

CURING PROPERTIES OF EPOXY RESINS FOR USE TO ABANDON WELLS  
DESTROYED BY HURRICANES IN THE GULF OF MEXICO

A Thesis

by

SUINING GAO

Submitted to the Office of Graduate Studies of  
Texas A&M University  
in partial fulfillment of the requirements for the degree of

MASTER OF SCIENCE

December 2011

Major Subject: Petroleum Engineering

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in the Gulf of Mexico

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Approved by:

Chair of Committee,	Robert H. Lane
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Head of Department,	Stephen Holditch

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## ABSTRACT

Curing Properties of Epoxy Resins for Use to Abandon Wells Destroyed by Hurricanes  
in the Gulf of Mexico. (December 2011)

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Chair of Advisory Committee: Dr. Robert H. Lane

Some Gulf of Mexico (GOM) wells destroyed by hurricanes have become environment and safety hazards and cannot be abandoned by conventional methods since pumping and circulating cement into the casing is impossible when the platforms have been completely destroyed and toppled. This project tested the curing properties of several epoxy resin systems in different environments. A bisphenol-F/epichlorohydrin (BPF) resin cured by curing agent MBOEA system was successfully tested in the laboratory as a potential plugging material to abandon wells destroyed in the GOM. The BPF/MBOEA resin system had the most suitable curing time in a synthetic seawater environment. The system could be successfully weighted by barite up to 16.8 ppg and cured properly. Weighting allows the resin system to fall more efficiently through the casing annulus. This laboratory verification of properties will lead to field test in the test wells.

## DEDICATION

This thesis is dedicated to my wonderful husband, my lovely daughter, and my parents, for their support.

## ACKNOWLEDGEMENTS

I would like to thank my advisor and committee chair, Dr. Robert Lane, for all his guidance, support, and encouragement throughout this project. He always showed me how to get onto the right path.

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## 1. INTRODUCTION

### 1.1 Statement of problem

About 400 offshore oil and gas producing platforms were destroyed or damaged by hurricanes in the past several years in the Gulf of Mexico (GOM) (EnergO Engineering, 2006, 2007, 2010). **Table 1.1** shows the detailed numbers of the wells destroyed or damaged.

TABLE 1.1- NUMBERS OF THE WELLS DESTROYED OR DAMAGED BY HURRICANES IN THE GOM			
Hurricanes	Ivan	Katrina and Rita	Gustav and Ike
Destroyed	7	116	60
Severe Damaged	18	163	31
Destroyed and Damaged	25	279	91
Total		395	

Since those wells have become an environment and safety hazard, they need to be plugged and abandoned. However, conventional abandonment methods may not be possible for some of the wells when the platforms have been completely destroyed and toppled (**Fig. 1.1**). Instead a subsea intervention well may have to be drilled to provide

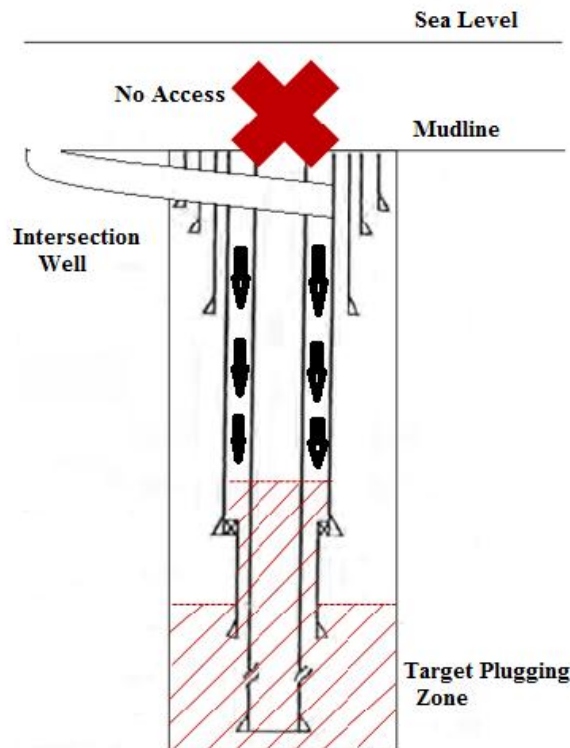
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This thesis follows the style of *SPE Journal*.



**Fig. 1.1—Platform was destroyed by hurricane Ike in the East Cameron Area. (BOEMRE, 2010)**

access to the wellbore (**Fig. 1.2**). The completion type and the well condition may limit or prevent the ability to pull tubing to circulate plugging material into the tubing or the annulus above or between packers. In these cases, the plugging material may have to be spotted at the intersection of the target well and dropped down the well's tubing and annulus through existing packer fluids by gravity. The conventional plugging material, cement, may not be the optimum choice because cement is miscible with seawater. It is probable that many of the offshore wells would be filled with seawater due to its use as a packer fluid and/or entry of additional seawater if the wellbore is breached at or above the mudline during the destructive storm event. The seawater in the casing annulus and tubing would diluent cement as it fall to the bottom, then preventing it from obtaining sufficient compressive strength and bonding strength.



**Fig. 1.2—Schematics of the situation of the wells destroyed by hurricanes.**

This thesis is part of the project funded by BOEMRE which aims to evaluate the use of epoxy resin as well abandonment material to permanently plug the wells destroyed by hurricanes. The current issue with using resins is that it is not yet known how effective they may be as a plugging material. Research must be conducted to determine if resin systems will provide sufficient properties, including suitable curing time and curing behavior, appropriate falling time and effective bonding and compressive strengths, as an alternative well abandonment material.

## 1.2 Literature review

### 1.2.1 Offshore well abandonment technology

When production from a well drops below an economic level, the well will be abandoned temporarily or permanently. *API Bull. E3, Environmental Guidance Document: Well Abandonment and Inactive Well Practices for U.S. Exploration and Production Operations* (API, 1993) provides guidance on environmentally-sound well abandonment practices in the petroleum industry. The main objective in well abandonment is controlling fluid movement to minimize the risk of pollution of the environment. Typical offshore well abandonment steps are described as following (Jordan and Head, 1995):

“Step 1-Blow off tailpipe and deploy tubing non-return-valve (NRV);

Step 2-Bullhead the contents of tubing below the NRV into formation using burst disc plug assembly;

Step 3-Perforate tubing above packer;

Step 4-Run 2nd NRV and set 200ft above packer;

Step 5-Launch 2nd burst disc plug and pump X-linked gel into tubing annulus (barrier/seal for bullheading contents);

Step 6-Bullhead the contents simultaneously of both the tubing and tubing annulus below the back pressure valve;

Step 7-Pump cement into both the tubing and tubing annulus;

Step 8-Squeeze of cement (Monitor Nitrogen pressure);

Step 9-Perforate all upper casing annuli and drain into nitrogen sump;

Step 10-Pump cement into all upper annuli.”

Traditional offshore abandonment operations are time and money consuming. Recently some innovative equipment and technology were developed to enhance safety and lower the cost. Vaucher and Brooks (2010) introduced the inflatable packer bridge plug technology which has been successfully used for temporary or permanent well abandonment in the GOM. Compared with the conventional mechanical packers, the inflatable packers have much more expansion ability and versatility. Olstad and McCormick (2011) developed the new pulling and jacking units which have been used in offshore operations that include intervention and well abandonment to address hurricane damaged platforms in the GOM. These operating systems are the lightweight, modular units with minimal footprint and sufficient hoisting and jacking capabilities. Both of the two operating units have demonstrated an excellent safety record and significant cost savings.

### **1.2.2 Plugging materials used in petroleum industry**

The most conventional plugging material, cement, cannot be the solution to the problem, as described in the statement of problem above. A wide range of unconventional plugging materials is actually used or has been proposed in petroleum industry for well abandonment or zone isolation, including epoxy resins (Gunningham et al., 1992), poly-acrylates (Cowan, 1996), phenol or melamine formaldehyde (De Landro and Attong, 1996) , compressed sodium bentonite (Engleharet et al., 2001), phenolic resins (Abdul-Rahman and Chong, 1997), and room temperature vulcanizing (RTV) silicone rubbers (Bosma et al., 1998). Although effective, some of them have certain



disadvantages to limit their uses in this project. Phenol formaldehyde has a strong exothermic reaction combined with shrinkage, which may affect the seal stability. Polyacrylates have doubtful long-term durability. Compressed sodium bentonite may have a bridge problem in the annulus when it is dropped through packer fluid. RTV silicone rubbers, which have to be used with cements to obtain the sufficient strength, are not suitable in our situation after the cements diluent by seawater. The selection is narrowed down to epoxy resin.

The reasons that epoxy-based resins may be the candidate material are:

1. Epoxy resins are generally not miscible with water.
2. Epoxy resins have high compressive and bonding strength after curing.
3. Epoxy resins have good resistance to most chemicals, including carbon dioxide, hydrochloric acid, and seawater components.
4. Both epoxy resin and curing agents are convenient to handle and store.

