Product fact sheet

Introduction

This FACT sheet describes procedures which are recommended for the repair of steel storage tanks (i.e. crude oil storage tanks) by a laminating technique, using a laminating system based on LEYCO-POX A resins. To use this technique the following materials are required:

- LEYCO-POX resin laminating binder (i.e. LEYCO-POX A resin / LEYCO-POX B curing agent).
- glass-fibre reinforcement and surfacing tissue.
- a 2-pack LEYCO-POX resin primer.
- suitable gap-filling compositions based on LEYCO-POX A resins, e.g. a trowelling mix and a putty (or knifing stopper).
- a final overseal system (optional).

Lamination system

The system designed for tank repair comprises LEYCO-POX A 171 with curing agents LEYCO-POX B 171 SL and LEYCO-POX B 171. LEYCO-POX A 171 has a medium-low viscosity and both curing agents are of low viscosity. This enables the binder to be applied without difficulty over large areas and ensures good wetting of the glass reinforcement.

Components

LEYCO-POX A 171

LEYCO-POX A 171 is a medium-low viscosity epoxy resin produced from bisphenol A and epichlorohydrin and containing an added proportion of CARDURA™ E10P (glycidylester of neodecanoic acid) as a reactive dIluent. LEYCO-POX A 171 is of low volatility and mild odour which give the resin its agreeable handling characteristics. Further information on LEYCO-POX A 171 is given in the data sheet.

LEYCO-POX B 171 SL and LEYCO-POX B 171

LEYCO-POX B 171 SL and EPTKORE 161 are modified aromatic amine type curing agents of differing reactivity. They can be used alone or in admixture with one another (in any required ratio). In this way the pot life (and curing characteristics) of the laminating binder can be easily adjusted to suit different ambient temperatures. Typical properties of these LEYCO-POX B curing agents are given in Table 1

Table 1: Typical properties of LEYCO-POX B 171 SL and LEYCO-POX B 171

Property	Test method	Unit	Value	
			LEYCO-POX B 171 SL	**LEYCO-POX B 171**
Appearance			Brown liquid	Brown liquid
Basic nitrogen content	ASTM D2896	%(m/m)	7.5-8.5	7.5-8.5
Specific gravity at 20 °C	ASTM D792	kg/l	1.12	1.11
Viscosity at 25 °C	ASTM D445	Pa.s	0.4-0.7	0.2-0.4
Flash point (PMCC)	ASTM D93	°C	100	100
Recommended proportion with LEYCO-POX A 171	phr*		50"	50**

* phr - parts w per 100 parts w of resin
** whether used singly or in admixture the amount of total curing agent recommended is 50 phr

Further information is given in the relevant technical data sheets.

Glass reinforcement

Two types of glass fibre are required for tank repair laminate. The main reinforcement is a chopped strand mat of size-coated chopped strands, normally 20 - 50 mm in length, of special low alkali boroslicate glass, evenly distributed in a random pattern and bound together with a chemical binder soluble in the laminating resin. Major manufacturers of glass reinforcements supply mats in various weights per unit area, with different sizes and binders to suit the many end-uses and processing methods of the glass reinforced plastics industry. For tank repair, a mat designed for hand lay-up, sometimes called contact moulding, is required with a size and binder suitable for use with epoxy resins. Normally a 300 g/m2 mat is used, but in some circumstances when a somewhat thicker, stiffer laminate is required a 450 g/m2 mat will be chosen. In addition, a glass surfacing tissue or surfacing mat is used as the final layer. This lightweight material ensures that no strands of chopped glass are exposed at the surface of the laminate; these could act as wicks along which the liquid in the tank could penetrate the laminate. The manufacturers of glass fibre reinforcements usually supply at least two types of surfacing tissue suited to different laminating methods; for tank repair laminates the types designed for hand lay-up (contact moulding) should be used.

Thixotroping agents

For use on vertical surfaces a thixotropic resin/curing agent mix is desirable to prevent sagging or dripping without impairing ease of application. The required thixotropy can be achieved by the addition of up to 5% (on LEYCO-POX A 171weight) of finely divided silica as 'Aerosil' 380. This has a negligible effect on the properties of the cured resin system.

