Energy to Br	Stress to	Elongation	Tear
		Break	Break		Strength
	N/mm^2	N.mm	N/mm^2	%	N/mm
D 201305129 w/o water in pot solution	0.498	492.552	1.691	176.194	1.530
D201305129 with water in Pot solution	0.447	267.091	0.971	148.023	1.556
Test1 w/o water in pot solution	1.470	433.900	2.434	116.193	1.668
Test1 with water in pot solution	1.282	290.664	1.192	96.627	1.689

Other Physical property change - Surface roughness

CREDA-1 Curing



4/25/2014

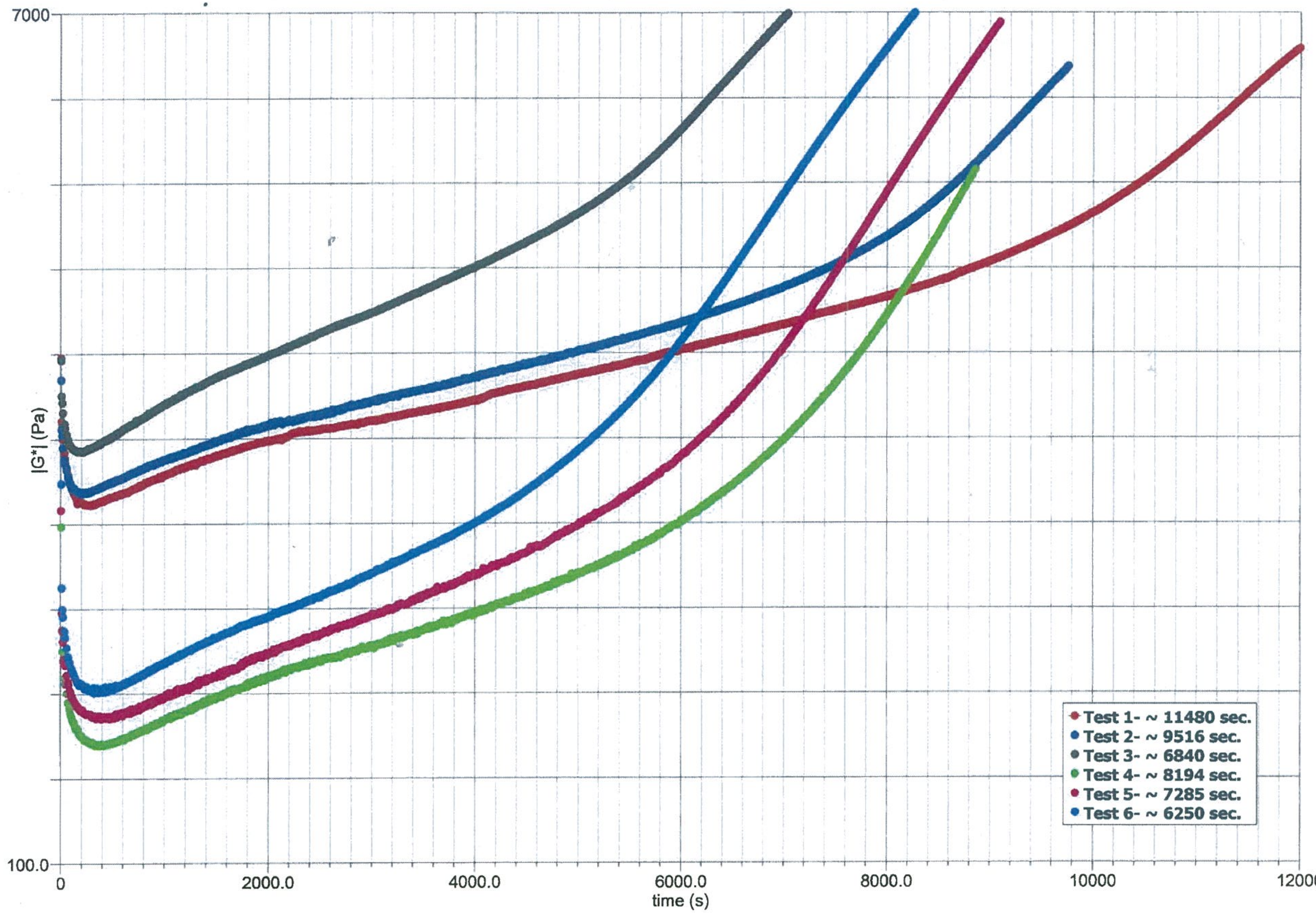
Part A Viscosity (Brookfield DVII+ Viscometer; RV Spindle #3)						
	Ref.	T1	T2	T3	T4	T5
		423.60	7000.00	262.80	847.10	1444.00
		422.90	7920.00	260.60	851.40	1486.00
		424.30	8290.00	260.60	848.60	1496.00
Average		**423.60**	**7737.00**	**261.33**	**847.03**	**1475.33**
rpm and torque		140, 59.3%	10, 70%	180, 47.3%	70, 59.2%	50, 72.2%
Pot solution mixing						
PartA	321.06	342.08	342.69	339.47	332.94	345.38
PartB	56.66	60.36	60.47	59.91	58.75	60.95
Water	7.554	8.05	8.06	7.99	7.83	8.12
Pot Solution Viscosity (Brookfield DVII+ RV Spindle						
Initial		680.00	773000.00	184.50	360.00	5360.00
5 min		674.30	735000.00	196.00	416.40	5240.00
10 min		687.10	723000.00	206.50	458.60	5270.00
Average		**680.47**	**743666.66**	**195.66**	**411.66**	**5290.00**
Spindleand rpm		#3; 70	#5; 0.3	#3; 200	#3; 140	#3; 50
torque		47.60%	58.00%	36.90%	50.40%	53.60%

Mechanical Properties						
Modulus N/mm^2	0.447	1.282	2.006	0.910	1.515	1.709
Energy to break N.mm	267.091	290.664	436.199	278.064	235.821	168.350
Stress @ break N/mm^2	0.971	1.192	2.127	1.365	1.564	1.243
Elongation %	148.023	96.627	87.264	104.513	78.400	60.560
Tear Strength N/mm	1.556	1.689	3.725	0.813	1.292	1.981
Shore A hardness	19	41	56	34	42	42

CRADA-2 - Sample Dry Film Thickness_072414

		1 - 2 mil			2 - 4 mil			4 - 6 mil			6 - 8 mil			8 = 10 mil	
		1			2			3			4			5	
Coat Layer	1a	1b	1c	2a	2b	2c	3a	3b	3c	4a	4b	4c	5a	5b	5c
Epoxy - Gray	238	222	282	200	226	258	250	300	196	234	276	288	300	222	246
	252	240	324	222	254	276	236	300	242	242	262	298	300	232	246
	234	254	292	246	268	274	238	312	242	254	264	284	296	270	256
	228	248	284	238	276	276	248	284	254	266	266	284	332	274	254
	248	252	256	264	272	282	212	266	256	262	250	304	292	272	260
	238	256	228	252	274	280	198	246	264	274	238	288	292	274	254
	252	246	206	252	308	276	232	238	266	284	258	292	312	262	266
	242	228	256	258	318	274	264	264	266	300	256	290	332	288	266
	222	240	282	256	328	264	264	300	254	282	266	298	320	268	266
	192	232	214	218	268	282	232	312	206	244	280	294	286	242	244
Ave.	235	242	262	241	279	274	237	282	245	264	262	292	306	260	256
S.D.	18	12	38	21	31	8	21	27	25	21	12	7	17	21	9
Ave.		**246**			**265**			**255**			**273**			**274**	
Tie Coat	226	206	276	200	196	232	244	302	234	226	282	250	278	256	260
	258	236	292	230	240	254	266	292	236	244	272	262	308	272	262
	254	246	276	260	256	274	238	278	256	258	270	276	272	282	282
	256	244	284	264	266	284	240	250	266	286	276	274	262	300	274
	228	210	248	262	284	274	214	224	268	270	254	278	238	292	274
	234	192	246	212	176	272	216	218	232	286	240	266	248	288	276
	232	228	222	230	212	218	248	216	242	264	246	252	276	272	264
	238	242	242	256	244	260	250	242	254	274	274	250	274	258	278
	236	232	202	256	260	276	256	252	246	246	272	240	296	258	270
	214	216	206	238	248	252	248	294	232	220	262	240	280	218	266
Ave.	238	225	249	241	238	260	242	257	247	257	265	259	273	270	271
S.D.	14	18	32	22	34	21	16	33	14	23	14	14	21	24	7
Ave.		**237**			**246**			**248**			**260**			**271**	
Δ					**9**			**11**			**23**			**34**	
Tie Coat Thickness, μ	3	-17	-13	0	-41	-15	5	-25	2	-7	3	-33	-33	9	15
Surface Coat-White	240	226	304	232	224	268	302	308	258	274	306	298	356	340	376
	254	246	322	230	228	256	298	298	268	296	326	296	356	352	372
	250	244	312	244	230	286	306	312	280	290	334	308	366	358	378
	258	250	272	242	234	274	298	300	284	302	352	316	352	352	368
	286	274	266	270	220	274	340	354	290	284	366	330	386	362	380
	244	270	298	278	258	268	298	302	320	316	374	298	374	388	354
	260	254	322	268	252	326	298	302	300	322	328	308	362	382	364
	270	240	328	272	256	276	290	304	312	324	332	296	380	374	378
	278	246	266	286	270	278	282	332	304	326	316	310	398	388	370
	306	264	264	310	270	286	290	362	326	318	340	338	396	392	404
	254	236	256	284	292	320	254	288	314	334	356	330	330	384	344
	214	240	264	274	282	260	276	286	296	336	314	326	328	372	356
	198	244	238	284	286	266	268	292	308	310	304	346	326	368	366
	220	222	238	308	304	288	274	316	302	328	320	312	318	372	374
	234	258	228	318	278	306	292	296	330	322	348	342	350	422	350
	256	228	218	296	278	272	280	276	310	324	326	326	360	378	354
	236	252	248	290	286	276	274	308	314	328	334	338	366	368	382
	238	256	244	300	250	290	308	310	298	304	324	290	372	388	378
	262	254	274	272	246	264	300	310	304	304	328	302	382	356	388
				276	254	274	342	358	292	322	364	304	390	376	378
														378	392
Ave.	250	248	272	277	260	280	294	311	301	313	335	316	362	374	372
S.D.	25	14	34	25	25	19	21	24	18	17	20	17	24	18	15
Ave.		**257**			**272**			**302**			**321**			**369**	
Δ					**16**			**45**			**65**			**113**	
Surface Coat Thickness, μ	13	22	22	36	22	21	52	54	54	56	70	57	89	104	101
Ave - S.C.DFT		***19***			***26***			***53***			***61***			***98***	