### **1.2.3 Epoxy resins**

Epoxy resins are one of a host of plastics developed commercially after 1940s. The special properties that have made epoxy resins successful in a competitive market are their high chemical resistance, low shrinkage, excellent adhesive strength to many substrates, heat resistance, very good electrical properties (Lee and Neville, 1986). The epoxy resin is formed from two different chemicals which are referred to as the “resin” and the “curing agent” (sometimes called “hardener”). When the two chemicals are mixed together, the curing agent polymerizes the liquid resin into hard, inert plastic. The properties of the formed plastics depend on the resin type and the choice of curing agent.

Liquid resin normally is diluted with a diluent which can decrease the viscosity or modify other properties of the resin.

The applications of epoxy resin in petroleum industry began from 1960s. Treadway et al. (1964) used epoxy resins to consolidate loose sand in producing formations. They injected epoxy resin into sand first followed by a fluid to establish permeability, and then injected a hardener-containing fluid to polymerize the resin. Laboratory tests showed that the loose sand which was placed in a cell and subjected to temperatures up to 200 °F and to pressures up to 4,000 psi was consolidated by epoxy resin. The consolidated sand retained about 50% of its original permeability and had high compressive strength even after exposure to brine for one year. The stability of the sand-consolidation resins in hot brine was examined by Rensvold (1983). Several resin systems were tested for durability in hot (160 °F) flowing brine for up to 28 months and in as much as 30 million PV brine. The epoxy resin systems described in the paper were characterized by good strength retention and long term protection against the production of formation sand. Scanning electron microscope (SEM) micrographs of the epoxy resin system showed that the resin system retained high strength after 15 million PV brine flow. The average compressive strength was still good.

Epoxy resins were also used for water control. Gunningham et al. (1992) developed an epoxy resin system which can be used with through-tubing straddle packers to treat intervals selectively. This epoxy resin system was claimed to reduce permeability to water by 99% when used for water shut-off purpose. Rice (1991), Ng and Adisa (1997) introduced the field applications in different oil companies (Tenneco/Chevron and Mobil) using coiled tubing placement method to squeeze epoxy resin into offshore gravelpack

production wells for water shut-off. Besides those applications mentioned above, epoxy resin was used as coating material for underwater and wet surface application (Dhanalakshmi et al., 1997), casing repair (Ng, 1994) and plugging wells (Bosma et al., 2004).

As previously described, curing properties of the epoxy resin system are determined by the type of resin and curing agent. There are two types of resins used in industry commonly, bisphenol-A/epichlorohydrin (BPA resin) and bisphenol-F/epichlorohydrin (BPF) resin, which are synthesized by reacting bisphenol-A or bisphenol-F with epichlorohydrin in presence of a basic catalyst (Lee and Neville, 1986). The BPF resin usually has greater heat and chemical resistance compared to the BPA resin, but also it is more expensive than the BPA one.

TABLE 1.2–COMMON ANINES AND AMIDES CURING AGENTS		
	Advantage	Disadvantage
Aliphatic Amine	good resistance to heat and chemicals	poor flexibility, reaction with moisture
Cycloaliphatic Amine	good resistance to chemicals, solvents and water	short pot life
Aromatic Amine	good physical properties, high resistance to heat and chemicals.	dark color
Polyamide and Amidoamine	excellent adhesion, water resistance and flexibility	low resistance to chemicals, solvents and acids

Selection of the curing agent plays the major role in determining the properties of the final cured resin system. Curing agents generally base upon amines or amides (Sen, 2000). Some of the most common amines and amides curing agents are listed in **Table 1.2**.

Besides the resin and the curing agent, the epoxy resin system usually also contains a reactive diluent which is added to decrease the viscosity of the system or to increase load capacity for the fillers (Ng et al., 1994). The filler can serve to increase the specific gravity of the epoxy resin system or to extend the volume to decrease the cost for some purposes.

Epoxy resins have been used widely in the petroleum industry. When used as plugging material, the epoxy resin systems are usually transported to the suitable position by through-tubing, dump bailer, work string or coiled-tubing methods. The problem addressed in this study is wellbores cannot be accessed by wireline, work string or coiled tubing due to tubing damage at or above mudline. In such cases, accessing the wellbore may be only through an intervention well, and plugging material may only be placed by bullheading where there is ample opportunity to contact wellbore fluids.

### **1.3 Objective**

The project funded by BORMRE includes several research points, including the curing properties of the epoxy resin, the falling behavior when the resin system drops through wellbore fluids, the effect of weighting material, and the mechanical strength of the cured resin system. In this thesis, we focus on the curing properties of epoxy resin

systems. The objective is to determine the optimum epoxy resin system with a suitable curing time which allows the system fall down through wellbore fluids up to 7,000ft and cure properly.

## 2. EXPERIMENTAL MATERIALS AND PROCEDURES

### 2.1 Chemical materials

#### 2.1.1 Epoxy resins and curing agents

After literature review and consulting with industrial professional, Dr. Hubert Monteiro, we chose two epoxy resins, BPA resin and BPF resin, and two curing agents to test. All the resins, curing agents, and reactive diluents were provided by Royce International Company. The four systems tested in our project are list in **Table 2.1** and the pictures of the four systems are taken when they are just prepared (**Fig. 2.1**).

TABLE 2.1– FOUR EPOXY RESIN SYSTEMS TESTED IN THIS PROJECT				
	BPA SYSTEM 1	BPA SYSTEM 2	BPF SYSTEM 1	BPF SYTEM 2
EPOXY RESIN	RAR 901	RAR 901	RAR 9281	RAR 9281
CURING AGENT	RAC 9907	RAC 9913	RAC 9907	RAC 9913
DILUENT			RAD 100	RAD 100
	EPOXY:CA *	EPOXY:CA	EPOXY:CA:DILUENT	EPOXY:CA:DILUENT
MIX RATIO	100:35	100:29	100:15:43.5	100:15:36

\*:CA=Curing Agent

Two BPA systems are the epoxy resin RAR 901, which is a BPA resin with an epoxide equivalent weight of 180-196 diluted by o-cresyl glycidyl ether (CGE), cured by two different curing agents RAC 9907 and RAC 9913. The BPF systems are the epoxy

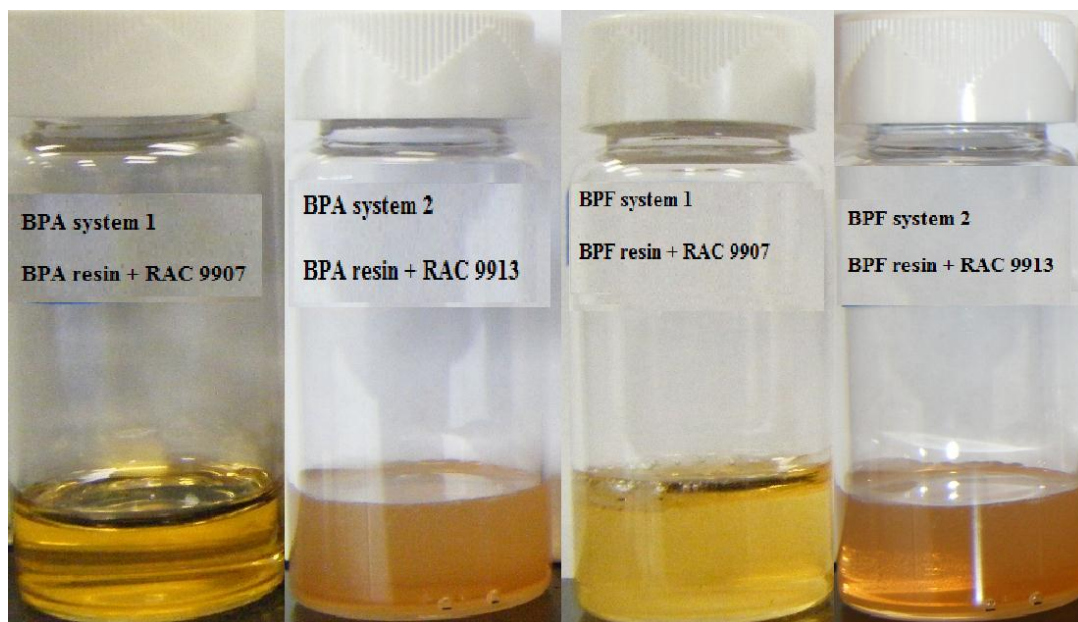


Fig. 2.1—The four epoxy resin systems tested in this project.

TABLE 2.2– PHYSICAL PROPERTIES OF THE COMPONENTS OF THE RESIN SYSTEMS					
	RAR 901	RAR 9281	RAC 9907	RAC 9913	RAD 100
APPEARANCE	Liquid	Liquid	Liquid	Liquid	Liquid
COLOR	Colorless	Pale yellow	Yellow-orange	Yellow-orange	Colorless
TYPE OF ODOR	Faint to slight epoxy odor	Barely perceptible aromatic odor	Perceptible odor	Slight amine odor	Slight characteristic odor
SPECIFIC GRAVITY	1.16	1.20	~1.0	~ 1.0	1.08
VISCOSITY @ 25 °C, cp	450-650	2000-5000	1000-5000	200-300	5-10

resin RAR 9281, which is a BPF resin with an epoxide equivalent weight of 165-175, cured by RAC 9907 and RAC 9913. Among the two curing agents, the RAC 9907 is one of the aromatic amine curing agents and the RAC 9913 is one aromatic/cycloaliphatic amine based curing agent. RAD 100 is the o-CGE diluent with the epoxide equivalent weight of 165-185. The basic physical properties of the components of the system could be got from **Table 2.2**.

