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SUGGESTED METHODS FOR THE PREPARATION AND TESTING OF VARIOUS PROPERTIES OF RESINS AND GROUTS

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ABSTRACT: In the absence of bolting standards for strata reinforcement in the Australian mines, though individual mines or companies have their in house practices, there remains a visible vacuum in assessing credibly the various properties of chemical resins and cementitious grouts. Currently, all methods used in evaluating the mechanical properties of both chemical resins and other bolting reinforcement grouts are dependent on the American, British and South African standards and accordingly there is no uniform and unified methodology of testing. A simplified approach has been discussed to enable mine operators and other interested parties to determine various pertinent properties of chemical resins and grouts in the bolting system only and this paper describes the various methods used to test a set of resin properties. A special Resin Mixing Container (RMC) was developed to permit multiple resin samples to be cast with consistent resin / grout quality. Various conclusions were drawn from the study enabling a better understanding between suppliers and end users.

INTRODUCTION

There is an increase in the variety of bolting systems used in Australian mines (rebar /cable tendons, chemical resins and cementitious grouts). Rock bolting systems constitute a major mining operation activity, particularly in underground coal mining and therefore a basic knowledge about the load transfer properties of the bolting systems must be clearly understood to enable both the suppliers of the products and end-users to maintain trust in their professional operations.

Australian mining and construction industry consumes around 7.5 m bolts (rebar and cables) annually and the installation of these supporting elements is carried out using chemical resins and cementitious grouts. In general, there are two types of resins in the market today; oil and water based resins and for each class of resin there are variations with respect to the mixing and setting times. Resins also vary in mechanical strength properties. Essentially chemical resins can be tailored to vary with respect to the setting time and curing. Some bolt installations are carried out using twin time setting, consisting of two setting time periods; the upper fast setting time of between 8-24 s, in the upper end of the bolt, and the slower setting time, of up to 90 seconds, with the latter being used in the lower section of the encapsulated bolts. Of course there are much slower resins used as pumpable products for long cable bolt installations.

The strength of the various resins can be related to the chemical composition and fillers and therefore it is useful to determine their various properties prior to use. The existing methods of evaluating various bolting system properties in Australia are still based on the established non-Australian standards, which is raising concern. Testing by double embodiments shearing of bolt in steel tube is unrealistic and therefore not applicable to shear testing of bolt in rock. Also evaluation and determination of various resin properties as specified by the American, British and South African standards guidelines may not be required for carrying out a set of tests that will be adequate for the user to gain an understanding of the strength characteristics of the resin type used. Therefore, the aim of this paper is to provide a simplified approach to determining various resin parameters, to allow both resin suppliers and end-users to make a start in the appreciation of the product availability and potential to ensure use of the correct type of resin for given ground conditions.

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RESIN PROPERTIES EVALUTION

Based on suggested methods by various standards (British standard- BS 7861: Part 1: (1996), American Standards (ASTM- C579) (1991), South African Standard (SANS1534) (2004), and ISRM (International Society of Rock Mechanics (2007)), the evaluation of the resin properties normally include the determination of:

- Uniaxial Compressive Strength (UCS),
- Modulus of Elasticity in compression (E),
- Shear strength, (τ) and
- Creep or Rheological properties.

Both Uniaxial Compressive Strength (UCS) and Young's modulus (E) values were examined at the University of Wollongong laboratory in relation to resin sample shape, size, height to width or diameter ratio (H/D), resin type, resin age and cure time. 40 mm cubes, rectangular prisms of L/D 2:1 and cylindrical specimens of diameters 20, 30, 40 and 50 mm were tested with L/D ratios of 1: 1 and 2: 1. The sample shear strength was determined using the Punch Shear Box testing method.