CREDA-3 Effect of Low Solid Content and High Level of Cross Linker



6/6/2014

CRADA - 3 6/30/14 (start 6/5/14)

T1	T2	T3	T4	T5	T6
41.5	45.5	51.5	41.5	44.5	52.5
42.5	47.0	54.0	42.5	47.0	54.0
40.0	48.0	55.0	41.5	46.0	53.0
41.0	46.0	53.5	41.5	47.0	56.5
40.0	48.0	53.5	42.0	48.0	53.5
40.0	48.0	53.0	43.0	48.0	53.0

Shane H

6/3/14 71°F 44%

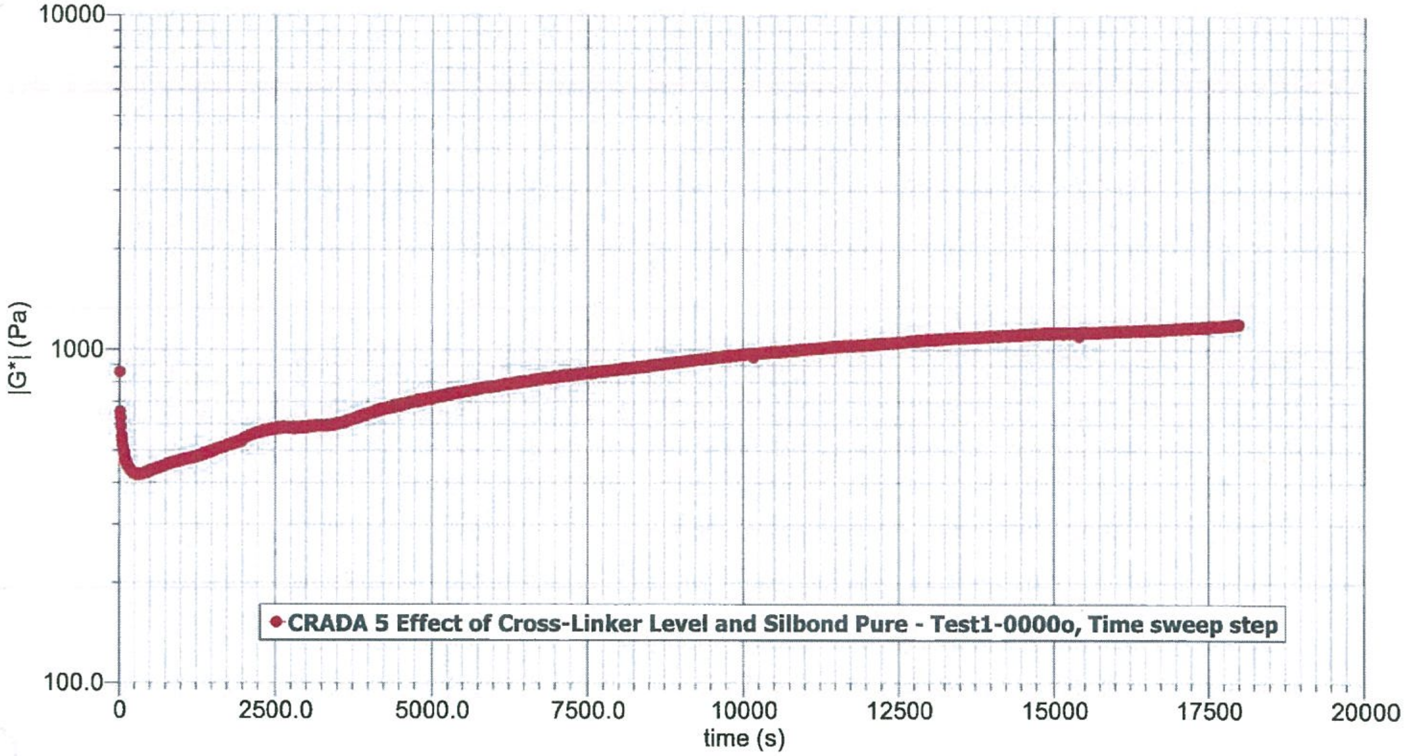
CRADA -3

	Test 1		Test 2		Test 3		Test 4		Test 5		Test 6
#3 15/64.7	4313					#3 50/63.9	1278				
	4320						1328				
	4293						1346				

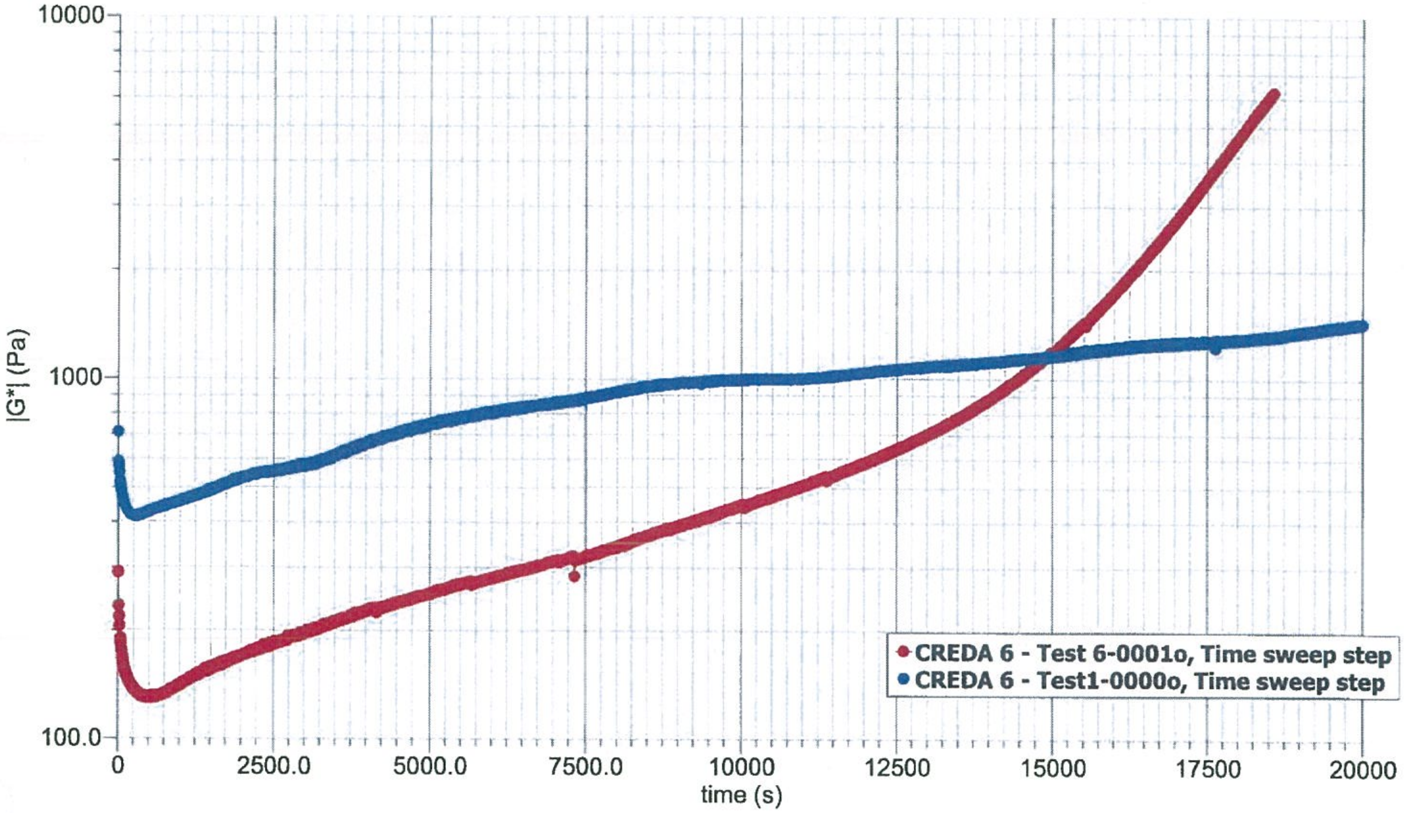
POT soluta

#5 50/46.1	3688	50/53.1	4248	50/57.6	4560	180/37.5	833.3	180/44.1	980.0	100/38.3	1532
	3648		4200		4440		837.8		993.3		1560
	3672		4248		4456		860.0		1033		1656
	3712		4320		4568		888.9		1089		1788
	3856		4576		4890		1004.0		1329		2408
	4040		4912		5360		1164.0		1742		3748
	4272		5360		6136		1384.0	140/96.7	2763	~~30~~ 20/91.0	18120