Resin/Curing Agent blends Mixing

Mixing

The resin and curing agent must always be thoroughly mixed to ensure satisfactory cure. LEYCO-POX B 171 SL and LEYCO-POX B 171 are mobile liquids even at low temperatures and can be readily mixed with LEYCO-POX A 171. Initial LEYCO-POX A 171/LEYCO-POX B curing agent mix viscosities at 25°C are given in Table 2 below.

Reactivity

LEYCO-POX B 171 SL is the more reactive of two curing agents. It has good low temperature curing properties so that at temperatures around 23°C an LEYCO-POX A 171/LEYCO-POX B 171 SL mixture has a short pot life and develops a comparatively high exotherm.

LEYCO-POX B 171 is less reactive and gives mixtures with LEYCO-POX A 171 having much longer pot lives, though the cure rates and low temperature curing properties are naturally reduced.

The exotherm characteristics of the two systems at 23°C, measured on 100g masses under near-adiabatic conditions, are given in Table 2.

Table 2: Properties and reactivity of LEYCO-POX A 171 with LEYCO-POX B 171 SL and LEYCO-POX B 171

Property	**Test method**	**Unit**	**Value**	
			LEYCO-POX B 171 SL	**LEYCO-POX B 171**
Blend viscosity at 25 °C		Pa.s	1.2	0.7
Reactivity (1 00 g mass under adiabatic conditions)	DIN 16945			
Time to reach 50 °C		min	49	180
Peak temperature		°C	158	119
Time to peak		min	75	260
Tecam geltime at 23 °C, 100 g mass	BS 2782	min	81	440

Curing agent ratio

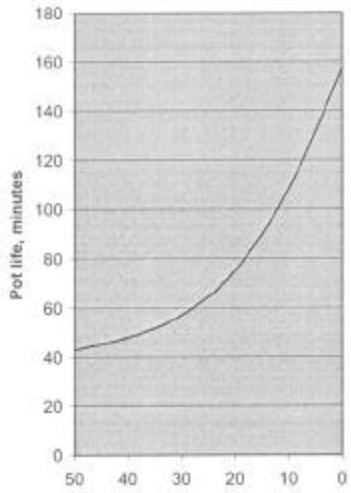
The proportions of LEYCO-POX B 171 SL and LEYCO-POX B 171 employed will depend on the balance required between pot life and speed of cure, as influenced by the ambient temperature prevailing during application and cure of the laminate. Only in exceptional cases will the thickness of the laminate influence the exotherm developed.

LEYCOCHEM LEYDE GMBH
D-50999 Köln • Industriestr. 155
Tel.: +49 (0) 2236 - 9 66 00 – 0
Fax: +49 (0) 2236 - 9 66 00 – 10
leycochem@leyde.com
www.leyde.com

Figure 1 shows the effect of different curing agent ratios on the pot life of 100g quantities (kept under near-adiabatic conditions) of LEYCO-POX A 171 with LEYCO-POX B 171 SL and LEYCO-POX B 171 mixtures, prepared at 23°C. The pot life is taken as the time for the mix to reach a temperature of 50°C. The total quantity of curing agent used was always 50 phr.

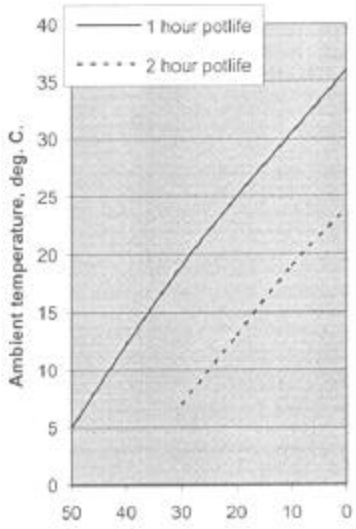
The curing agent ratios required to give a pot life of 1 hour, or 2 hours, at different ambient temperatures (for 100g samples) can be determined from the graphs in Figure 2.

Figure 1: Pot lives of LEYCO-POX B 171 SL/LEYCO-POX B 171 mixtures with LEYCO-POX A 171(100 g mass)



LEYCO-POX B 171 SL phr

Figure 2: Ratio LEYCO-POX B 171 SL to LEYCO-POX B 171 to give 1 or 2 hours pot life at different temperatures



LEYCO-POX B 171 SL, phr

The total amount of curing agent is always 50 phr. The amount of LEYCO-POX B 171 SL is shown in the figures, the remainder is LEYCO-POX B 171.