Uniaxial compressive strength

Traditionally in Australia resins are tested for the compressive strength using cube/prism or cylinders, H/D of 2:1. The BS 7861- part 1 Annex (M) and part 2 (Annex G) for testing resin grout uses prisms 12.5 mm x 12.5mm x 25 mm in size with respect to the fast and medium resin set time and 50 mm cubes for slow set time. On the other hand the ASTM C579 recommends testing all resins using 40 mm cubes. Opinions vary with respect to the shape and size of the tested resin sample as recommended by the British Standard of resin testing. Generally the manufacturers prefer the UCS values of the resin by testing 40 mm cubes, similar to the recommended methods for testing resin grouts according to ASTM-C579. It is a recognised fact that the strength values obtained by testing cube samples tend to be higher than the UCS values obtained by testing cylindrical samples. Also, the strength values tend to vary significantly, irrespective of the sample shape and size when samples are individually cast. The recent approach in sample preparation in bulk as reported by Aziz, *et al.,* (2013a) and Aziz, *et al*., (2013b) provided a new methodology of sample casting thus yielding consistent test results.

Modulus of elasticity

The determination of modulus of elasticity or Young's modulus of the resin as prescribed in BS 7861: part 1: (1996), recommends that a prism of H/D (aspect ratio) of 4 be subjected to a controlled compressive load. The axial and lateral strain to be monitored by four strain gauges mounted on the samples, or by using other means of monitoring the axial and later deformation of the tested sample, such as Linear Variable Differential Transducers (LVDTs), compression testing machines, optical devices or other suitable measuring devices. The tested sample is subjected to cyclic loading and the elastic modulus is the mean of the three-secant moduli measure between two levels of the applied load. This method of calculating the E value, through a recommended method may yield E values, which can be used for homogenous material with fractures, pores (anisotropic) and fissures such as concrete and rock or anisotropic rock. Additional benefit of sample instrumentation will include determination of the Poisson's ratio and modulus of shear, cohesion and angle of internal friction. Using the data from samples tested without instrumentation, the E value can be determined simply from the straight line extrapolation of the 20-60 kN or 40-80 kN range of the load-displacement profile as shown in Figure1. This is an average value of the compression test, which is ideal for materials such as steel with homogeneous and isotropic structure with no voids and irregularities that will cause the sample to squeeze and undergo slight displacement during the early stage of loading.

Shear strength

Table 1 lists various apparatus used for general testing of rocks and composite material in shear. The testing for shear falls into two categories, direct and indirect methods. All listed methods are applicable for testing resins, but the resin characteristics, time and effort restrict their selection for any particular resin type.

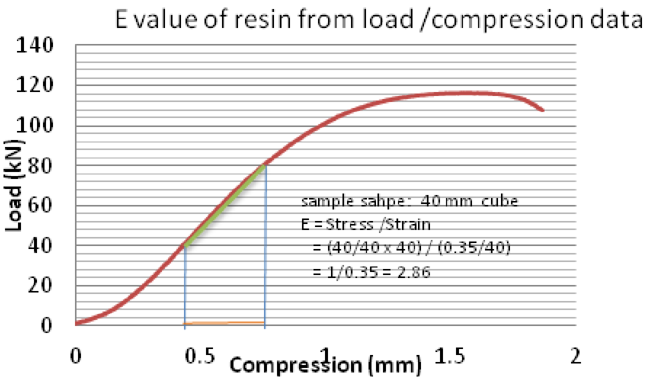
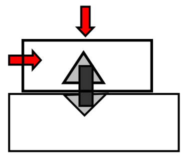
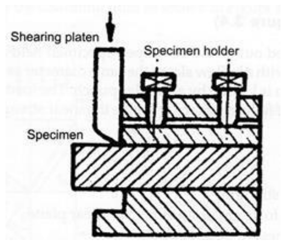
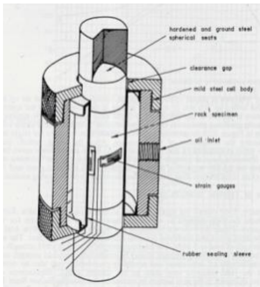
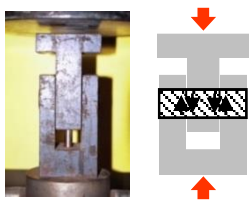
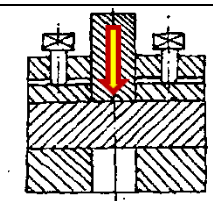