### 2.1.2 Weighting material

There are many weighting materials used in resin industry, such like barite, chalk power and hematite. Considering the price and high density, barite becomes the first choice. We obtained the 4-10 micron barite powder from the drilling lab in the Department of Petroleum Engineering of Texas A&M University. **Table 2.3** shows the physical properties of the barite powder used in this project and **Fig. 2.2** is the picture of the barite powder.

TABLE 2.3 - PHYSICAL PROPERTIES OF THE BARITE	
APPEARANCE	Powder, dust.
COLOR	Tan. To Grey.
ODOR	Odorless or no characteristic odor.
SOLUBILITY DESCRIPTION	Insoluble in water.
SPECIFIC GRAVITY	4.22
DENSITY, PPG	35.21





**Fig. 2.2—Barite powder used in this project.**

## **2.2 Experimental conditions**

### **2.2.1 Curing environment**

The epoxy resin systems should cure in the tubing and tubing annuli after falling to bottom if they are to be successfully used as the plugging material. We determined model wellbore conditions by investigating the properties of the GOM. Since the likely packer and wellbore fluid are seawater, the properties of the bottom water of the GOM are necessary. **Table 2.4** shows the geochemical compositions of bottom brine in the GOM (Joye et al., 2005).

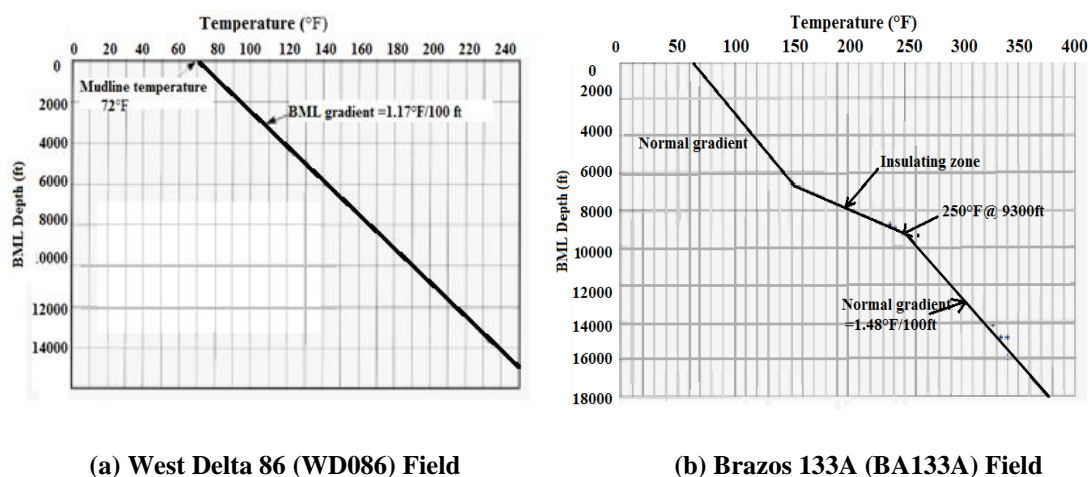
According to **Table 2.4**, we can calculate the types and amounts of the chemicals used to synthesize seawater. For preparation 500 ml synthetic seawater, the amounts of the chemicals are listed in **Table 2.5**.

TABLE 2.4- GEOCHEMICAL COMPOSITIONS OF BOTTOM BRINE IN THE GOM										
(AFTER JOYE ET AL., 2005.)										
Site ID	Salinity	[Cl <sup>-</sup> ]	[Na <sup>+</sup> ]	[K <sup>+</sup> ]	[Ca <sup>2+</sup> ]	[Mg <sup>2+</sup> ]	[SO <sub>4</sub> <sup>2-</sup> ]	[H <sub>2</sub> S]	[Fe <sup>2+</sup> ]	[NH <sub>4</sub> <sup>+</sup> ]
Bottom water <sup>b</sup>	34	564	462	43	11	11	29	~0	~0	0.1
Unit: mMol/L										

TABLE 2.5-COMPONENTS OF SYNTHETIC SEAWATER IN THE PROJECT					
Chemicals	Na <sub>2</sub> SO <sub>4</sub>	NaCl	KCl	CaCl <sub>2</sub> •2H <sub>2</sub> O	MgCl <sub>2</sub> •6H <sub>2</sub> O
Concentration, mM	29	404	43	11	11
Molecular Weight	142.04	58.44	74.55	147.01	203.3
Mass Concentration, g/L	4.119	23.61	3.206	1.617	2.236
Amount, g (for 500ml)	2.06	11.81	1.60	0.81	1.12

From **Fig. 2.3**, we can see the temperature/depth plots of the sands below mudline in different fields in the GOM.

The temperature range from mud line to below mudline 8,000 ft is from 60 °F to 200 °F. Since casing extends from mudline and the packers and completed intervention located in different depths (from mudline to below mudline 8,000 ft), the curing properties of the epoxy resin systems should be tested in the environment with temperature range from 60 °F to at least 200 °F.



**Fig. 2.3—Average temperature/depth plot of sands in two different fields in the GOM, the temperature range at the positions the plugging material placed is from 60 F to 200 F. (After Joye et al.).**

From discussion above, the curing environment condition in the wellbore in the GOM is seawater environment with temperature from 60 F to at least 200 F. To compare with this condition, we also tested the curing properties in air and fresh water environment with same temperatures.

### 2.2.2 The components of the resin system with/without barite

In this project, we tested curing properties of the resin systems with or without barite to evaluate the weighting material effect. We added different amounts of barite to get the epoxy resin system with different densities. **Table 2.6** and **Table 2.7** show the components of the BPA and BPF systems with different densities. In our experiments, we added barite to weight the system up to 16.8 ppg.

<b>TABLE 2.6–COMPONENTS OF BPA SYSTEM WITH DIFFERENT DENSITIES</b>					
RAR 901, g	RAC 9907, g	Barite, g	Specific Gravity (water=1)	Density, ppg	
4.7	1.6	0	1.108	9.25	
4.7	1.6	1.34	1.257	10.49	
4.7	1.6	2.80	1.407	11.74	
4.7	1.6	5.31	1.578	13.17	
4.7	1.6	8.52	1.795	14.98	
4.7	1.6	13.51	2.012	16.79	

<b>TABLE 2.7– COMPONENTS OF BPF SYSTEM WITH DIFFERENT DENSITIES</b>					
RAR 9281, g	RAC 9907, g	RAD 100, g	Barite, g	Specific Gravity (water=1)	Density, ppg
4.0	1.0	1.73	0	1.096	9.15
4.0	1.0	1.73	1.41	1.257	10.49
4.0	1.0	1.73	2.87	1.407	11.74
4.0	1.0	1.73	4.73	1.578	13.17
4.0	1.0	1.73	7.47	1.795	14.98
4.0	1.0	1.73	10.75	2.012	16.79

### 2.2.3 The curing time we prefer

According to the previous tests, conducted by Dr. Schubert's group in Petroleum Engineering department of TAMU (EL-Mallawany, 2010), which are about the falling

behavior of the epoxy resin systems in the water-filled pipe, the epoxy resin system spreads throughout the water column and then recollects at the bottom of the pipe. **Fig. 2.4** shows the falling resin system in the pipe filled with water. The average falling velocities of the resin systems in a water environment are shown in the **Table 2.8**. From



**Fig. 2.4—The epoxy resin system spreads in the water column during falling down.**

this table, we can read that the average falling velocity of the resin systems in the water environment is about 45 ft/min at the first 25ft. Assuming the resin system falling at this velocity, the falling time for the system falling 7,000ft should less than 3 hours. During the falling process, the temperature of the packer fluid is changed from 40 °F at the mudline to around 200 °F at the bottom. It means the average pot life of the epoxy resin

systems under the changed-temperature conditions should be longer than 3 hours. The pot life mentioned here is the length of time that a catalyzed resin system retains a viscosity low enough to be used in processing. The pot life is an important data which shows the time allowed for the epoxy resin system to fall down through the casing and coalesce into a single mass. At the bottom of the casing annulus, the epoxy resin system also needs about 1-hour time to settle and cure. The discussion above gives us criterion as following to determine the suitable systems:

1. From temperature 40 °F to 200 °F, the average pot life should be more than 3 hours.
2. At temperature 200 °F, the pot life should be at least 1 hour.