Curing rate

At low temperatures, e.g. 5°C, LEYCO-POX B 171 SL will normally be used as the sole curing agent to obtain a reasonable rate of cure. In less cold conditions, a proportion of LEYCO-POX B 171 is usually desirable to increase the pot life of the resin/curing agent mix; the higher the temperature the greater will be the proportion of LEYCO-POX B 171 in most circumstances. For example at temperatures around 20-25°C, a good balance of working life and speed of cure

is provided by a curing agent blend consisting of 30-35% LEYCO-POX B 171 and 65-70% LEYCO-POX B 171 SL. Under these conditions the system will cure satisfactorily in 7-10 days. Except at high ambient temperatures, e.g. 35°C and above, LEYCO-POX B 171 as the sole curing agent may be found rather slow curing, so up to these temperatures a small proportion of LEYCO-POX B 171 SL may be necessary.

Cured system properties

Thin film properties

The following hardness properties were measured on LEYCO-POX A 171 based, 400 micron clear films cured at 23°C.

Table 3: Hardness properties of LEYCO-POX A 171 with LEYCO-POX B 171 SL and LEYCO-POX B 171

Property	**Test method**	**Unit**	**Value**	
			LEYCO-POX B 171 SL	**LEYCO-POX B 171**
Hardness, Konig	DIN 53157			
after 24 hours		s	98	8
after 72 hours		s	162	141
Hardness, Buchholz	DIN 53153			
after 72 hours		83	81	

Mechanical properties of binders

Typical properties of castings of LEYCO-POX A 171 cured with 50 phr of LEYCO-POX B 171 SL or LEYCO-POX B 171 , without glass reinforcement or fillers, are given in the following table 4:

Table 4: Properties of LEYCO-POX A 171 with LEYCO-POX B 171 SL and LEYCO-POX B 171

Property	**Test method**	**Unit**	**Value**	
CURE: 7 DAYS AT 23 °C			**LEYCO-POX B 171 SL**	**LEYCO-POX B 171**
Shore D hardness	DIN 53505		83	82
Vicat softening point	ASTM D1525	°C	45	41
Flexural properties at 23 °C	ISO R178			
Maximum flexural strength		MPa	88	82
Strain at maximum strength		%	5.7	4.6
Strength at 5% strain		MPa	87	81
Flexural modulus		GPa	2.55	2.5
Compressive properties at 23 °C	ISO604			
Maximum compressive strength		MPA	90	81
Compressive modulus		GPA	3.20	2.95
CURE: 7 DAYS AT 23 °C PLUS 1 HOUR AT 100°C				
Vicat softening point	ASTM D 1525	°C	54	48
Flexural properties at 23 °C	ISO R178			
Maximum flexural strength		MPa	92	86
Strain at maximum strength		%	5.2	4.9
Strength at 5% strain		MPA	93	86
Flexural modulus		GPa	280	2.62

Chemical resistance of binders

Castings (65 x 25 x 6 mm size) prepared from LEYCO-POX A 171 and 50 phr of LEYCO-POX B 171 SL or LEYCO-POX B 171, were cured and immersed in a number of reagents at 23°C. The % increases in weight of test pieces, with time, are given in the table 5.

Table 5: Chemical resistance of LEYCO-POX A 171 castings with LEYCO-POX B 171 SL and LEYCO-POX B 171

Property	Test method	Unit	Value	
CURE: 7 DAYS AT 23 °C			**LEYCO-POX B 171 SL**	**LEYCO-POX B 171**
Weight gain after immersion in:		%(m/m)		
Boiling water for				
24 hours			1.2	1.2
30% sulphuric acid for				
7 days			0.4	0.5
28 days			0.8	1.0
70 days			1.6	1.8
10% caustic soda for				
7 days			0.2	0.3
28 days			0.5	0.6
70 days			0.9	1.0
Acetone for				
4 hours			4.6	4.9
24 hours			11.8	12.9
CURE: 7 DAYS AT 23 °C PLUS 1 HOUR AT 100 °C				
Weight gain after immersion in:		%(m/m)		
30% sulphuric acid for				
7 days			0.4	0.5
28 days			0.8	1.0
70 days			1.6	1.8
10% caustic soda for				
7 days			0.3	0.3
28 days			0.5	0.6
70 days			0.9	1.0