Figure 1 - Determination of E value from load–displacement (compression) testing

Table 1 - Laboratory methods of testing of shear strength of resin and grout

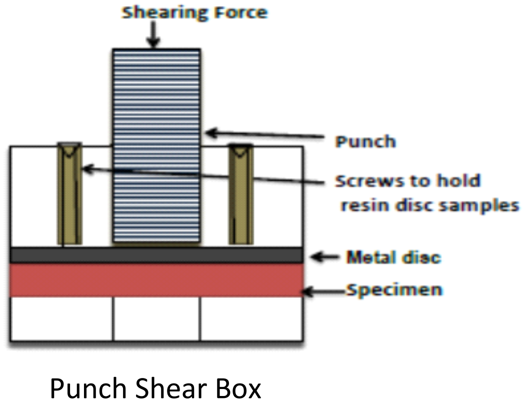
Method	Type	Procedure	Comments	Apparatus
Direct	Direct shear test	Resin sample in plaster or cement and shear the sample to failure peak and residual shear strength	Difficult to match resin strength with the cast medium and testing is a slow process.	
Direct	Single shear	The sample is clamped on the specimen holder and a shear force is applied perpendicular to the curved surface through a sharp edged platen. The shear strength is the force at failure divided by the area of cross-section of the failure surface	Not commonly used	
Indirect	Triaxial test	The specimen is enclosed in an airtight flexible membrane; confining pressure is applied and held constant during the test by means of a cell fluid. Apply axial load/hence stress until the sample fails. Test yields, UCS, Angle of Friction, Shear angle, failure angle	Good method of determining the shear strength of rock/resin; Requires expensive equipment, Difficult to do the test, slow, and time consuming	
Indirect	Double shear test	Lateral shearing of the sample with the samples ends supported. The specimen is sheared along two parallel planes. Shear strength = sheared failure load divided by twice the sample cross section area	Can be used for shear testing of 90 mm long and 30 mm diameter samples. Yields good results but require great quantity of resin samples cast	
Indirect	Punch shear	Shear strength carried out over a very short period of time	Easy to cast discs for testing. Several punch tests can be carried out from one large disk. Allows testing for shear strength over several weeks.	

Punch shear test method is most suited for testing resin. This method of shear strength determination is currently advocated by the South African Standards for testing of resins and grouts (SANS 1534:2004) and has been used by various resin manufacturers. Two methods are used for the preparation of the samples for the test, the 30 mm diameter disc cast in a steel ring and 65 mm discs cast in a polymer mould. Both sample types are 3 mm in thickness as shown in Figure 2. Only one test is possible from a 30 mm disc, while several tests (up to four) can be carried out on 65 mm diameter disc. The diameter of the punch rod is in the order of 12.5 mm, similar to the punch diameter specified in the SANS 1534 standard.

The test is carried out on a disc-shaped specimen at the bottom of a shear box fitted with a hollow slot of the same diameter as the punch. A disc shaped specimen is loaded by a circular punch. The shearing strength is determined using;

$$\tau = \frac{F}{\pi DT}$$

τ : Shear strength of the tested sample (MPa)
F: Failure load (kN)
T: Disc thickness (mm)
D: Punched disc diameter (mm)



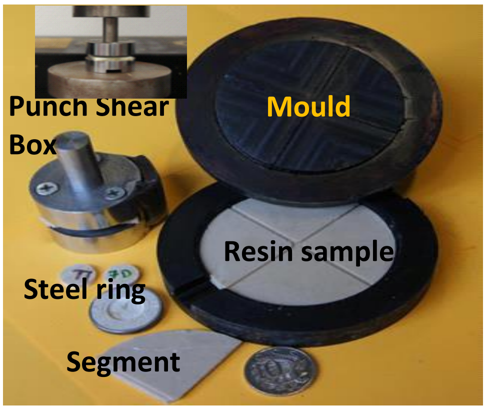


Figure 2 - Punch shear box and mould for casting 3 mm resin samples

Based on the experience, the punch shear test appears to be superior to other test methods because of:

1. Ability to prepare a number of samples in a very short period of time and produces a number of samples from a single resin mix with up to four sample segments being obtained from one large 65 mm cast disc. This ensures repetition of the test results for consistency.
2. Requires a small amount of resin preparation for testing, hence mixing time is not a problem.
3. Ability to test fast setting resin.
4. Consistent results for different period of times.
5. Ability to compare the resin shear strength between specifically prepared samples with results of the sections cut from the cylindrical or cube samples. This comparative study has been found to be a good indicator of the quality of the resin cast for various testings.