<b>TABLE 2.8–AVERAGE VELOCITIES OF EPOXY RESIN SYSTEMS WITH DIFFERENT DENSITIES IN ANNULUS WITH DIFFERENT SIZES</b>		
DENSITIES OF THE SYSTEMS, ppg	ANNULUS SIZES (OD- ID), in	AVERAGE VELOCITIES, ft/min
11.7	6" - 0"	38.44
13.2	6" - 0"	41.00
14.7	6" - 0"	51.25
11.7	6"-1.9"	37.85
13.2	6"-1.9"	42.41
14.7	6"-1.9"	60.00
11.7	6" - 3.5"	36.72
13.2	6" - 3.5"	43.93
14.7	6" - 3.5"	51.25

## **2.3 Experimental procedures**

### **2.3.1 Seawater preparation**

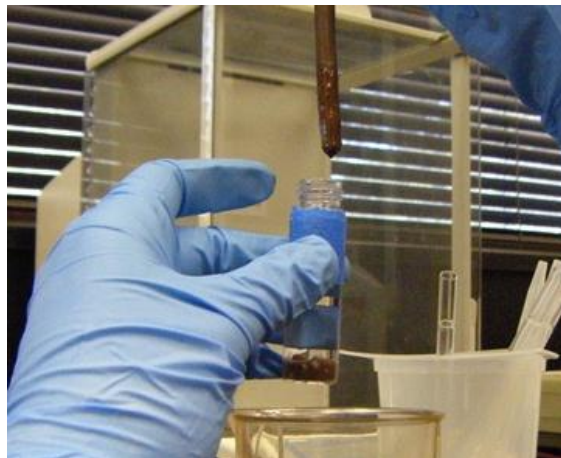
1. Prepare a 500 ml measuring flask and a glass beaker. Make sure the flask and beaker are clean and dry.
2. Measure and add the specific amount of the chemicals listed in **Table 2.5** into the beaker.
3. Add about 100 ml purified water into the beaker and shake the beaker gently until the chemicals dissolved.
4. Pour the water from the beaker into the flask.
5. Add small amount of purified water into the beaker and shake gently, then pour the water into the flask.
6. Repeat step 5 several times.
7. Add purified water into the measuring flask until the liquid is 500 ml. Shake the flask gently to make sure the chemical dissolved evenly.

### **2.3.2 Resin systems preparation**

1. Prepare several beakers and make sure the bakers are clean.
2. Add the epoxy resin into the beakers.
3. Add the reactive diluent if needed into the beakers.
4. Add the barite if the system needed weighting into the beakers.
5. Add the curing agents into the beakers.
6. Stir all the components hardly until the components mixed evenly.

### 2.3.3 Curing time tests

1. Prepare several clean 20-ml vials.
2. Use pipets to put about 10 ml purified water or seawater prepared in section 2.3.1 if the test curing environment are fresh water or seawater environments.
3. Decide the test temperature and pre-heat the oven to the test temperature.
4. Put the vials prepared in step 2 into the oven to be pre-heated about 10 minutes.
5. Get the vials out of the oven and put the epoxy resin samples prepared in section 2.3.2 into the vials (**Fig. 2.5**). Then put the vials back to oven and record the time.



**Fig. 2.5** —Use pipet to put the resin sample into the vials.

6. Check the samples every 15 minutes until the epoxy resin system cured completely. Record the pot life and curing time of the system at the set temperature.



### 2.3.4 Viscosity tests

We used the Brookfield DV-III Ultra Programmable Rheometer (the cone/plate version) to determine the viscosities of these epoxy resins at varying of temperatures. To protect the viscometer, we only tested the samples without barite because the barite may scratch the spindle. There are two types of the viscosity tests conducted in this project.

A. Test the viscosity changes with increasing temperature:

1. Assemble and level the rheometer.
2. Autozero the rheometer.
3. Select the suitable spindle and enter the spindle number to the rheometer.
4. Attach the spindle to the coupling nut.
5. Use pipet to put sample into the cup and attach the cup to the rheometer. Set the gap between the spindle and cup carefully.
6. Set the temperature the thermostatic water bath system as 30 °C and wait for the temperature stable.
7. Measure the viscosity at the speed of rotation of 5, 10, 20, 30, 50, 100, 250RPM.
8. Record the viscosities and the % torque.
9. Increasing the 10 °C temperature every time and repeat step7 and 8 until the temperature reach 100 °C.

B. Test the viscosity changes with time at the specific temperature:

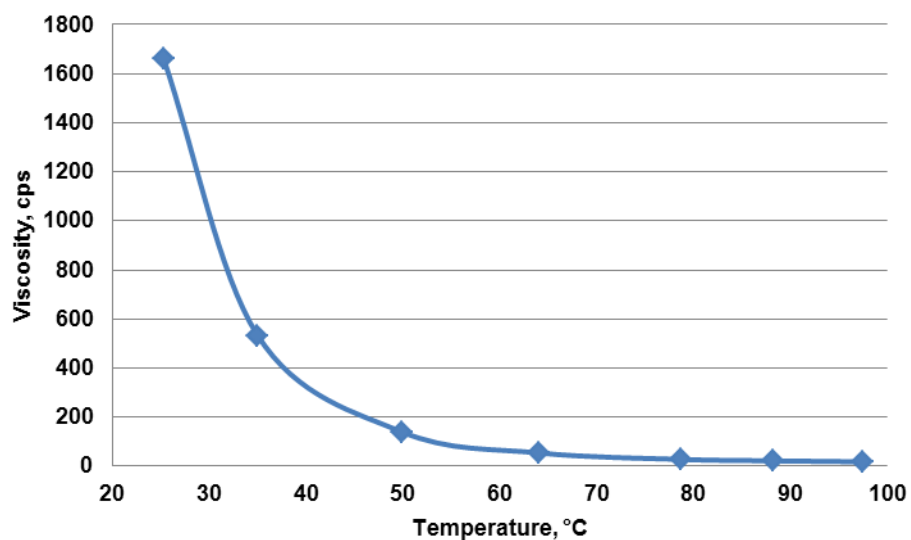
1. Open the temperature the thermostatic water bath system and set the temperature as the specific test temperature (in our project, the temperature is 96.5 °C). Wait for the temperature stable.
2. Autozero the rheometer.

3. Select the suitable spindle and enter the spindle number to the rheometer.
4. Attach the spindle to the coupling nut.
5. Use pipet to put sample into the cup and attach the cup to the rheometer. Set the gap between the spindle and cup carefully.
6. Measure the viscosity at the speed of rotation of 250RPM. Record the time, the viscosity and the % torque.
7. Wait for 5 minutes, repeat step 6 until the viscosity exceed 1000 cp.
8. Get the test sample out of the testing can immediately and use rubbing alcohol to clean the testing can and spindle quickly to avoid the samples cured in the testing can.

### 3. RESULTS AND DISCUSSIONS

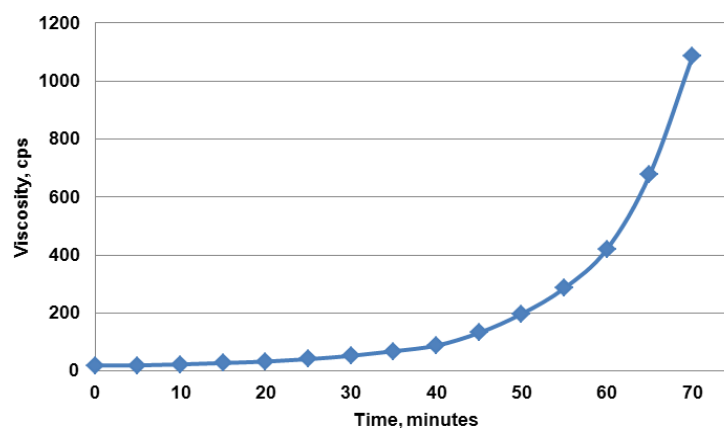
#### 3.1 The viscosity of the resin systems

We measured the viscosities of both the BPA resin system and BPF resin system following the procedure described in section 2.3.4. We did those tests just after we mixed the resin and the curing agent together. **Fig. 3.1** shows the viscosity of the BPA system 1 (BPA resin with curing agent RAC 9907) at temperature from 25 °C to 97 °C. We can see that the viscosity of the resin systems decreases dramatically with increasing temperature. The viscosities of the other three epoxy resin systems and all the components of those four resin systems have similar behavior.



**Fig. 3.1—Viscosity of the BPA resin system 1 decreases with increasing temperature.**

We also tested the viscosity of the resin system changes with time at 96.5 °C. At a specific temperature, after the resin mixed with the curing agent, the viscosity of the mixture becomes larger and larger with increasing time. We tested the viscosity of the BPA system 1 after the two components mixed together at 96.5 °C. **Fig. 3.2** shows the change of the viscosity during the curing process. We stopped the test when the viscosity is more than 1,000 cps because we did not want the epoxy resin cured in the chamber of the viscometer. It is difficult to clean the resins at the end of the curing process.