CRADA 5 Effect of Cross-Linker Level and Silbond Pure - Test1-0000o



CREDA 6 - Test 6-0001o

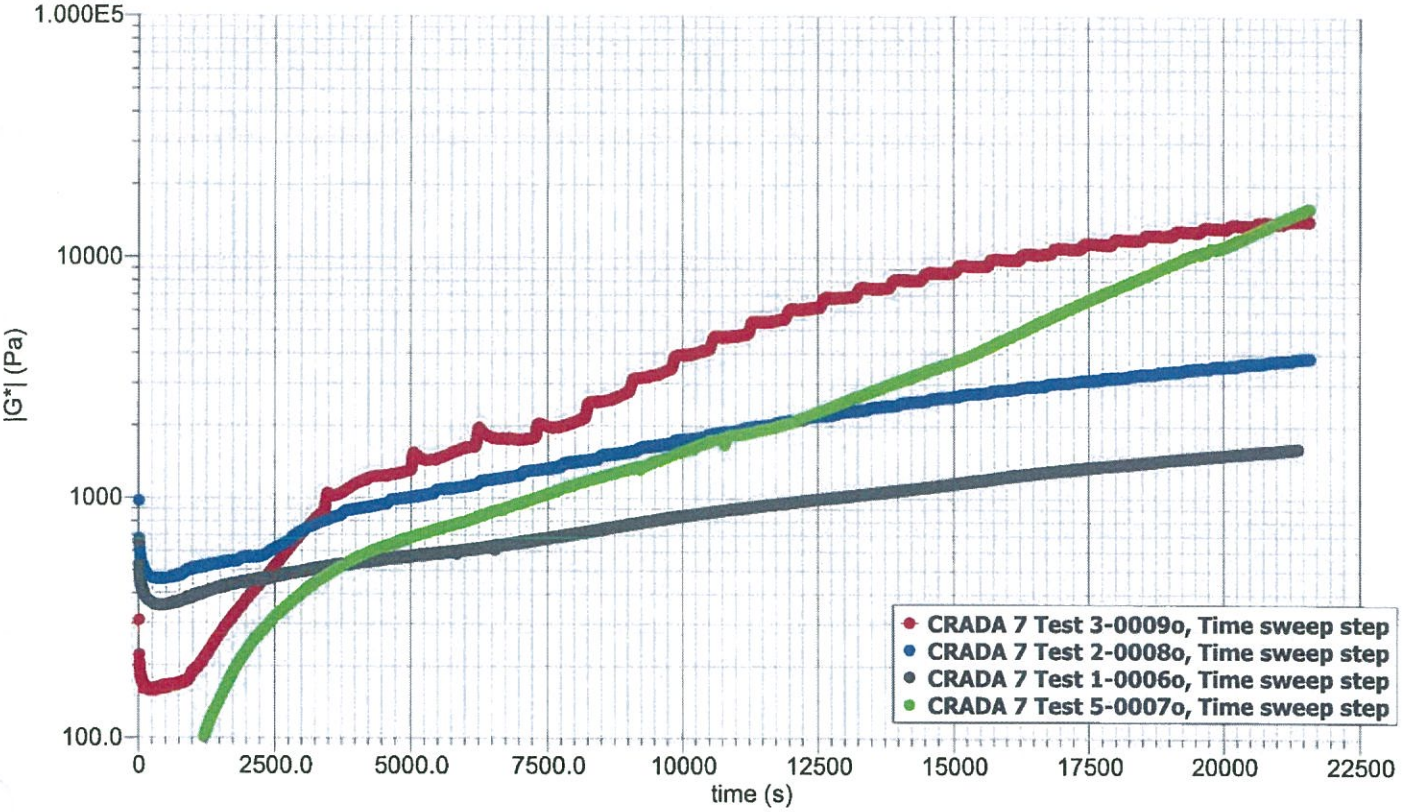


8/25/2014

CRADA-6						
	T1	T2	T3	T4	T5	T6
Part A Viscosity (Brook field)	1280.00	874.00	704.00	1092.00	994.00	916.00
#3 spindle;	1226.00	830.00	678.00	884.00	936.00	902.00
	1214.00	818.00	674.00	866.00	922.00	896.00
Average	**1240.00**	**840.67**	**685.33**	**947.33**	**950.67**	**904.67**
	50; 64%	50; 43.7%	50; 35.2%	50; 54.6%	50; 49.7%	50; 45.8%
Apperance	flowable					
Mixing						
Part A	434.41	453.67	455.66	452.88	449.21	466.30
Part B	76.66	80.06	80.41	79.72	79.27	82.28
Apperance	gel-flowable	Flowable				
Pot Viscoaity (Brookfield)	6300.00	1713.00	564.30	250.70	215.00	1387.00
	6240.00	1640.00	544.30	245.70	214.30	1257.00
	6260.00	1627.00	538.60	247.90	218.60	1227.00
Average	**6266.67**	**1660.00**	**549.07**	**248.10**	**215.97**	**1290.33**
spindle; rpm; torque	3; 10; 63%	3; 30; 51.4%	3; 70; 39.5%	3; 140; 35.1%	3; 140; 30.1%	3; 30; 82.28%
Dry to touch in minutes	**435**	**427**	**296**	**250**	**233**	**245**
Curing X Time Brookfield Viscometer						
Fresh	6300	1713	564.3	250.7	215.0	1387
15 min	6260	1623	540.0	250.7	222.1	1217
30 min	6360	1647	552.9	264.3	237.9	1230
45 min	6480	1667	571.4	279.3	257.9	1263
60 min	6620	1723	591.5	298.6	283.6	1307
spindle; rpm; torque	5, 50. 25.7%	5,50, 24.2	5, 50, 21.5%	5, 50, 26%	5, 50, 23.3%	5, 50, 21.4%
Mechanical Properties						
Modulus N/mm^2	1.87	2.30	2.97	3.72	4.67	4.15
Energy to Break N.mm	324	300	433	426	505	361
Stress @ Break N/mm^2	2.72	2.46	3.22	3.47	3.62	3.48
Elongation %	103.52	82.83	89.31	76.04	77.15	75.43
Trear Strength N/mm	3.91	3.20	2.96	3.34	4.79	4.23
Shore A Hardness	36	35	36	40	52	41
7/10/14-7/22/14	34.00	32.00	32.00	36.00	43.00	37.00
	37.00	33.00	35.00	37.00	40.00	45.00
	34.00	33.00	37.00	40.00	53.00	42.00
	36.00	33.00	37.00	40.00	51.00	43.00
	36.00	33.00	36.00	35.00	55.00	43.00
	38.00	33.00	38.00	42.00	52.00	42.00
	36.00	37.00	37.00	43.00	52.00	43.00
	37.00	34.00	36.00	42.00	55.00	42.00
	37.00	36.00	35.00	38.00	54.00	43.00
	37.00	34.00	34.00	42.00	51.00	38.00
				37.00	55.00	36.00
					43.00	38.00
					50.00	
Ave	36	35	36	40	52	41
S.D.	1	2	2	3	5	3

7/10/14-8/21/14	40	37	47	49	52	46
	40	45	46	53	58	47
	38	46	47	47	52	52
	39	44	45	51	55	58
	43	43	38	47	57	58
	41	42	48	54	56	60
	41	42	48	48	62	59
	41	45	47	51	63	60
	41	45	50	51	61	61
	45	44	50			62
Ave	41	43	47	50	57	56
S.D.	2	3	3	3	4	6

CRADA 7 Test 3-0009o



CRADA-7b

	T1	T2	T3	T4	T5	T6	T7
						Not able to make	

Mixing 90:10

PartA Viscosity	1660	2223	1029	1446	954
spindle #4	1626	2203	960	1240	889
	1603	2129	914	1157	880
Average	**1629.7**	**2185.0**	**967.7**	**1281.0**	**907.7**
	70 rpm; 58.1%	70 rpm; 77.8%	70 rpm; 36.0%	70 rpm 50.5%	70 rpm 33.4%