Rheological properties (Creep)

The recommended approach to determine resin creep properties is to use BS-7861 (1996). During testing the sample is loaded at a stress rate of 0.75 (N/mm^2)/s ±0.25 (N/mm^2)/s to a load of 5 kN for fast set resin or 20 kN for slow set resin and the load is maintained constant for a duration of 15 min. The resin strain is monitored between 0.5 and 15 min. After 15 min, the load is removed completely. The resin creep must not be more than 0.12 %, when the sample is tested 24 hours after casting.

EXPERIMENTAL STUDY

Sample preparation

Preparation of competent samples is an important aspect of testing resins and grouts for strength properties evaluation. The consistency of the testing results is dependent on the quality of the cast resin. Chemical resin setting time is the deciding factor in preparing competent and uniform samples. The methodology of sample casting is invariably carried out by preparing resin samples by manually mixing and casting of samples individually, particularly for fast setting resins. This method inevitably leads to less uniform or inconsistent resin grout composition and wider scatter of results. Additional drawback of manual mixing includes the difficulty of removing air bubbles from the sample, unless the sample is mechanically vibrated.

A new approach has been successfully developed for multi-sample casting of resins. The new system enables a relatively large quantity of resin to be mixed mechanically in a cylindrical container using a paint mixer. Powered by a hand held drill, the system can be used to prepare several resin samples from one mix. Both cylindrical and cube/prism samples can be cast. Once it is mixed, the resin is either poured directly into moulds as shown in Figure 3a, or the sample mould(s) are pushed into the MRC as shown in Figure 3b (Aziz, *et al.,* 2013a and 2013b). The set cast samples are then removed individually from the mould by gentle tapping. Alternatively, the whole resin block is first removed from the PVC container outer wall layer, then broken down to release individual sample moulds as shown in Figure 4.