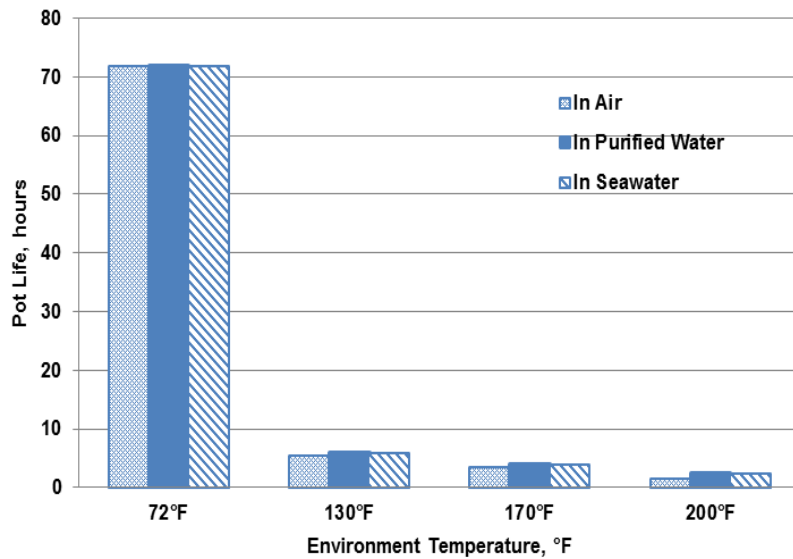


**Fig. 3.2**—The viscosity change of the BPA system a during the curing process at 96.5 °C.

## 3.2 The curing environments effect

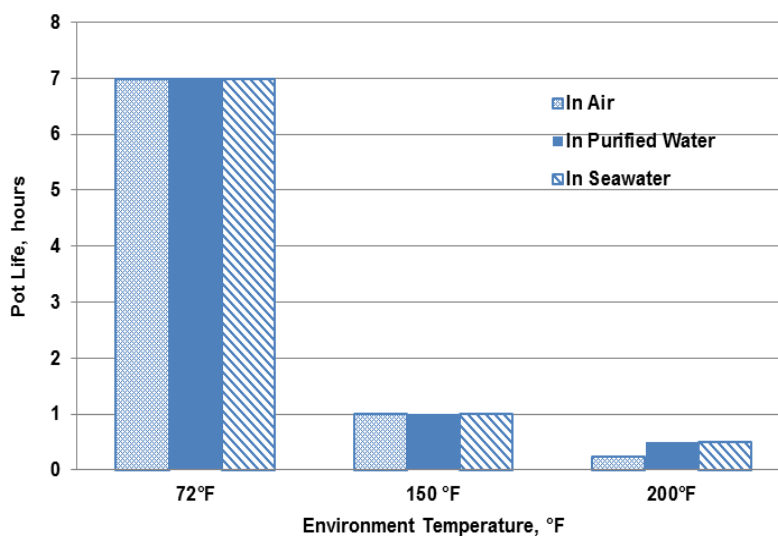
### 3.2.1 Curing time and pot life are temperature sensitive

We tested the pot life and curing time of the four epoxy resin systems at different temperatures in different curing environments included atmosphere, purified water, and synthesized seawater environment. The results are listed in APPENDIX A.



**Fig. 3.3—The pot life of the BPA resin cured by RAC 9907 becomes shorter with increasing temperature.**

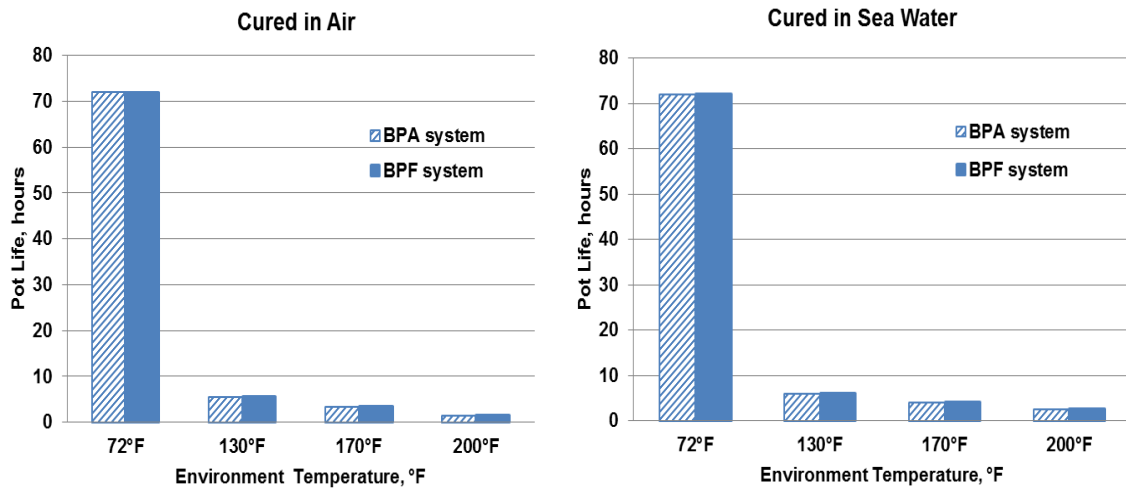
The curing process becomes faster when the system is heated. So the pot life of the system is much shorter when the temperature is higher no matter what kind of curing environment and what kind of curing agent. We could see the tendency clearly from **Fig. 3.3** and **Fig. 3.4**, the pot life of the two BPA systems become shorter when the temperature increases in three different curing environments. The BPF systems also have the same tendency.



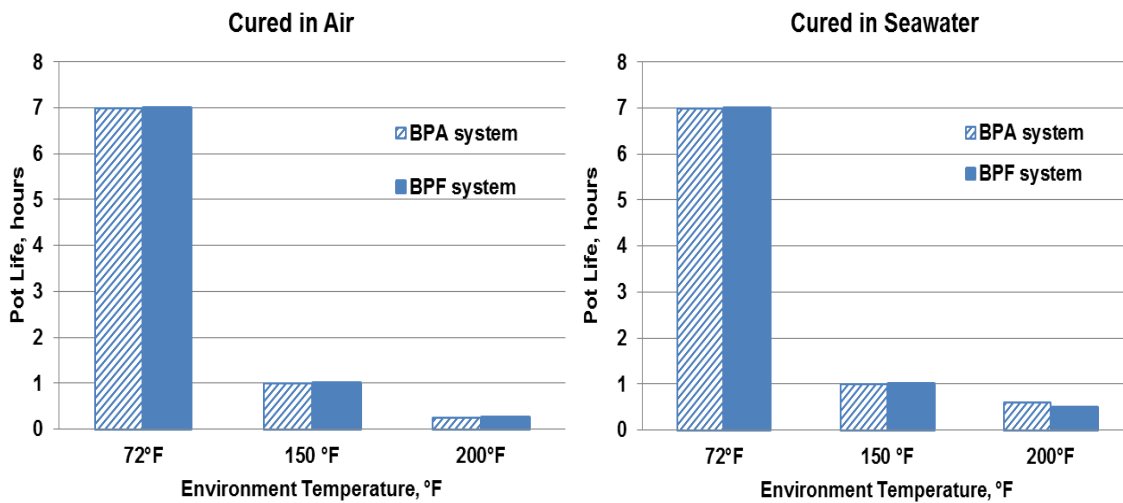
**Fig. 3.4—The pot life of the BPA resin cured by RAC 9913 decreases when the temperature increases.**

### **3.2.2 The curing time and pot life are determined by the curing agents mainly**

We compared the curing time and pot life of the different epoxy resins cured by the same curing agent at the same condition. The different epoxy resins cured by the same curing agent have similar pot life and curing time. We can get this conclusion clearly from **Fig. 3.5** and **Fig. 3.6**. In **Fig. 3.5**, the BPA System 1 and BPF System 1, which contain the same curing agent RAC 9907, have the exactly same pot life in both air and seawater environment in different temperatures. In **Fig. 3.6**, BPA and BPF system 2, which also contain the same curing agent RAC 9913, have almost same pot life except when cured in seawater at high temperature. From the figures we could get the conclusion that the curing time and pot life were decided by the choice of curing agent

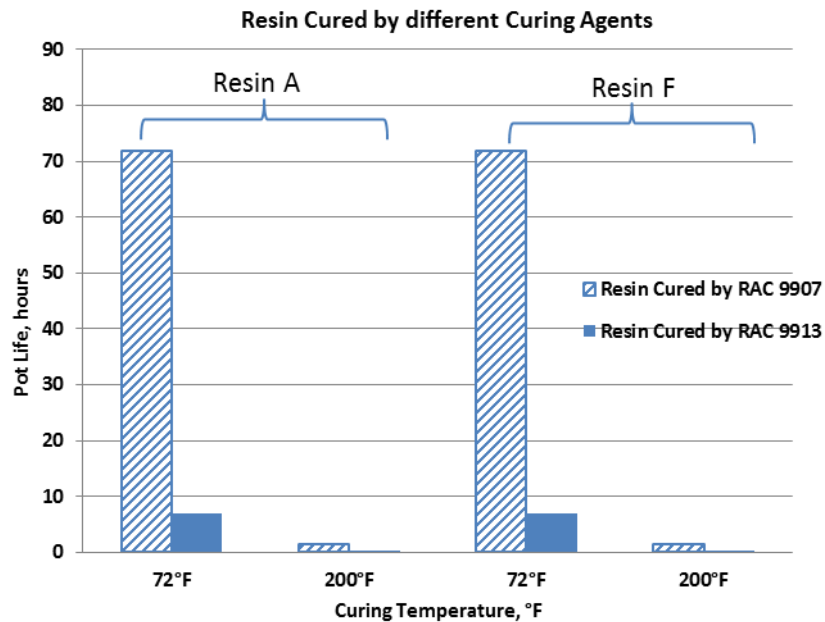


**Fig. 3.5—The BPA resin and BPF resin cured by the same curing agent RAC 9907 have the same pot life in various cured conditions.**



**Fig. 3.6—The BPA resin and BPF resin cured by the same curing agent RAC 9913, have similar pot life in various cured conditions.**

mainly. Different epoxy resins cured by the same curing agent have the similar pot life and curing time.