Mixing	328.21	352.31	490.16	559.17	440.37
	36.77	39.14	54.45	62.12	48.93

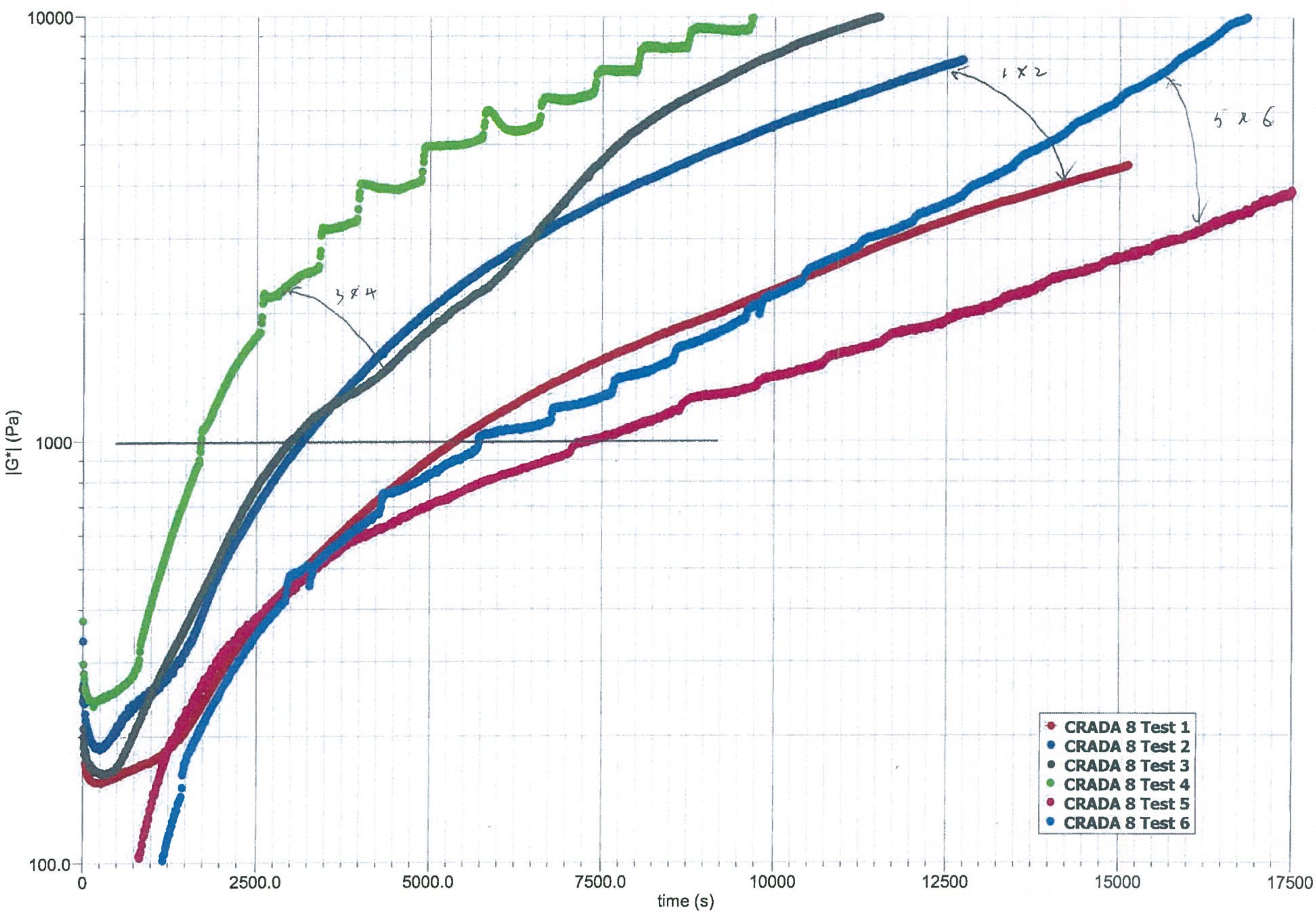
T1
T2
T3
T4
T5

Pot solution Viscosity	11240	13400	1580	1705	252
spindle #3	10620	12700	1505	1640	239
	10400	12480	1495	1625	235
Average	**10753.3**	**12860.0**	**1526.7**	**1656.7**	**242.0**
	5 rpm; 56.2%	5 rpm; 87.0%	20 rpm; 31.6%	20 rpm 34.1%	100rpm 25.2%

Mechanical Properties							
Modulus N/mm^2	5.53	7.72	13.98	20.66	19.75		
Energy to Break N.mm	535.31	545.07	522.59	521.87	430.44	N/mm	lb/in
Stress @ Break N/mm^2	4.49	4.89	5.54	5.38	4.41	1.000	5.710147
Elongation %	87.05	77.87	62.78	54.68	48.67		
Trear Strength N/mm	7.93	7.29	9.28	10.68	8.46		
	45.27	41.60	53.01	61.00	48.30		
Shore A Hardness	54.25	64.00	84.67	86.22	82.00		
Measured 2014/09/03	57.00	62.00	80.00	90.00	80.00		
Started 2014/07/29	52.00	67.00	87.00	82.00	87.00		
	52.00	61.00	77.00	92.00	84.00		
	55.00	57.00	87.00	94.00	80.00		
	53.00	57.00	78.00	86.00	80.00		
	49.00	66.00	87.00	93.00	84.00		
	57.00	71.00	85.00	86.00	82.00		
	54.00	64.00	90.00	83.00	81.00		
	60.00	62.00	86.00	85.00	82.00		
	55.00	58.00	85.00	90.00	83.00		
	53.00	67.00	90.00	85.00	79.00		
	54.00	67.00	83.00	78.00	84.00		
			78.00	90.00	83.00		