Figure 3a - Mixed resin poured into moulds

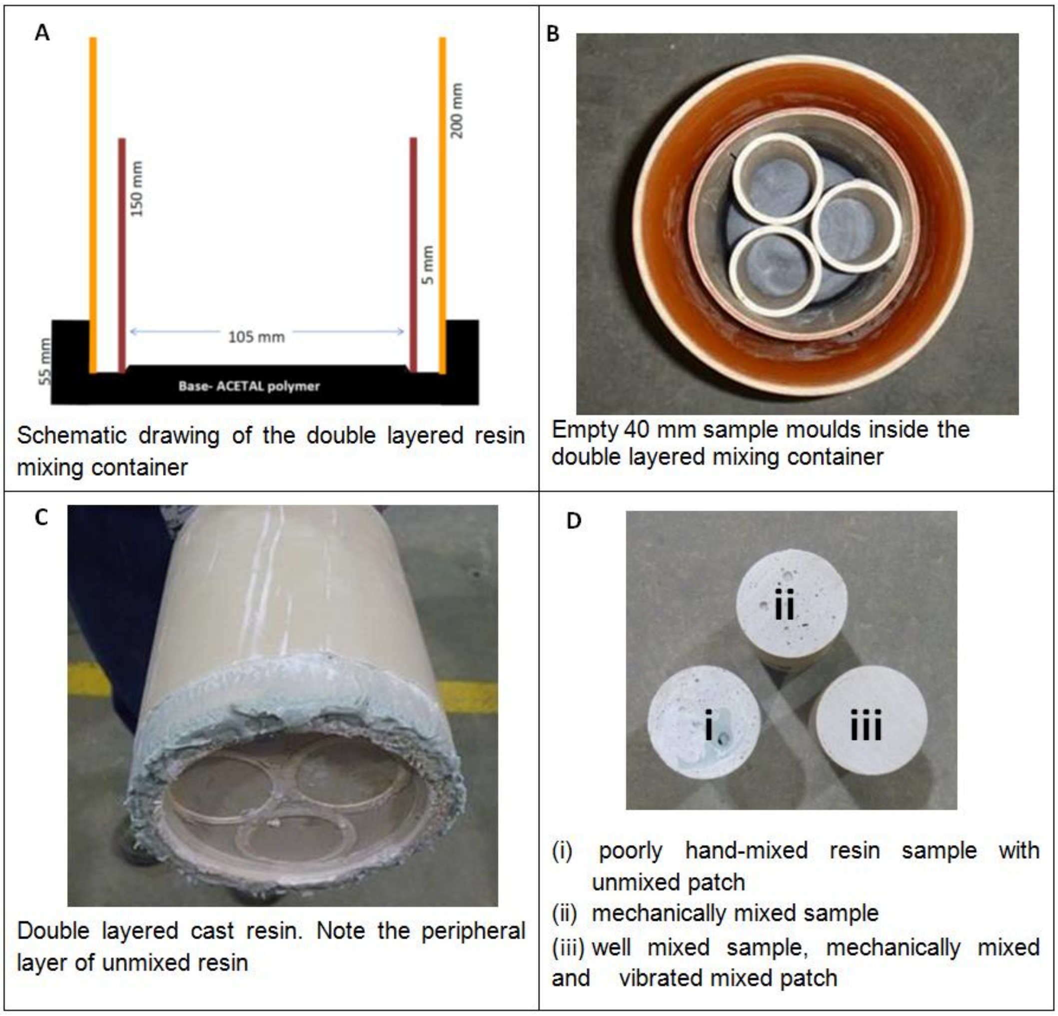
Figure 3b - Moulds pushed into MRC

Figure 4 - The process of separating cast samples moulds and removal of individual samples

Both the mixing container and samples mould are lubricated with inert grease prior to use for ease of releasing samples once set. It is worth noting that casting resin samples using cube moulds was possible only by pouring of the resin into sample mould. Irrespective of the sample shape, preparation of the resin samples as described would invariable lead to less uniform cast resin composition resulting in inconsistent and variable strength values. To overcome this deficiency in sample preparation a new mixing container was designed, consisting of a double layered concentric PVC cylinder mounted on a black ACETAL polymer base. The base had a circular groove at the outer periphery to accommodate the concentric walls as shown in Figure 5. The outer 200 mm long concentric PVC cylindrical layer is 140 mm in diameter, while the inner 110 mm diameter PVC cylinder is 150 mm long. There is a gap of 5 mm between the concentric walls of the RMC, where the layer of the poorly-mixed resin accumulates, thus the inner circular PVC separates the well mixed resin from the poorly mixed outer layer. The occurrence of badly mixed layer between the RMC walls is clearly evident in Figure 5D. A slit in the sides of both layers allows the set resin easier to remove with minimum chance of damaging the mould.

In casting samples the resin mastic and hardener are fully mixed in the empty RMC and the sample casting moulds arranged in the inner cylinder are then pushed down into the mixed resin in the RMC. A 5 mm thick plastic circular disc 100 mm in diameter, with 10 mm tapered holes, is pressed over the resin cast moulds to permit excess resin to be forced out of the mould for easy sample-ends preparation. Once the samples have set in the predetermined time, the set resin is removed from the main inner mould holder and the cast samples released from each individual moulds as shown in Figure 4. For casting cube samples the mould can also be used for resin mixing. The mixed resin is then poured into cubes / prisms as shown in Figure 6. Normally the cube size is 40 mm^3. Irrespective of the sample shape and size, the quality of the cast samples can be improved with proper vibration to remove trapped air bubbles and seal any remaining voids.

In compliance with the established standard requirements for sample end smoothness, the cylindrical samples have ends cut perpendicular to the sample axis and then subsequently lapped prior to testing.