**Fig. 3.7—The comparison of the two curing agents, RAC 9913 has too short time to make the system falling and curing.**

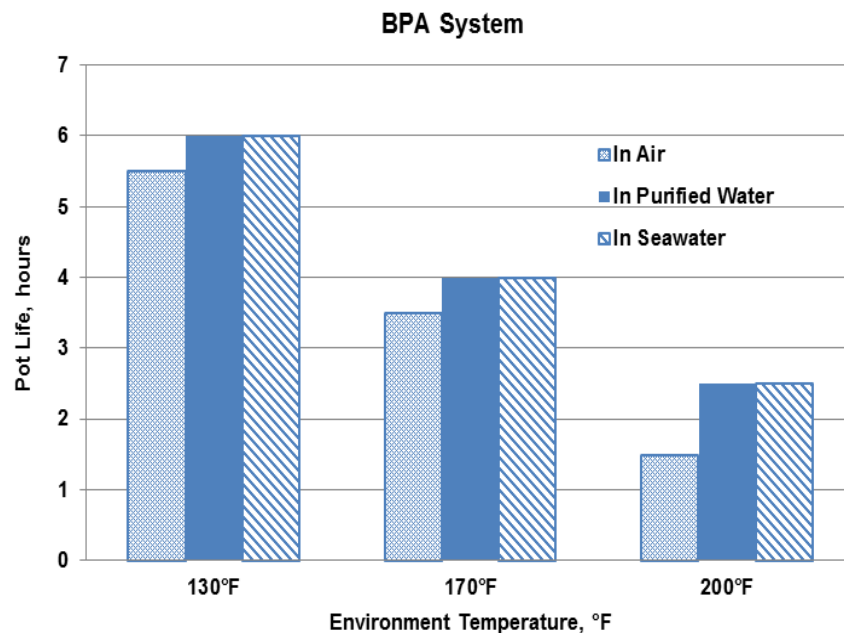
Since the pot life and curing time are determined by the curing agent, we compared the two curing agents we tested to determine the more suitable one. From the **Fig. 3.7**, the systems cured by RAC 9913 have only a few-minute pot life time at high temperature, which is not long enough to meet our requirement which is discussed in section **2.3.3**. Our requirements include at least 3-hours pot life under the environment with temperature changed from 40 °F to 200 °F and 1-hour settling time under the



reservoir condition. The systems cured by RAC 9907 have a suitable pot life which allows the systems to fall down, coalesce and cure at the bottom of the casing. In the following tests, we would choose RAC 9907 as the curing agent and give up RAC 9913. In the following discussion, we use “BPA system” and “BPF system” instead of the original “BPA system 1” and “BPF system 1” because we would not discuss the “BPA system 2” and “BPF system 2”.

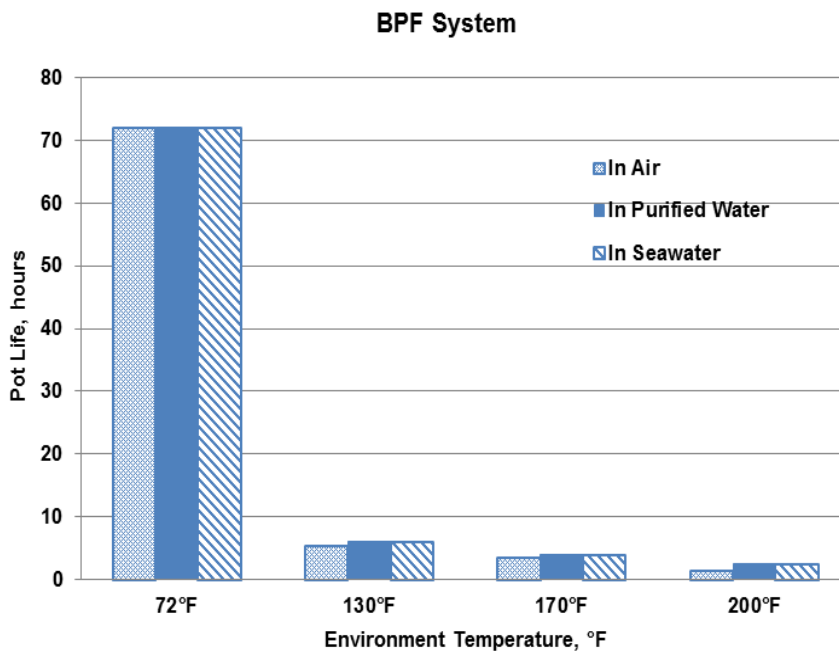
### 3.2.3 The curing environment have effects at high temperature

We would like to discuss the effect of the curing environments. For the BPA system, we could see from the **Fig. 3.3** which is at low temperature the system cured in different



**Fig. 3.8—BPA system has longer pot life in water environments compared with cured in air at elevated temperatures.**

environments have same pot life. And at high temperature, we could see more clearly from **Fig. 3.8**, the systems cured in the purified water and seawater environments have same pot life which is longer than the system cured in air environment. The difference is higher when the temperature is higher. The most difference of the pot life between the system cured in air and in water environment is at the highest temperature 200 °F, which is about 1 hour difference. Compared with temperature, the curing environment has less effect on the pot life and curing time. **Fig. 3.9** shows the pot life of BPF system in different curing environments. The results are similar with the BPA's. The pot lives are same at room temperature and have 0.5 hours difference at elevated temperatures between the systems cured in the air environment and the underwater environments.



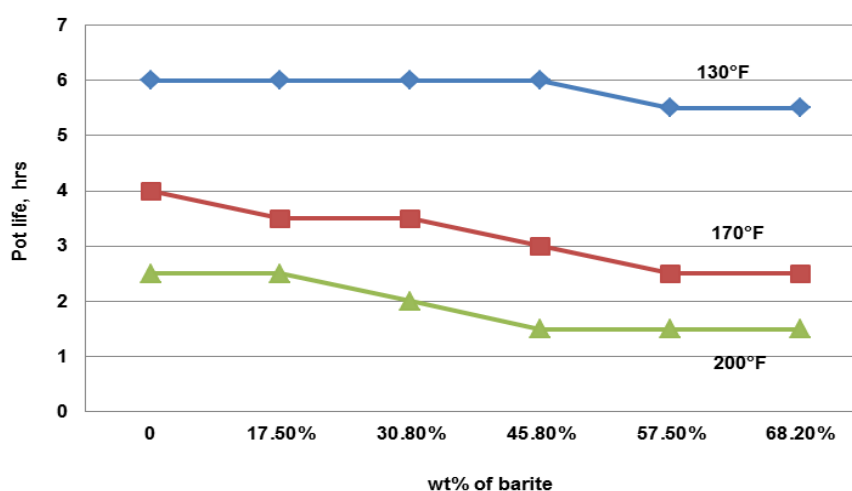
**Fig. 3.9—BPF systems cured in different environments have same pot life at low temperature, and have longer pot life in water environments at high temperature.**

### 3.3 The weighting material effect

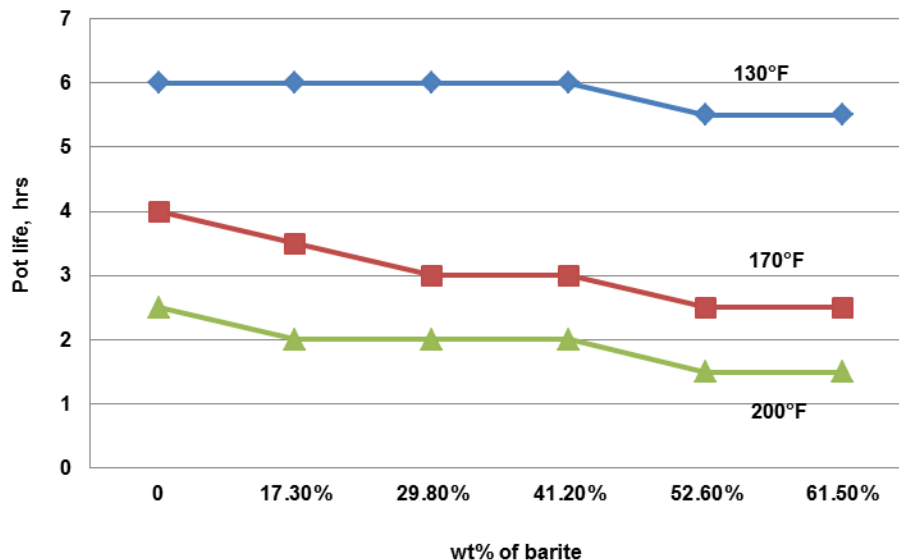
We already knew the effect of the temperature and the curing environments. In this section, we would discuss the effect of the weighting material barite. The weighting material will increase the falling velocity of the resin system due to the increasing density. In this thesis, we only discuss the effect of weighting material on the curing properties of the systems cured in seawater environment. The pot life and curing time of the epoxy resin systems with barite cured in seawater environment are listed in APPENDIX B.