Ave	54	64	85	86	82
S.D.	3	5	4	5	2

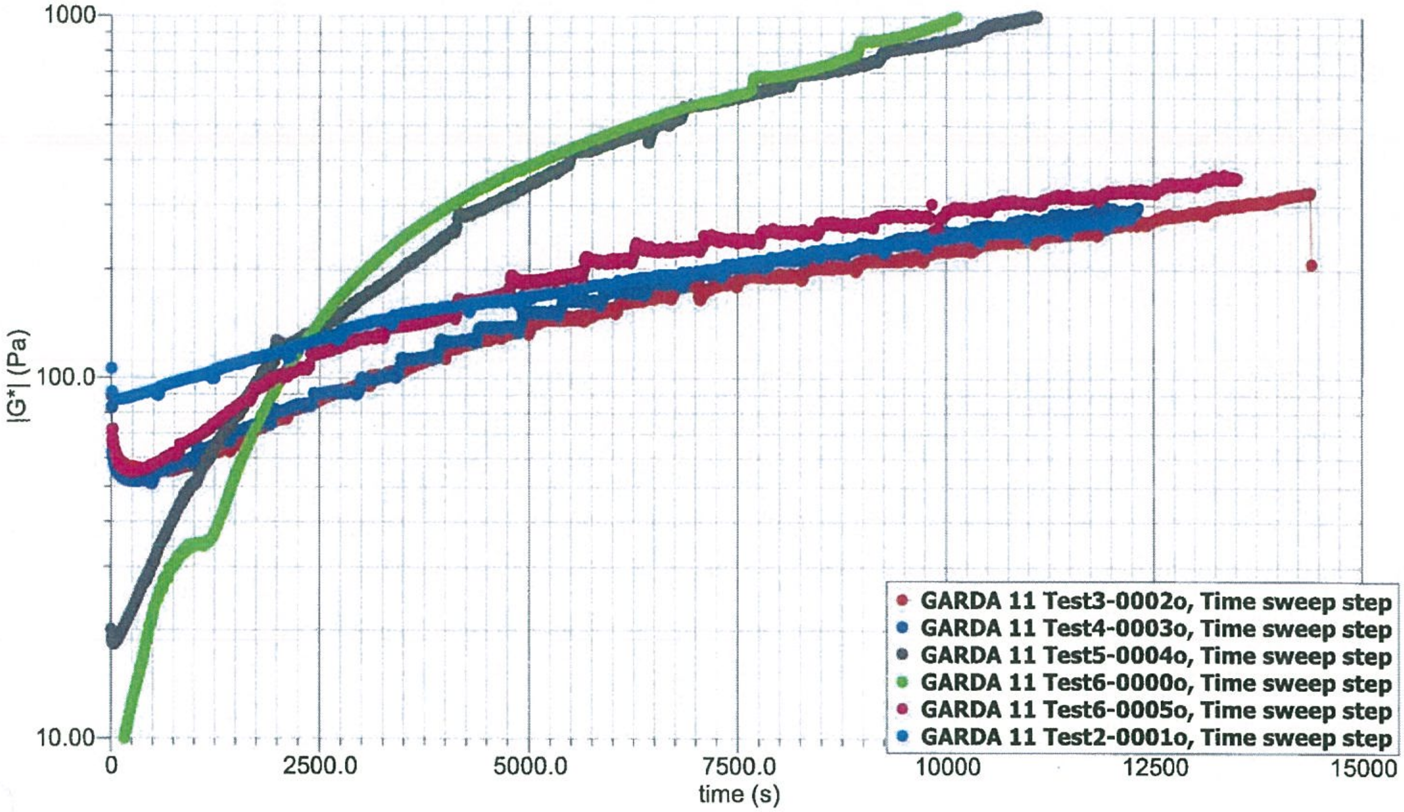
CRADA 8 Curing



9/4/2014

113

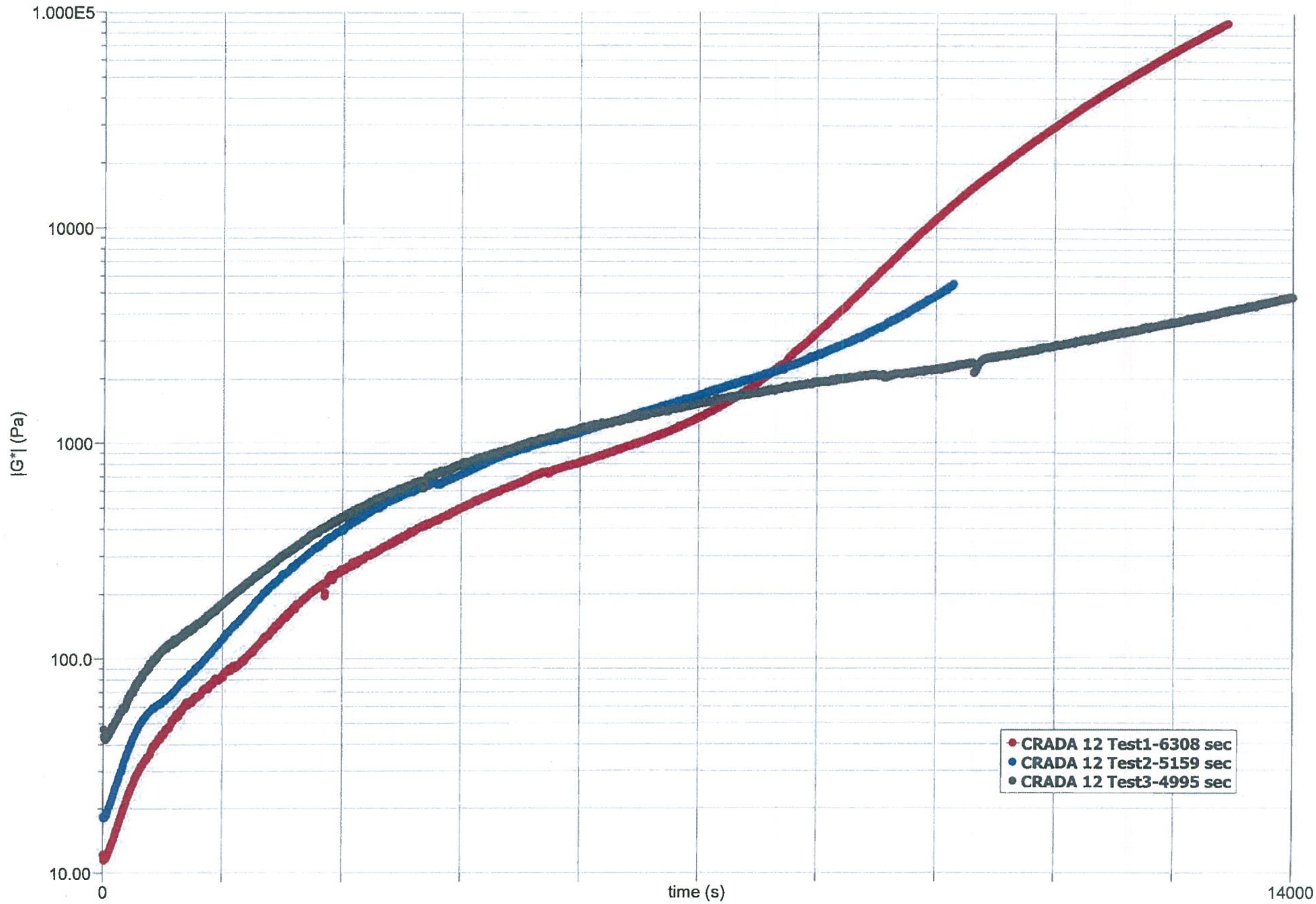
GARDA 11 Test3-0002o



CRADA 11 b

	T1	T2	T3	T4	T5	T6
Part A Viscosity						
	1213	20000	1763	1364	870	1560
	1123	19700	1713	1344	844	1530
	1110	19660	1700	1336	836	1522
	1148.67	**19786.67**	**1725.33**	**1348.00**	**850.00**	**1537.33**
#3 spindle Torqe	30 rpm 36.4%	5 rpm 100%	30 rpm 52.8%	50 rpm 68.2%	50 rpm 43.4%	50 rpm; 78%
Flowability of Part A	Flow	Flow	Flow	Flow	Flow	Flow
Mixing Ratio	630.85	631.67	652.49	653.56	651.69	649.81
	111.33	90.23	72.50	72.62	72.40	72.81
Flowability Pot	Flow	Flow	Flow	Flow	Flow	Flow
Dry to touch time in min	11	12	12	10	14	8
Pot Soultion Viscoaity	210	1464	728	455	333	557.1
	205	1434	694	437	319.5	531.4
	205	1420	680	432	319	521.4
	206.67	**1439.33**	**700.67**	**441.33**	**323.83**	**536.63**
Spindle #3 rpm;torque	200; 42%	50; 73.2%	50; 36.4%	100; 45.3%	200; 66.3%	70; 39%
Mechanical Properties						
Modulus N/mm^2	25.349	43.276	34.326	29.755	13.407	27.198
Energy to Break N.mm	455.797	136.881	144.684	192.981	447.558	196.752
Stress @ Break N/mm^2	4.89	6.037	5.739	6.018	5.194	5.996
Elongation %	51.325	14.554	14.672	20.377	43.14	21.16
Trear Strength N/mm	10.02	7.44	5.61	6.54	6.16	7.28
Shore A Hardness	74	87	86	82	78	82
started on 9/24/14	68	85	82	82	78	84
measured on 10/27/14	74	87	87	82	77	82
	75	88	90	81	80	85
	70	85	87	83	77	86
	74	88	89	82	78	78
	74	85	90	84	78	84
	69	83	83	82	79	82
CRADA11b 9/2014	77	86	85	83	82	86
	79	87	84	83	80	79
	77	85	84	81	74	81
	72	92	90	81	76	74
	80	86	85	84	83	84
	75	86	86	82	78	85
	76	87	85	81	77	85
	72	91	82	84	75	81
Ave	74	87	86	82	78	82
SD	3	2	3	1	2	3

CRADA 12 Curing



10/21/2014

CARDA 12

	T1	T2	T3	T4	T5

Part A Viscosity					
	860.00	1138.00	3090.00		
	800.00	1090.00	3060.00		
spindle #3	784.00	1084.00	3055.00		
Average	**814.67**	**1104.00**	**3068.33**		
rpm;Torqe	50; 43.0%	50;56.8%	20; 61.8%		
Flow	good	good	good		

Mixing Ratio	652.92	658.58	650.12		
	72.55	73.17	72.24		

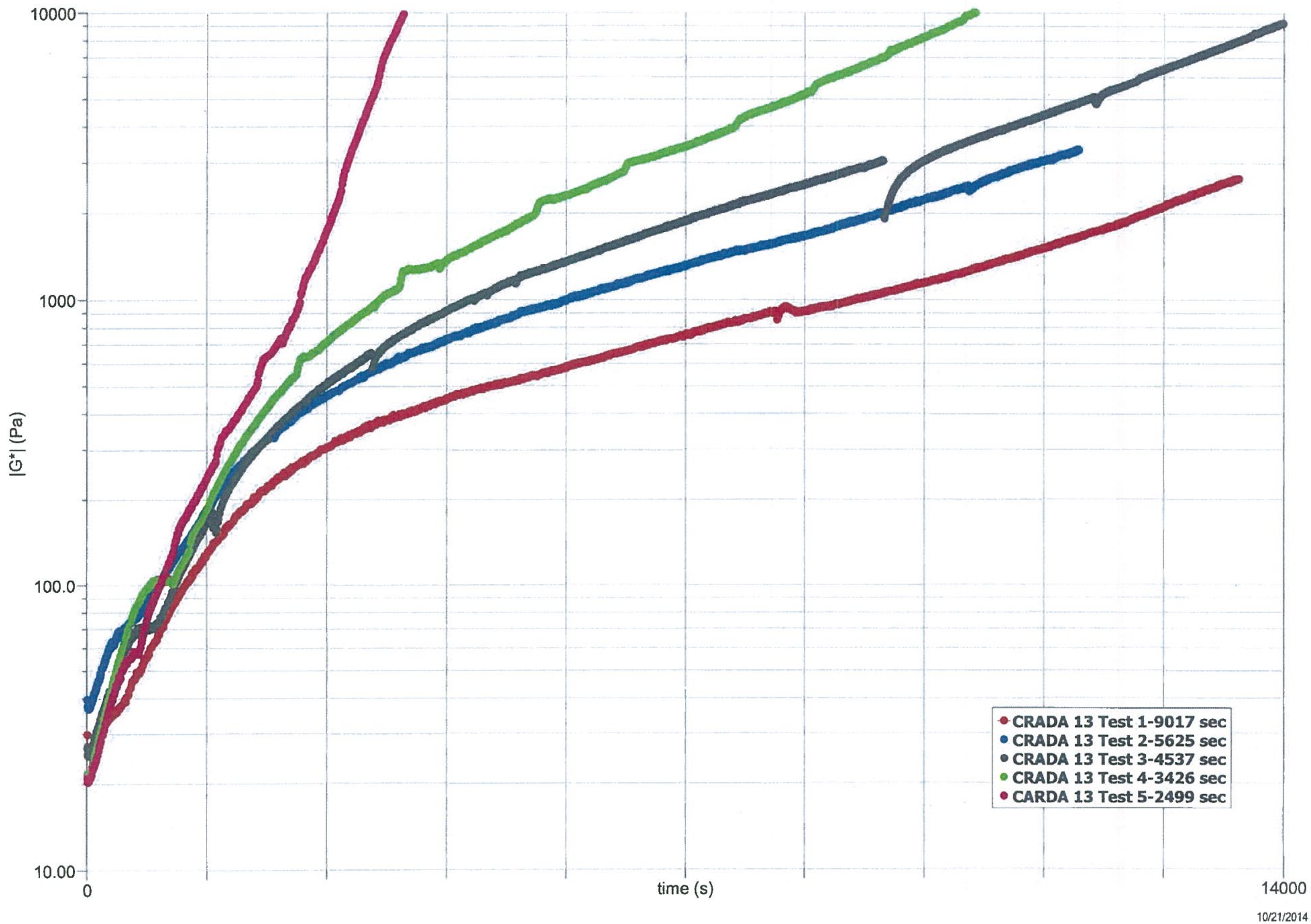
Pot Soultion Viscoaity	221.00	282.00	428.00		
	216.50	263.00	406.00		
	215.50	260.00	401.00		
Average	**217.67**	**268.33**	**411.67**		
Spindle #3 rpm;torque	200; 44.2%	100; 28.2%	20; 61.8%		
Flow	good	good	good		
Leveling	good	good	good		

Dry to touch time min.	~12	~9	~10		
Sag resistance		10			

Was not able to make test4 and test 5

Mechanical Properties					
Modulus N/mm^2	23.537	26.518	20.592		
Energy to Break N.mm	156.129	286.682	212.044		
Stress @ Break N/mm^2	3.522	4.558	3.861		
Elongation %	23.92	30.966	27.76		
Trear Strength N/mm	8.287	8.541	9.116		
Shore A Hardness	73	81	75		
Started on 10/20/14	77	78	77		
Measured on 11/24/14	65	82	73		
	68	79	73		
	65	84	70		
	62	82	75		
	74	80	79		
	81	70	82		
	81	78	78		
	77	84	70		
	66	80	77		
	79	82	75		
	80	83	80		
	84	81	76		
	70	85	75		
	65	81	77		
	76	80	70		
	76	75	77		
	77	84	72		
	72	85	78		
	75	80	79		
	73	78	72		
Ave	73	81	75		
SD	6	4	3		