Schematic drawing of the double layered resin mixing container

Empty 40 mm sample moulds inside the double layered mixing container

Double layered cast resin. Note the peripheral layer of unmixed resin

(i) poorly hand-mixed resin sample with unmixed patch
(ii) mechanically mixed sample
(iii) well mixed sample, mechanically mixed and vibrated mixed patch

Figure 5 - Double layered container for mixing chemical resin mechanically and variations in resin quality due to differing mixing techniques

Uniaxial compressive strength

A total of 100 chemical resin samples of various shapes, sizes, set time, cure time and age as described previously were tested for UCS values. Figure 7 shows the load displacement profiles of various shaped samples prepared from the same set time resin (90 sec gel time resin and catalyst removed from a capsule)). The samples tested were prepared from Orica resin mastic and hardener (catalyst) scraped from the mine supplied sheathed capsules, as part of the overall ACARP project investigation C21011). Figure 8 shows bar charts of the variation in average UCS values with changing sample shape and size of one day old cast samples. It is obvious the UCS values determined from various shaped samples differed with respect to the sample shape and size and load to height /diameter (H/D) ratios. Typically the UCS values were highest for 40 mm cubes and lowest for 40 mm diameter cylindrical samples with H/D ratio of two. The quality and repeatability of the tested samples are evident from Figure 9.

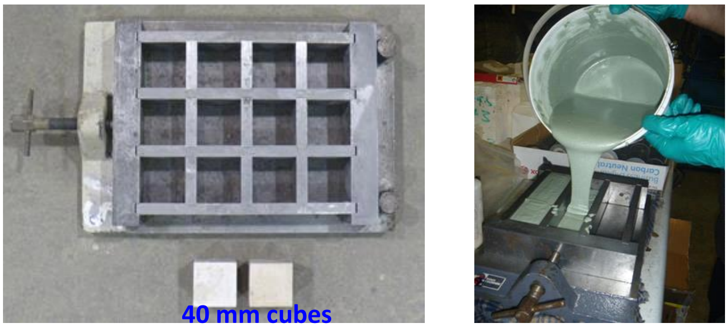


Figure 6 - Casting cube samples in cubical mould

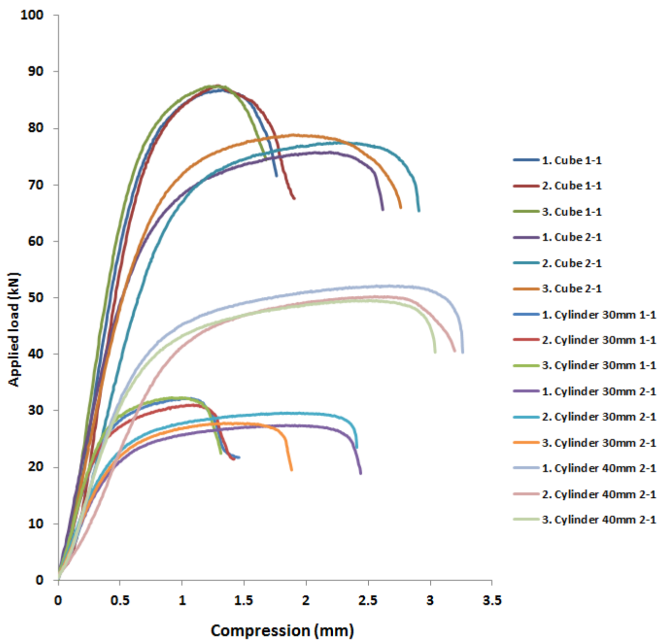


Figure 7 - Load /displacement profiles of various shaped samples prepared from the same set time resin