Resistance to aviation fuels

When refined products such aviation fuels which must be maintained at high purity are stored in contact with a laminate or a protective coating, the effect of

the product on the laminate or coating is not the only point of importance. Any possible effect of the laminate or coating on the refined product must also be thoroughly tested. Laminates prepared from LEYCO-POX A 171/LEYCO-POX B 171 SL and LEYCO-POX A 171/LEYCO-POX B 171 have been tested in the following manner.

Four steel drums approximately 30 cm diameter and 40 cm deep were degreased and the interiors shot-blasted to a white metal finish. Within one hour, a coat of a 2-pack LEYCO-POX resin primer was applied by brush and allowed to dry overnight. The drums were then lined with a laminate using one layer of 300 g/m2 chopped strand mat plus one layer of surfacing tissue. Two drums were lined with an LEYCO-POX A 171/LEYCO-POX B 171 SL laminate and two with an LEYCO-POX A 171/LEYCO-POX B 171 laminate. The laminates were cured for 2 weeks at room temperature (18-23°C) and tested for porosity before determining fuel resistance.

The effect on aviation fuels storage in contact with the laminates was determined by placing a 1.5 cm layer of water in each drum and then filling with approximately 27 litres of fuel. The fuels tested were Jet A-1 (containing an antistatic additive) and Widecut Jet B. The drums were sealed and stored for six months at room temperature. At the end of this period the fuels were tested for gum content, copper corrosion, water reaction and thermal stability.

Almost identical results were obtained for both fuels and these are summarised below. Although these results indicate that the laminate systems are potentially suitable for aviation fuel service, it is recommended that users intending to repair tanks holding refined fuel should satisfy themselves that the materials are satisfactory in practical conditions of application.

Table 6: Fuel properties of LEYCO-POX A 171 with LEYCO-POX B 171 SL and LEYCO-POX B 171 after 6 months storage

Property	**Test method**	**Unit**	**Value**	
			LEYCO-POX B 171 SL	**LEYCO-POX B 171**
Gum content	ASTM D381	mg/100 ml	1	1
Copper corrosion, 2 hours at 100 °C	ASTM D130		1A	1A
Water reaction	ASTM D1094			
Interface rating			Pass 1B	Pass 1B
Separation rating			Pass 2	Pass 2
Thermal stability	ASTM D2341			
Tube rating at 260 °C			1P	3A
Filter pressure drop		Kpa	< 0.04	< 0.04

Test Methods

ASTM Standards are published by the American Society for Testing and Materials, 100 Barr Harbor Drive, West Conshohocken, USA or can be found under www.astm.org.

SMS methods mentioned are available from Resolution Research Europe BV. Information on a number of SMS methods is given in Bulletins in the EK 1.5 series and can be found under www.resins.com.

DIN Methods are published by Verlag GmbH, 1 Berlin, Burggrafenstrasse 4-7, Germany.

ISO Standards are published under the supervision of the International Standards Organisation.

Additional materials

LEYCO-POX RESIN PRIMER

A properly formulated two-pack LEYCO-POX resin primer should be used to coat the steel substrate prior to filling overlaps, curb angles etc. with putty and trowelling compounds. The major requirements for a primer for this particular service are that a conventional pigmentation, usually a mixture of corrosion inhibiting and inert pigments, should be used; metallic pigmented primers (zinc-rich) are not recommended for use under a repair laminate. A solvent-free system is highly recommended, but if really necessary a solvent-based system can be used. Than the solvent mixture in the paint should be suitable for the conditions of ventilation and temperature inside the tank being repaired; a solvent mixture which evaporates too slowly may lead to solvent being trapped in the primer film when the laminate is applied, while too rapid evaporation will make the paint application difficult.

Suitable primers are available from many paint manufactures, whose directions on mixing, thinning, rates of application and working life of their products should be followed strictly.