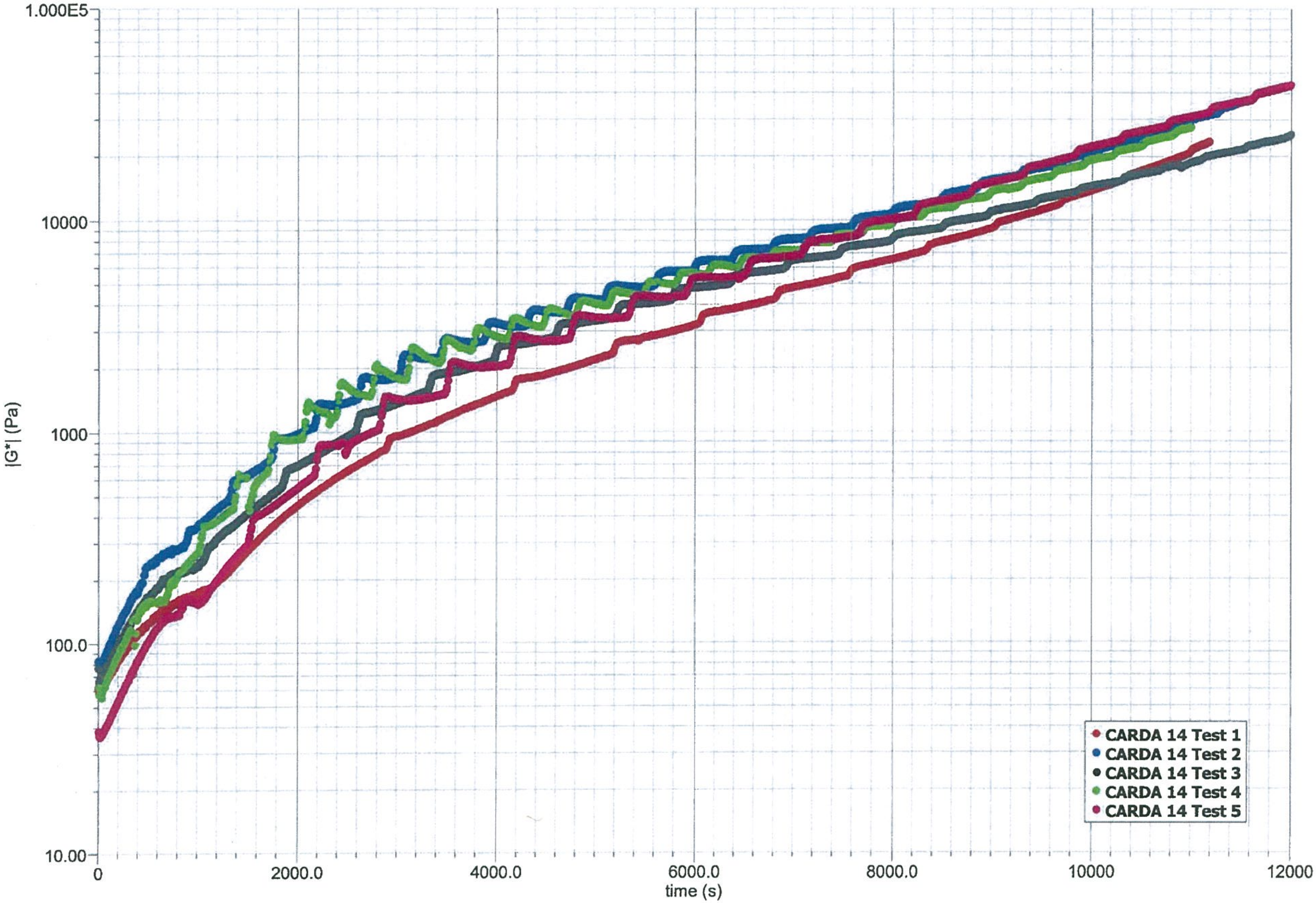
CRADA 13 Curing



CARDA 13

	T1	T2	T3	T4	T5
Part A Viscosity					
	896.00	926.00	1548.00	3300.00	4250.00
	844.00	874.00	1460.00	3085.00	3900.00
spindle #3	828.00	866.00	1444.00	3020.00	3790.00
Average	**856.00**	**888.67**	**1484.00**	**3135.00**	**3980.00**
rpm;Torqe	50; 44.8%	50; 46.3%	50; 77.4%	20; 66.0%	20; 85.0%
Flow	good	good	good	good	good
Mixing Ratio	690.08	688.34	686.91	687.92	685.08
	76.67	76.49	76.32	76.42	76.12
Pot Soultion Viscoaity	221.00	244.50	360.00	421.00	552.00
	216.50	239.00	340.00	402.00	528.00
	215.50	238.00	335.00	395.00	521.00
Average	**217.67**	**240.50**	**345.00**	**406.00**	**533.67**
Spindle #3 rpm;torque	200; 44.2%	200; 48.9%	100; 36.0%	100; 42.1%	100; 55.2%
Flow	good	good	good	good	good
Leveling	good	good	good	good	good
Dry to touch time min.	<10	<12	<12	<13	13
Sagresistance	10+	12+	12+	12+	12++
Mechanical Properties					
Modulus N/mm^2	25.906	23.54	21.563	21.462	20.551
Energy to Break N.mm	190.676	158.991	432.278	640.919	656.55
Stress @ Break N/mm^2	3.572	3.24	4.531	4.864	4.815
Elongation %	26.46	24.545	47.86	68.051	71.365
Trear Strength N/mm	7.597	9.49	9.534	9.49	9.846
Shore A Hardness	76	72	77	72	75
Started on 10/20/2014	77	76	74	81	80
Measured on 11/24/2014	76	71	79	70	72
	82	65	80	65	76
	72	77	80	67	77
	72	71	78	63	79
	83	77	73	73	80
	78	71	72	70	78
	76	66	77	78	65
	71	72	80	68	75
	74	65	80	82	75
	83	74	75	73	74
	80	74	76	79	72
	71	75	80	74	77
	69	67	66	64	73
	71	72	75	64	74
	80	72	80	80	75
	76	76	76	79	76
	70	76	82	65	78
	70	70	80	73	65
	75	70	80	78	77
	81	69	75	74	78
Ave	76	72	77	72	75
SD	5	4	4	6	4

CARDA 14 Curing



11/3/2014

CRADA 14 10/24/2014

Raw Material	T1	T2	T3	T4	T5	T6
Part A Viscosity						
	1840.00	4120.00	18480.00	3952.00	4496.00	
	1740.00	4008.00	17400.00	3824.00	4344.00	
	1724.00	3968.00	17280.00	3792.00	4328.00	
	1768.00	**4032.00**	**17720.00**	**3856.00**	**4389.33**	
#5 spindle; rpm and Torq	100; 46.00%	50; 51.6%	10; 46.2%	50; 49.4%	50; 56.2%	

Flowability of Part A	Flow	Flow	Flow	Flow	Flow	

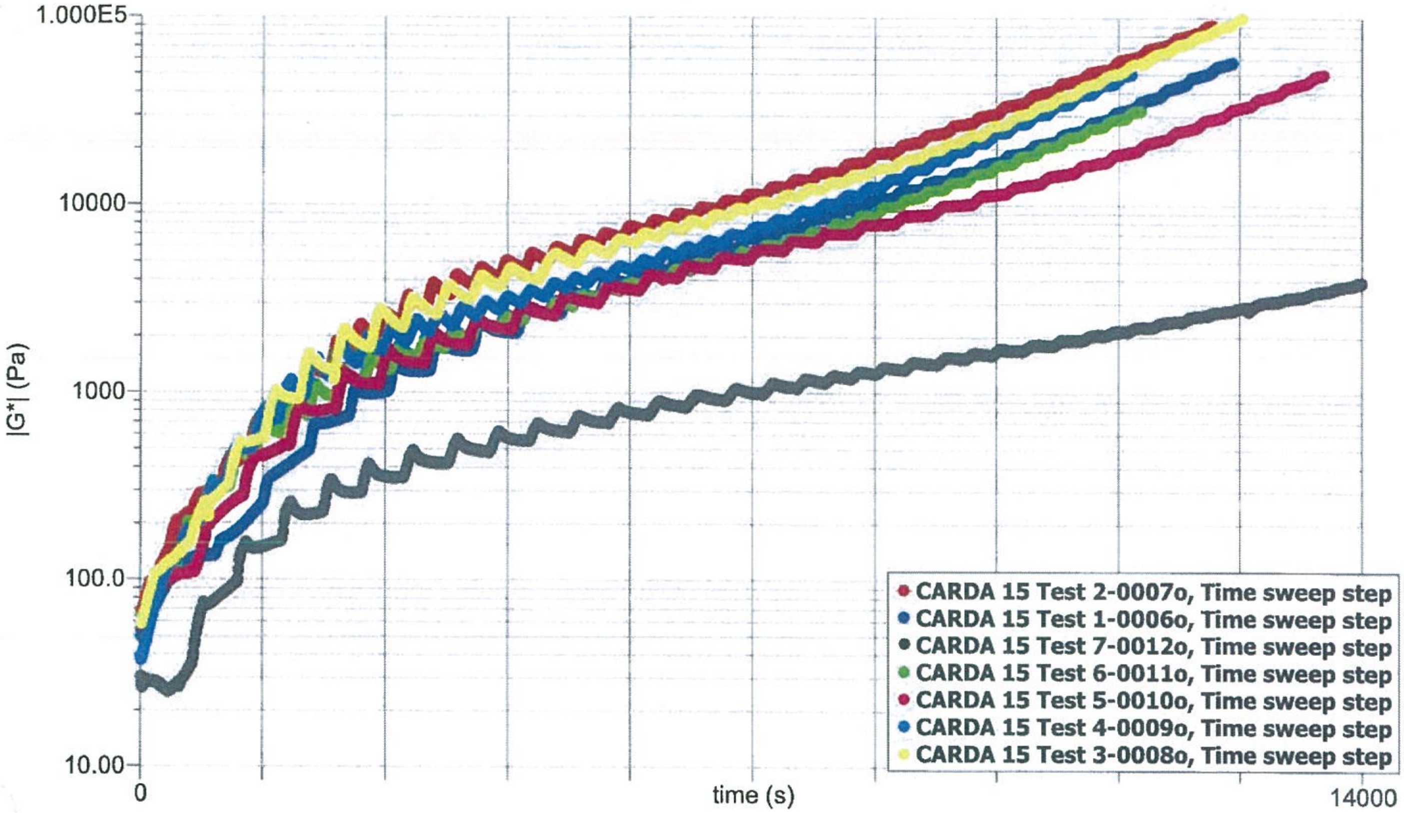
Mixing Ratio	618.28	602.68	608.27	612.29	610.35	
	68.70	66.96	67.57	68.03	67.82	