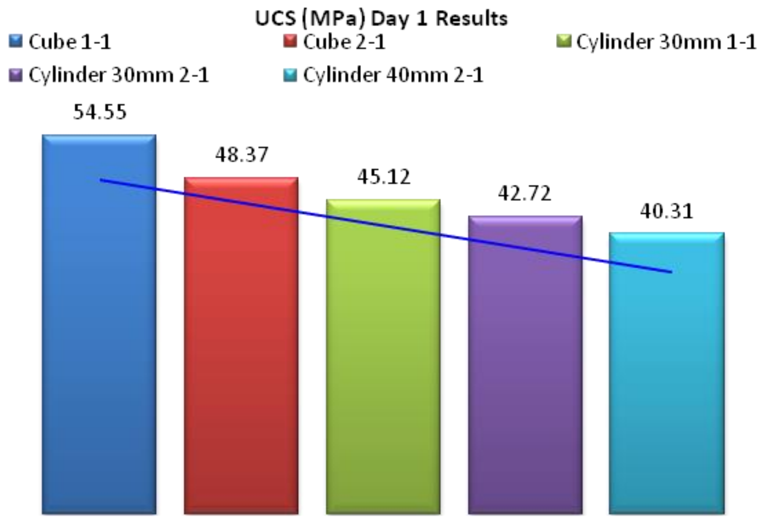


Figure 8 - Bar charts of the variation in UCS values with changing sample shape and size for one day old cast samples

The ratio between cube strength and cylinder strength varied and could be between 1.10 to 1.30. The high cube UCS value is attributed to friction between the platens of the compression machine and the specimen ends creating much more confinement (triaxial compression) than cylindrical specimens of the H/D ratio 2 and greater. The comparatively high values for cubes compared to cylinders are also the case with cementitious grouts (Minders, *et al.,* 2002). Figure 10 shows the changes in resin strength with cure time, which is expected. Figure 11 shows the variation in resin strength with respect to sample H/D ratio for cylindrical samples. As expected, the strength of the sample is influenced by the sample size and this is similar to rocks and cement grouts (Neville, 2069; Minders, *et al.,* 2002). The comparison between freshly and stored resin for various curing time is shown in Figure 12. It is observed that a higher uniaxial compressive strength is attained by using fresh resin in comparison to stored resin. It is evidently clear that the strength values of the resin used in bolt encapsulation is influenced by the above mentioned factors and in particular the shape and size of the samples used. Universality of the samples shape and size is thus an issue which requires addressing.

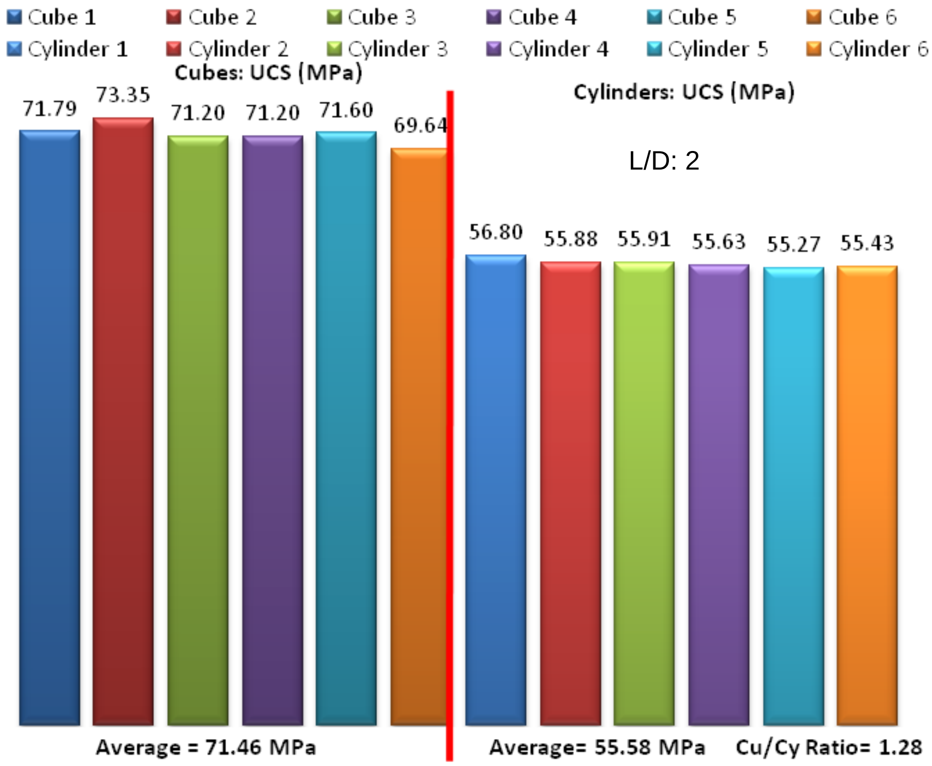


Figure 9 - Variations in UCS values between cube and cylinder resin samples (Orica slow setting resin (90 secs setting time). Note the consistency of the test results