#### 3.3.1 Weighting material shorten the pot life and curing time

First, we would discuss the effect of the weighting material on the curing time of the epoxy resin system. We could get the effect of the weighting material from the **Fig. 3.10** and **Fig. 3.11**.



**Fig. 3.10**—The pot life of the BPA system decreases when the amount of the adding barite increases.

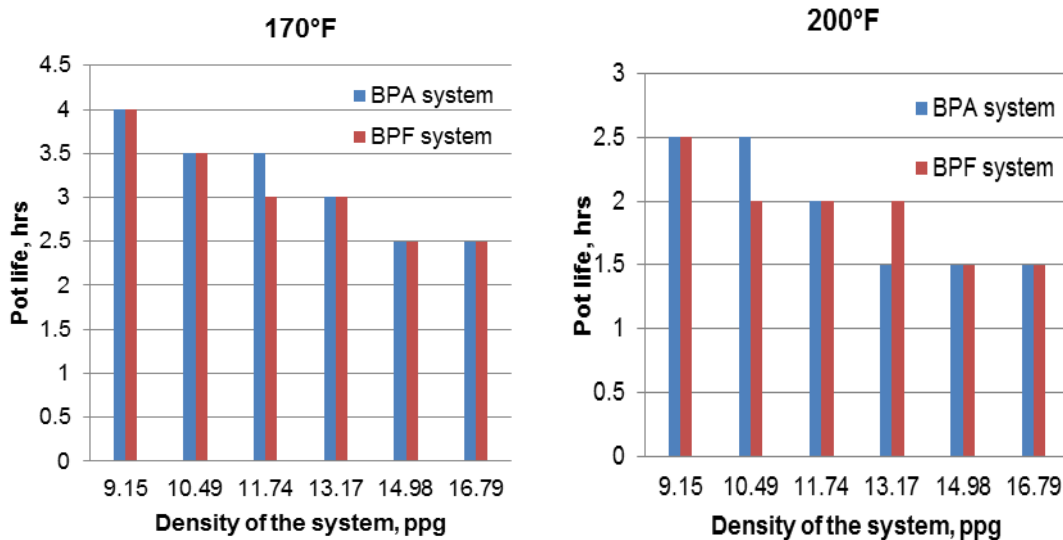


**Fig. 3.11**—The pot life of the BPF system decreases when the amount of the adding barite increases.

The pot lives of both BPA and BPF systems decrease with increasing amount of the barite when the curing temperature and the curing environment are same. The pot life of the system with barite is about 0.5 hour shorter than the one without any filler at temperature 130 °F and below, and more than 1 hour shorter at temperature above 170 °F. At temperature 200 °F, the pot life of the densest system is 1.5 hours. Although it is shorter than the pure system, from **Table 2.8** the falling time of the denser system is also shorter. Thus, the pot life is still enough to meet our requirements.

Now, we would compare the pot life of BPA and BPF systems with barite. At low temperature, 130 °F, the pot lives of those two systems are exactly same. At higher temperatures, 170 °F and 200 °F, the pot lives have a little difference, which we can see

clearly from **Fig. 3.12**. We will discuss other curing properties to decide the better system to meet our requirements.

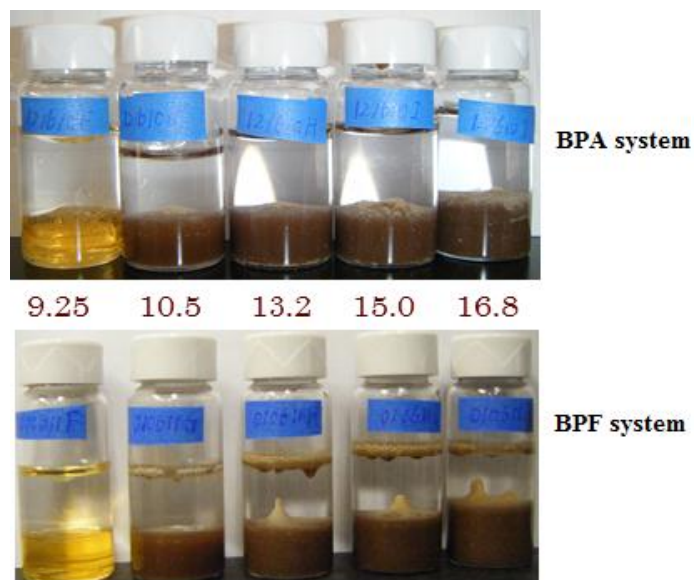


**Fig. 3.12—The BPA and BPF systems with barite have a little different pot life at higher temperatures.**

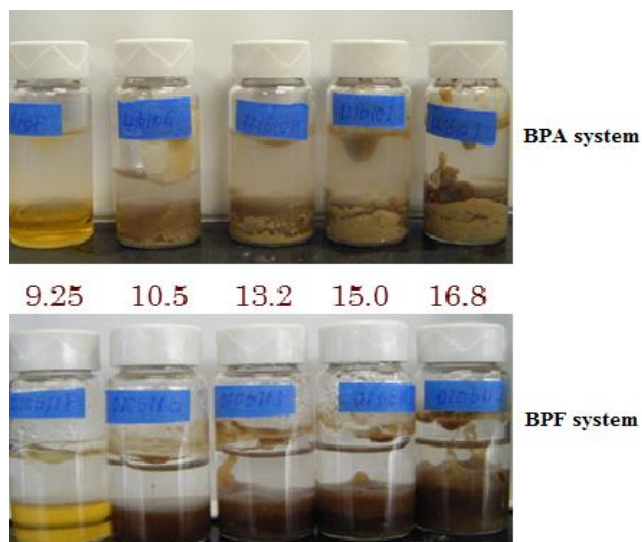
### 3.3.2 Curing properties of the system with barite

The curing time of the BPA system and BPF system with fillers are almost the same. We need to consider other curing properties of those two systems to determine the most suitable one. Now we would like to discuss what happened to those two systems after they are added some barite during the curing process.

From **Fig. 3.13** and **Fig. 3.14**, the barite is distributed evenly in both the BPA and BPF system when they just mixed and stirred into those samples. After the epoxy resin



**Fig. 3.13—The samples of BPA and BPF systems with barite are just prepared, the barite is evenly distributed in the epoxy resin systems.**



**Fig. 3.14—The samples are fully cured. During the curing process, the barite is separated out from BPA system and is still distributed in the BPF system.**

systems are fully cured, the situations are different. The barite came out from the BPA and stay between the cured epoxy resin and the container, which prevents strong bonding between the resin and the bottle. We could easily separate the cured BPA system from the bottle because the bonding is weak meanwhile the bonding between the BPF system and the bottle is much stronger.

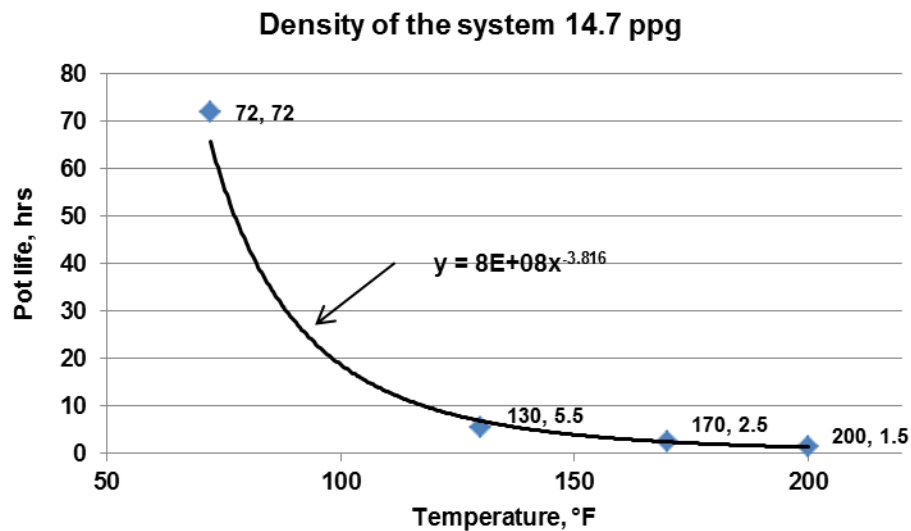
From **Fig. 3.13** and **Fig. 3.14**, the barite is distributed evenly in both the BPA and BPF system when they just mixed and stirred into those samples. After the epoxy resin systems are fully cured, the situations are different. The barite came out from the BPA and stay between the cured epoxy resin and the container, which prevents strong bonding between the resin and the bottle. We could easily separate the cured BPA system from the bottle because the bonding is weak meanwhile the bonding between the BPF system and the bottle is much stronger.