Flowability Pot	Flow	Flow	Flow	Flow	Flow	
Leveling	Good	Good	Good	Good	Good	
Dry to touch time in mi	14	19	16	16	16	

Pot Soultion Viscoaity	635.70	1212.00	1890.00	870.00	1038.00	
	595.70	1156.00	1807.00	838.00	986.00	
	584.30	1132.00	1777.00	824.00	970.00	
	605.23	**1166.67**	**1824.67**	**844.00**	**998.00**	
Spindle rpm;torque	70; 44.5%	50; 60.6%	30; 56.7%	50; 43.5%	50; 51.9%	

Mechanical Properties						
Modulus N/mm^2	18.844	13.893	23.487	26.339	30.435	
Energy to Break N.mm	565.808	569.740	444.882	503.036	457.473	
Stress @ Break N/mm^2	3.629	3.983	3.802	3.967	3.527	
Elongation %	73.280	70.949	56.665	68.817	62.105	
Trear Strength N/mm	8.741	8.819	9.020	8.798	8.740	
Shore A Hardness	75	71	75	78	78	
Started on 11/3/14	77	74	75	78	76	
Measured on 12/3/14	65	66	78	78	72	
	80	60	73	82	81	
	74	70	70	84	80	
	79	73	73	82	74	
	76	72	70	80	77	
	78	74	74	72	83	
	76	77	73	80	84	
	70	71	73	80	80	
	83	75	73	79	75	
	72	76	77	76	75	
	73	75	74	78	72	
	74	77	77	82	77	
	78	65	78	78	76	
	72	72	72	79	79	
	79	69	76	74	76	
	70	65	78	74	74	
	75	69	75	75	83	
	80	70	77	80	80	
	69	75	72	79	81	
	77	75	78	78	77	
Ave	75	71	75	78	78	
SD	4	5	3	3	4	

CARDA 15 Test 2-0007o



CARDA 17 Surface Properties

	Test1				Test2				Test3			
	Diido	Water	Adv	Reced	Diido	Water	Adv	Reced	Diido	Water	Adv	Reced
	99.91	137.40	148.30	126.60	97.87	136.1	147.5	127.8	101.6	138.2	147	126
	98.28	136.90	150.60	126.80	99.37	137.60	147.20	127.90	101.20	139.00	145.70	124.20
	100.60	136.90	147.10	124.90	99.06	135.20	143.00	122.60	98.08	138.50	147.90	125.40
	99.66	133.20	150.30	126.50	100.40	138.10	146.00	124.10	99.09	136.40	147.80	127.00
	100.80	133.70	149.20	128.00	101.50	138.60	147.60	127.00	98.28	138.00	138.80	114.50
	101.00	134.40	149.00	128.30	102.70	137.90	146.40	126.60	101.00	138.80	142.90	124.80
Avg.	100.04	135.42	149.08	126.85	100.15	137.25	146.28	126.00	99.88	138.15	145.02	123.65
SE	**13.21**	**8.87**			**13.18**	**8.94**			**13.27**	**9.11**		
Disp	**13.21**	**8.66**		**22.23**	**13.18**	**8.62**		**20.28**	**13.27**	**8.72**		**21.37**
Polar	**0.00**	**0.21**			**0.00**	**0.32**			**0.00**	**0.40**		
	WU	**OWRK**			**WU**	**OWRK**			**WU**	**OWRK**		

	Test4			
	Diido	Water	Adv	Reced
	103.4	136.9	143.4	120.6
	98.59	137.30	146.80	122.80
	102.80	137.10	147.60	122.80
	100.90	139.00	147.90	122.3
	99.27	136.80	148.70	123.9
	102.30	139.30	146.20	122.7
Avg.	101.21	137.73	146.77	122.52
SE	**12.81**	**8.55**		
Disp	**12.80**	**8.24**		**24.25**
Polar	**0.00**	**0.30**		
	WU	**OWRK**		

CRADA 18 Pseudo Barnacle Comparison

	CRADA 18 T1	CRADA 18 T2	CRADA 18 T3	CRADA 18 T4	SSC 003-1 Control
Force @ peak (N)	288.245	295.774	264.224	265.123	3.514
Energy to peak (N.m)	0.119	0.1127	0.113	0.113	0.001
dolly dia (mm)	20	20	20	20	20
	DMS S-35	DMS S35+HM 4100	DMS S-33	DMS S33+HM 4100	

CARDA 18 Contact angle mesurement

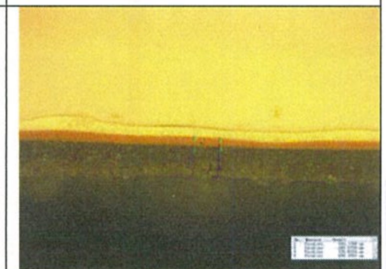
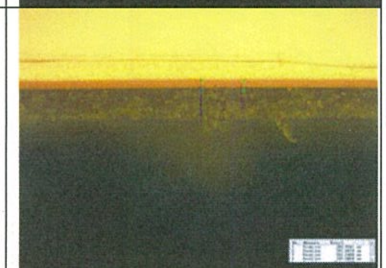
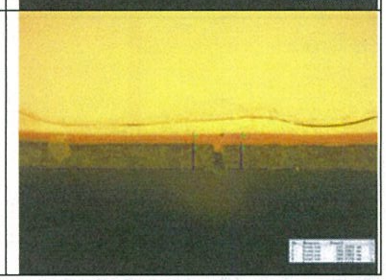
	CARDA 18 T9				CARDA 18 T7				CARDA 18 T5			
	Diido	Water	Adv.	Receding	Diido	Water	Adv.	Receding	Diido	Water	Adv.	Receding
	62.34	106.90	111.10	101.30	95.29	134.00	141.40	109.90	86.66	132.10	139.60	108.90
	62.14	106.70	111.60	101.90	93.25	132.60	139.90	110.20	91.09	133.60	137.10	111.20
	63.78	107.00	111.60	101.00	93.75	136.80	141.10	108.90	88.79	130.10	140.70	110.30
	64.48	107.00	113.50	103.90	94.78	135.90	141.40	102.50	92.90	132.20	142.30	115.10
	63.33	107.20	113.10	103.50	96.48	135.30	142.30	105.90	91.94	132.30	139.10	113.10
	63.40	106.80	111.30	101.70	94.22	136.80	141.50	105.50	93.08	133.20	137.90	106.80
Avg.	63.25	106.93	112.03	102.22	94.63	135.23	141.27	107.15	90.74	132.25	139.45	110.90
SE	**29.37**	**26.76**			**15.16**	**11.17**			**16.64**	**12.77**		
Disp	**28.89**	**26.71**		**9.81**	**15.16**	**10.73**		**35.12**	**16.64**	**12.37**		**28.55**
Polar	**0.48**	**0.05**			**0.00**	**0.44**			**0.00**	**0.40**		
	WU	**OWRK**			**WU**	**OWRK**			**WU**	**OWRK**		

	CRADA 18 T3				CRADA 18 T1							
	Diido	Water	Adv.	Receding	Diido	Water	Adv.	Receding				
	92.65	132.60	135.00	105.80	93.73	132.40	139.30	112.80				
	96.33	132.00	137.90	107.70	97.05	134.60	138.40	112.90				
	94.59	134.40	140.70	112.60	95.65	133.40	146.10	118.90				
	93.92	134.60	138.60	112.00	93.12	133.40	144.10	119.10				
	91.74	135.30	141.00	111.60	93.20	136.60	139.50	110.20				
	95.32	135.00	145.10	113.10	93.48	135.10	141.00	113.20				
Avg.	94.09	133.98	139.72	110.47	94.37	134.25	141.40	114.52				
SE	**15. 36**	**11.32**			**15.26**	**11.21**						
Disp	**15.36**	**10.95**		**29.25**	**15.26**	**10.84**		**26.88**				
Polar	**0.00**	**0.37**			**0.00**	**0.37**						
	WU	**OWRK**			**WU**	**OWRK**						

<u>SSC 127-2a Black Surface Coat</u>

03/30/2015

SSC 8"X8" Tiles

Sample ID	Description/Date	Scratch Test(g)	Tie Coat thickness	Surface coat thickness	Total thickness of both	Scratch Hardness@6mil tie coat & 12mil Surface Coat	Photo
CRADA 18 T1	CRADA 18 T1	1210	2.89	10.85	13.74	1056.77	
CRADA 18 T3	CRADA 18 T1	1260	2.81	10.80	13.61	1110.95	
CRADA 18 T5	CRADA 18 T5	1110	3.50	8.08	11.58	1150.26	