FILLED COMPOSITIONS

Two types are generally used in the repair of oil storage tanks. A putty or knifing stopper containing fine filler is used to fill pitted areas and overlap welds; a more heavily filled mix is used to fill the curb angle and the area around rivet heads.

Putty or knifing stopper

This can be purchased as a 2-composition from, for instance, paint manufacturers, or it can be made on site using the resin/curing agent laminating system:

Component	Parts by weight
LEYCO-POX A 171	100
LEYCO-POX B 171 SL/ LEYCO-POX B 171	50
Quartz flour of silica flour (120 mesh size)	200-400

The ratio of LEYCO-POX B 171 SL to LEYCO-POX B 171 is selected as for the laminating mix described earlier i.e. depending on the ambient temperature and the pot life required. The resin and curing agent are thoroughly mixed and the filler is then added, with good stirring, to give a thick mix suitable for application by knife. The addition of up to 5 phr of 'Aerosil' 380 may be necessary if the mix is used on vertical surfaces.

Heavy filled mix

This can be as a towelling compound or, more conveniently made on site using similar resins and curing agent as in the laminating system:

Component	Parts by weight
LEYCO-POX A 171	100
LEYCO-POX B 171 SL/ LEYCO-POX B 171	50
Quartz flour of silica flour (120 mesh size)	750-100

The LEYCO-POX B 171 SL to LEYCO-POX B 171 ratio should be the same as that used for the laminating mix. The resin and curing agent are thoroughly mixed and the sand is then incorporated preferably using a rotating drum mixer. The sand used must be washed, dried and sieved silica sand; the weight of sand incorporated in a specific mix will depend on the grading. If the sand has a particle size distribution predominantly in the 0.2-0.3 mm (60 to 80 mesh ASTM E12) then 750 parts of sand per sand per 150 parts (on weight basis) of resin/curing agent mix is the maximum that can be incorporated. Fully graded sands, with the particle size distribution shown in Figure 3, can be used at the higher loading of 1000 parts weight.

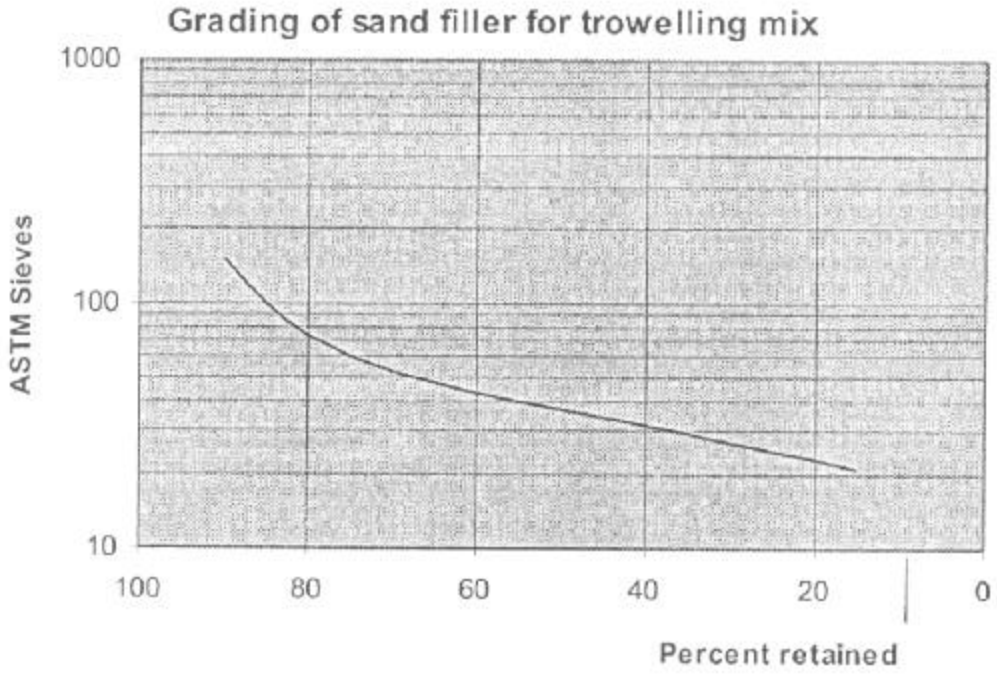


Figure 3: Grading of sand filler for towelling

When using these heavily filled compounds, the ease of application and adhesion after cure are improved by brushing a tack coat of unfilled resin/curing agent mix onto the primed metal and then towelling the sand-filled mix into place before the tack coat gels.