Comparison of the barite effects on both BPA and BPF systems, the BPA system could add less than 10%wt of barite and the BPF system could add as much as 60%wt barite.

### **3.4 Discussions**

Through **Table 2.8**, we get the average falling velocities for the epoxy resin systems with different densities. During the falling tests, they only tested three resin systems with density 11.7 ppg, 13.2 ppg and 14.7 ppg respectively. The average falling velocities for these three systems are 37.7 ft/min, 42.4 ft/min and 54.2 ft/min respectively. We assume these three systems falling through the casing annulus and tubing up to 7,000ft with the corresponding falling velocity constantly. For the BPF

system with density 14.7 ppg cured in synthetic seawater, the tendency of the pot life changed with temperature is shown in **Fig. 3.15**.



**Fig. 3.15**—The trendline of the pot life change with temperature for the BPF system with density 14.7 ppg could be described as  $y = 8E+08x^{-3.816}$ .

Now we discuss about the curing process during the falling. The environment temperature changes during the falling process. We divide the 7,000ft falling distance to 14 even parts while each part has 500ft falling distance. We assume the temperature in each part is constant, which is the average temperature in this part according to **Fig. 2.3**. Also we assume the epoxy resin system instantly reaches the environment temperature present at the given part and we could get the pot life of the system at the given temperature from the trendline in **Fig. 3.15**. For the BPF system whose density is 14.7



TABLE 3.1–FRACTION OF POT LIFE AT EACH 500FT FALLING DISTANCE					
Depth BML, ft	Falling velocity, ft/min	Falling time, hrs	Average temperature, °F	Pot life at given temperature, hrs	Fraction of pot life
0-500	54.2	0.154	77	50.61	0.0030
500-1000	54.2	0.154	81	41.72	0.0037
1000-1500	54.2	0.154	87	31.76	0.0048
1500-2000	54.2	0.154	93	24.62	0.0062
2000-2500	54.2	0.154	99	19.40	0.0079
2500-3000	54.2	0.154	105	15.50	0.0099
3000-3500	54.2	0.154	111	12.54	0.012
3500-4000	54.2	0.154	117	10.25	0.015
4000-4500	54.2	0.154	123	8.47	0.018
4500-5000	54.2	0.154	129	7.06	0.022
5000-5500	54.2	0.154	135	5.94	0.026
5500-6000	54.2	0.154	141	5.03	0.031
6000-6500	54.2	0.154	147	4.29	0.036
6500-7000	54.2	0.154	158	3.26	0.047
7000-7500	54.2	0.154	170	2.46	0.062
7500-8000	54.2	0.154	182	1.90	0.081
8000-8500	54.2	0.154	192	1.55	0.099

ppg, the falling velocity is 54.2 ft/min, which means the falling time of the system in each 500ft part is 9.23 minutes, 0.154 hours. We could get fraction of the pot life by comparing the falling time, 0.154 hours, with the pot life at each 500ft part. We can see the results clearly from **Table 3.1**. If we assume the epoxy resin system falling through the casing annulus from depth 500ft to 7,500ft below mudline, the accumulated fraction

of pot life 0.302. It means there is about 70% pot life left when the epoxy resin system reached the bottom to let the system coalesce. Even the resin system falling from 1,500ft below mudline to 8,500ft, the fraction of the accumulated pot life is less than 0.50. We can use the same method to calculate the other two systems whose densities are 11.7 ppg and 13.2 ppg. We assume they fall from depth 500ft below mudline to 7,500ft. The results are listed in **Table 3.2**. The weighting material could shorten the pot life of the epoxy resin system, but it also could shorten the falling time. From **Table 3.2**, the denser system has less fraction of pot life. It means the overall effect of adding weighting material is favorable.

<b>TABLE 3.2—THE ACCUMULATED FRACTION OF POT LIFE FOR EACH SYSTEM FALLING FROM 500FT TO 7,500FT BELOW MUDLINE</b>			
System Density, ppg	11.7	13.2	14.7
Accumulated fraction of pot life	0.360	0.357	0.302

#### 4. CONCLUSIONS

The curing properties of the four epoxy resin systems were tested. From all the results and discussions, we draw conclusions as follow:

1. Curing properties of epoxy resin systems are consistent with needs for abandoning wells destroyed by hurricanes.
2. The BPF system which contains the RAR 9281 BPF resin, the RAC 9907 curing agent, and RAD 100 reactive diluent is the optimal system we tested in this project. This system has suitable pot life and curing time and large barite capacity which could weigh the system as much as 16.8 ppg.
3. BPF resin system has superior properties compared with BPA resin system. The BPA system which contains the RAR 901 and the RAC 9907 curing agent has suitable pot life, but the small filler capacity means this system could only be weighed to 10.5 ppg. Considering the lower price compared with BPF system, this BPA system may be used in the cases that require low density.
4. Laboratory verification of shear bond strength properties should lead to field test in test wells.

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## APPENDIX A

## POT LIFE AND CURING TIME OF FOUR TESTED EPOXY RESIN SYSTEMS

<b>TABLE A.0.1–CURING TIME OF BPA SYSTEM 1 AT DIFFERENT TEMERATURES</b>					
		72 F	130 F	170 F	200 F
AIR	Pot Life, hr	72	5.5	3.5	1.5
	Cure Time, hr	84	7.5	5.5	2.5
PURIFIED WATE	Pot Life, hr	72	6	4	2.5
	Cure Time, hr	84	8	6	3
SEA WATER	Pot Life, hr	72	6	4	2.5
	Cure Time, hr	84	8	6	3

<b>TABLE A.0.2– CURING TIME OF BPA SYSTEM 2 AT DIFFERENT TEMERATURES</b>					
		72 F	150 F	200 F	
AIR	Pot Life, hr	7	1	0.25	
	Cure Time, hr	16	1.5	0.33	
PURIFIED WATE	Pot Life, hr	7	1	0.5	
	Cure Time, hr	16	1.5	1.2	
SEA WATER	Pot Life, hr	7	1	0.5	
	Cure Time, hr	16	1.5	1.2	



<b>TABLE A.0.3– CURING TIME OF BPF SYSTEM 1 AT DIFFERENT TEMERATURES</b>					
		72 F	130 F	170 F	200 F
AIR	Pot Life, hr	72	5.5	3.5	1.5
	Cure Time, hr	84	7.5	5.5	2.5
PURIFIED WATE	Pot Life, hr	72	6	4	2.5
	Cure Time, hr	84	8	6	3
SEA WATER	Pot Life, hr	72	6	4	2.5
	Cure Time, hr	84	8	6	3

<b>TABLE A.0.4– CURING TIME OF BPF SYSTEM 2 AT DIFFERENT TEMERATURES</b>					
		72 F	150 F	200 F	
AIR	Pot Life, hr	7	1	0.25	
	Cure Time, hr	16	1.5	0.33	
PURIFIED WATE	Pot Life, hr	7	1	0.6	
	Cure Time, hr	16	1.5	1	
SEA WATER	Pot Life, hr	7	1	0.6	
	Cure Time, hr	16	1.5	1	

## APPENDIX B

## POT LIFE AND CURING TIME OF THE EPOXY RESIN SYSTEM WITH BARITE

<b>TABLE B.0.1–CURING TIME OF BPA SYSTEM 1 WITH BARITE IN SEAWATER</b>							
Density, ppg	9.25	10.49	11.74	13.17	14.98	16.79	
Barite wt%	0	17.5%	30.8%	45.8%	57.5%	68.2%	
130 F	Pot Life, hrs	6	6	6	6	5.5	5.5
	Cure Time, hrs	8	8	7.5	7.5	7	7
170 F	Pot Life, hrs	4	3.5	3.5	3	2.5	2.5
	Cure Time, hrs	6	6	6	6	5.5	5.5
200 F	Pot Life, hrs	2.5	2.5	2	1.5	1.5	1.5
	Cure Time, hrs	3	3	3	2.5	2.5	2.5

<b>TABLE B.0.2– CURING TIME OF BPF SYSTEM 1 WITH BARITE IN SEAWATER</b>						
Density, ppg	9.15	10.49	11.74	13.17	14.98	16.79
Barite wt%	0	17.3%	29.8%	41.2%	52.6%	61.5%
130 F	Pot Life, hrs	6	6	6	5.5	5.5
	Cure Time, hrs	8	8	7.5	7.5	7
170 F	Pot Life, hrs	4	3.5	3	3	2.5
	Cure Time, hrs	6	6	6	5.5	5.5
200 F	Pot Life, hrs	2.5	2	2	2	1.5
	Cure Time, hrs	3	3	3	2.5	2.5

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