CARDA 19 Work Sheet and results

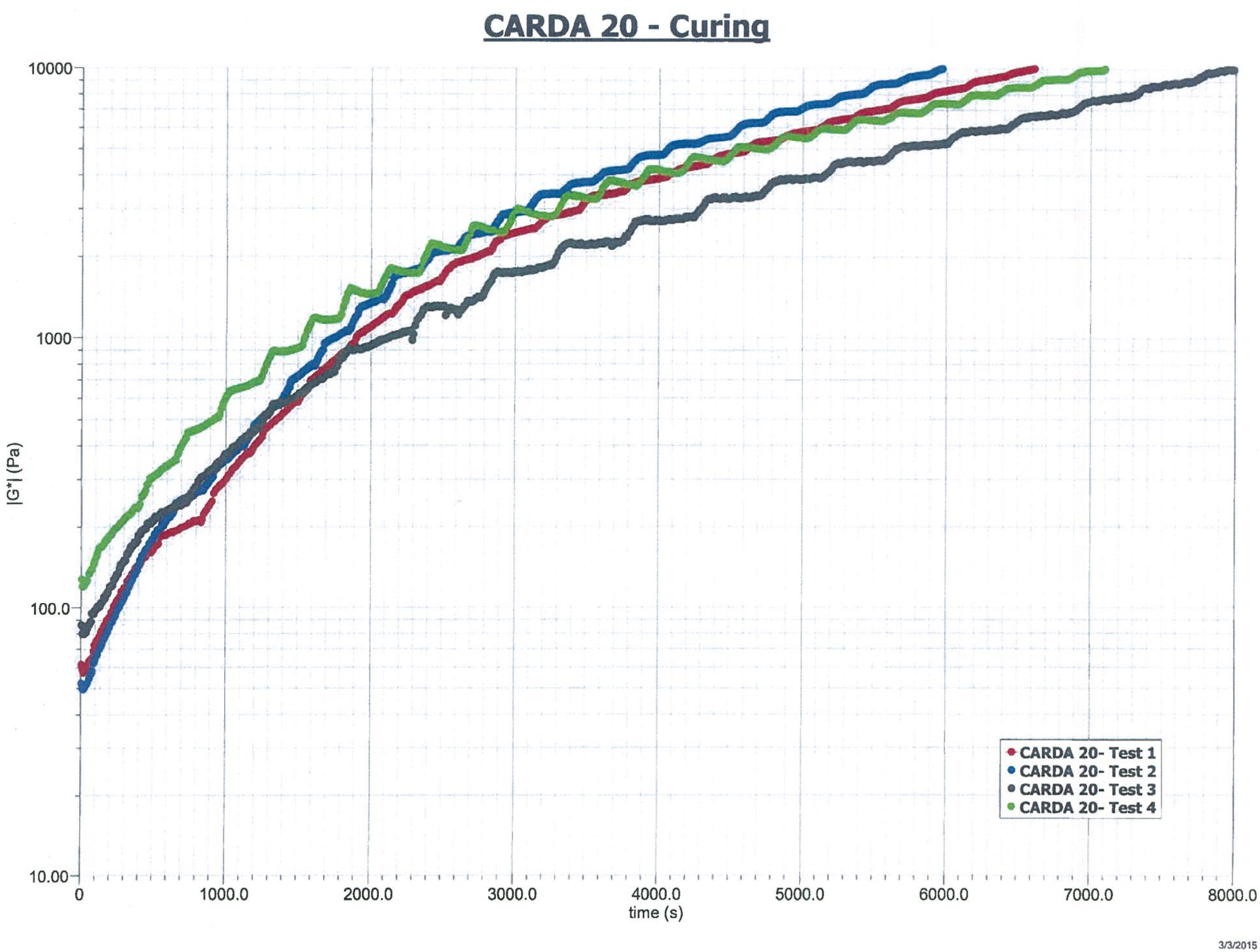
Raw Material	T1	T2	T3	T4
Part A Viscosity				
	2704.00	3872.00	5496.00	9267.00
	2616.00	3624.00	5256.00	8800.00
spindle #5	2560.00	3528.00	5168.00	8653.00
Average	**2626.67**	**3674.67**	**5306.67**	**8906.67**
rpm;Torqe	50; 33.8%	50; 48.4%	50; 68.70%	30; 69.50%
Flow	gel	gel	gel	gel

Mixing Ratio	312.45	319.60	328.18	321.22
	19.94	20.40	20.95	20.50

Pot Soultion Viscoaity			1587.00	2173.00	4533.00	6200.00
			1480.00	1987.00	4093.00	5547.00
			1460.00	1950.00	3960.00	5373.00
Average			**1509.00**	**2036.67**	**4195.33**	**5706.67**
Spindle #3	rpm;torque		30; 47.60%	30; 65.20%	15; 68.00%	15; 92.90%
		Flow	OK	OK	OK	OK
		Leveling	good	good	OK	OK

Dry to touch time min.	less than 15 min			
Sag resistance	16 ++	24	24	24
Pot Soultion Viscoaity				
Fresh	1587.00	2173.00	4533.00	6200.00
15 min	1470.00	1960.00	3947.00	5333.00
30 min	1583.00	2113.00	4140.00	5580.00
45 min	1777.00	2370.00	4533.00	6100.00
60 min	2043.00	2740.00	5093.00	6773.00

Mechanical Properties				
Modulus N/mm^2	41.663	34.397	31.830	35.672
Energy to Break N.mm	290.475	330.688	237.407	281.914
Stress @ Break N/mm^2	3.308	3.198	3.313	3.967
Elongation %	41.248	41.967	35.787	40.469
Trear Strength N/mm	9.031	9.014	8.971	9.200
Shore A Hardness				


CARDA 20 - Curing
10000
1000
100.0
10.00
|G*| (Pa)
0
1000.0
2000.0
3000.0
4000.0
5000.0
6000.0
7000.0
8000.0
time (s)
CARDA 20- Test 1
CARDA 20- Test 2
CARDA 20- Test 3
CARDA 20- Test 4
3/3/2015

CRADA 20

	T1	T2	T3	T4

Part A Viscosity				
	3448.00	3488.00	3560.00	4016.00
	3224.00	3248.00	3416.00	3704.00
spindle #5	3176.00	3208.00	3392.00	3640.00
Average	**3282.67**	**3314.67**	**3456.00**	**3786.67**
rpm;Torqe	50; 43.10%	50; 43.60%	50; 44.50%	50; 50.20%
Flow	good	good	good	good
Mixing Ratio	310.95	316.33	317.73	326.87
	19.85	20.19	20.28	20.86

Pot Soultion Viscoaity						
			2060.00	1583.00	1833.00	1893.00
			1823.00	1453.00	1687.00	1730.00
			1750.00	1437.00	1623.00	1667.00
Average			**1877.67**	**1491.00**	**1714.33**	**1763.33**
Spindle #3		rpm;torque	30; 61.0%	30; 47.50%	30; 55.0%	30; 56.70%
		Flow	good	good	good	good
		Leveling	good	good	good	OK

Dry to touch time min.	less tha n 15 min			
Sag resistance	24	24	24	24

Pot Soultion Viscoaity				
Fresh	2060.00	1583.00	1833.00	1893.00
15 min	1727.00	1457.00	1603.00	1657.00
30 min	1760.00	1600.00	1630.00	1690.00
45 min	1880.00	1817.00	1707.00	1767.00
60 min	2057.00	2117.00	1813.00	1873.00

Color Measurements	L*	41.50	37.96	35.03	34.00
	a*	1.16	1.17	1.16	1.17
	b*	2.05	1.91	1.75	1.75

Mechanical Properties				
Modulus N/mm^2	47.454	38.193	39.922	37.195
Energy to Break N.mm	290.049	309.775	232.170	266.891
Stress @ Break N/mm^2	3.494	3.830	3.450	3.567
Elongation %	38.944	43.933	38.467	42.967
Trear Strength N/mm	9.594	8.848	8.560	8.823
Shore A Hardness				

CRADA 21 Abrasion

No weight added

	1500 strokes			3000 strokes			4500 strokes		
	initial wt	final wt	loss	initial wt	final wt	loss	initial wt	final wt	loss
Test1	144.32	144.27	0.05	144.27	144.26	0.01	144.26	144.23	0.03
Test2	141.85	141.74	0.11	141.74	141.72	0.02	141.72	141.72	0.00
Test3	141.80	141.61	0.19	141.61	141.57	0.04	141.57	141.53	0.04
Test4	139.51	139.45	0.06	139.45	139.43	0.02	139.43	139.43	0.00
Epoxy	143.20	143.19	0.01	143.19	143.18	0.01	143.18	143.18	0.00
Control	139.88	139.84	0.04	139.84	139.83	0.01	139.83	139.83	0.00

500g weight added

	1500 strokes			3000 strokes			4500 strokes		
	initial wt	final wt	loss	initial wt	final wt	loss	initial wt	final wt	loss
Test1	138.93	138.72	0.21	138.72	138.66	0.06	138.66	138.60	0.06
Test2	144.16	143.91	0.25	143.91	143.88	0.03	143.88	143.80	0.08
Test3	142.49	142.14	0.35	142.14	142.12	0.02	142.12	142.04	0.08
Test4	140.75	140.47	0.28	140.47	140.35	0.12	140.35	140.27	0.08
Epoxy	140.28	140.28	0.00	140.28	140.27	0.01	140.27	140.27	0.00
Control	139.13	139.01	0.12	139.01	138.91	0.10	138.91	138.89	0.02

1000g weight added

	1500 strokes			3000 strokes			4500 strokes		
	initial wt	final wt	loss	initial wt	final wt	loss	initial wt	final wt	loss
Test1	139.07	138.71	0.36	138.71	138.43	0.28	138.43	138.38	0.05
Test2	144.20	143.91	0.29	143.91	143.79	0.12	143.79	143.76	0.03
Test3	142.65	142.17	0.48	142.17	142.11	0.06	142.11	141.88	0.23
Test4	144.77	144.33	0.44	144.33	144.14	0.19	144.14	144.09	0.05
Epoxy	142.55	142.55	0.00	142.55	142.55	0.00	142.55	142.55	0.00
Control	138.81	138.62	0.19	138.62	138.55	0.07	138.55	138.51	0.04