OVERSEAL

A properly prepared laminate made from the recommended LEYCO-POX resin laminating system and chopped strand mat, together with a surfacing tissue, should not require an overseal. However where a completely resin-rich surface is required, an overseal of the resin/curing agent system can be applied, preferably within 24 to 48 hours of laying the laminate. The mixture is the same as that used for laminating:

Component	**Parts by weight**	**Parts by weight**
	Clear finish	**Light coloured finish**
LEYCO-POX A 171	100	100
LEYCO-POX B 171 SL/ LEYCO-POX B 171	50	50
Quartz flour of silica flour (120 mesh size)	0	15

As before, the ratio of the two curing agents is selected to suit the prevailing temperature conditions. The resin and curing agent are thoroughly mixed before application.

If desired a small quantity of titanium dioxide, up to 15% on LEYCO-POX A 171 weight, can be incorporated to provide a light coloured overseal. The pigment should be dispersed in the LEYCO-POX A 171 by roller mill or high-speed, high-shear stirrer. For on-site working the most convenient method is to have available a prepared concentrated paste of pigment in LEYCO-POX A 171; an appropriate quantity of this is stirred into the resin to be used for overseal before adding the curing agent. The quantity of curing agent, of course, must be increased by the amount required to cure the resin in the pigment paste.

However, when a semi-decorative overseal is required, principally to provide lighter conditions in the tank to facilitate inspection, a solvent-thinned LEYCO-POX resin based paint will more frequently be selected when the average ambient temperature is suitable for curing this type of coating. These paints are usually based on LEYCO-POX 124 cured with an amine-adduct or polyaminoarhide and are available from many paint manufacturers. As in the case of the primer the overseal paint must be suitable for use in the ventilation conditions prevailing in the tank without solvent retention in the film. When the tank is to be used for the storage of refined products, the paint must have been tested and approved for this type of service to ensure that no product contamination occurs.

Storage

LEYCO-POX A 171

LEYCO-POX A 171 should be stored in conditions so that moisture is excluded, preferably in the original containers kept tightly closed. Under these conditions and at normal temperatures, the storage life is at least one year. LEYCO-POX A 171 has good resistance to crystallisation but storage at low temperatures may result in some haziness. The resin can be restored to its original condition by gentle warming to 50-60°C. The maximum temperature recommended for pumping or handling liquid LEYCO-POX A 171 is 85-90°C.

LEYCO-POX B 171 SL and LEYCO-POX B 171

These curing agents are storage stable. They should, however, be stored in sealed containers and away from direct sources of heat.

Handling precautions

LEYCO-POX A 171

LEYCO-POX A 171 is a skin irritant on prolonged or repeated contact and can cause sensitisation. Skin and eye contact with this resin should be avoided and the wearing of protective clothing including gloves and goggles or face shield is advisable. LEYCO-POX A 171 has a low volatility at the recommended usage temperatures and any vapour hazard from the resin will be slight. Reference is made to the safety data sheet of this product, which gives up-to-date information about safe handling.

LEYCO-POX B 171 SL and LEYCO-POX B 171

LEYCO-POX B 171 SL and LEYCO-POX B 171 contain 4,4'-diaminodiphenyl methane (MDA), a toxic chemical which should be handled with care. MDA can be adsorbed through the skin in harmful amounts and is also both a mild skin irritant and a sensitizer. LEYCO-POX B 171 SL and LEYCO-POX B 171 are mild eye irritants both through direct contact or by exposure to the vapour.

Particular care must therefore be taken to avoid all contact with LEYCO-POX B 171 SL and LEYCO-POX B 171 in liquid form and with any vapour from the materials. Protective clothing including gloves and goggles or facemask should be worn when handling them. Adequate ventilation, forced if necessary, must be provided when there is any possibility of inhaling vapour or droplets.

It is strongly discouraged to apply this system via spraying.

It is unlikely that LEYCO-POX B 171 SL or LEYCO-POX B 171 will be ingested. However, care must be taken to prevent any accidental contamination of food or crockery because MDA is toxic if ingested.