Modulus of elasticity

Three methods, namely 40 kN range, tangent and secant modulus were used to make a comparative study. The use of 40 mm cube samples simplifies the determination of E value as the value of E for the 40 kN load range will be equivalent to the sample compression. However the calculated value from this approach is markedly outside the values obtained from other more credited methods.

Figure 13 shows the comparison between the E-values obtained through different ways for resin samples with various curing time ranging from 7 to 21 days. The E-values determined by the 40 kN range (manufacturers recommended) are generally higher than those obtained from ISRM recommended methods such as tangent and secant modulus for various curing intervals. Also, the E-values increased as the resin sample curing time increased from 7 to 21 days.

Figure 14 compares the E-values determined from the strained gauged samples and specimens without strain gauges. It is observed that the data extracted from strained gauged samples provide higher Elastic modulus when compared to samples without strain gauges. It should be noted that the E values obtained using strain gauges are restricted to the middle section of the tested sample and not the entire length of the sample under compression, hence the variation in E values reflects on the condition of testing and is in line with various test standards indicated previously.

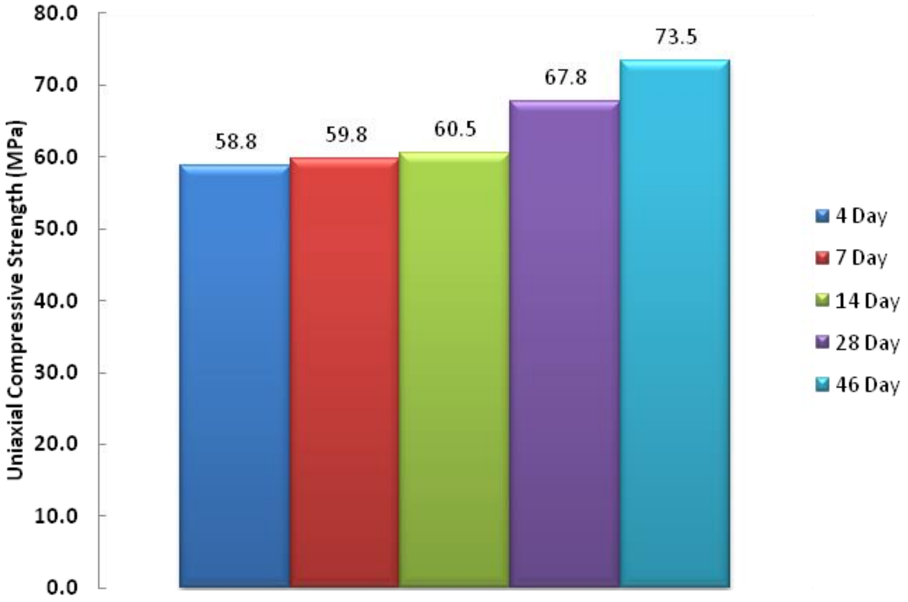


Figure 10 - Variation in resin strength with sample cure time for 30 mm diameter 2:1 ratio cylinder samples

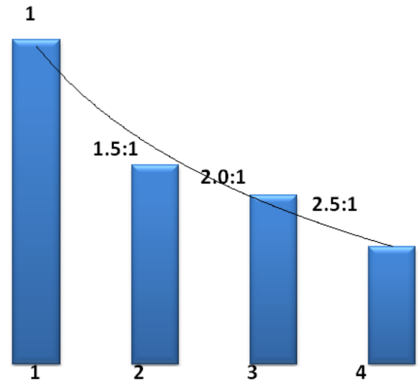


Figure 11 - Variation in UCS values with respect to sample height / diameter (H/D) for slow setting resin cylindrical samples

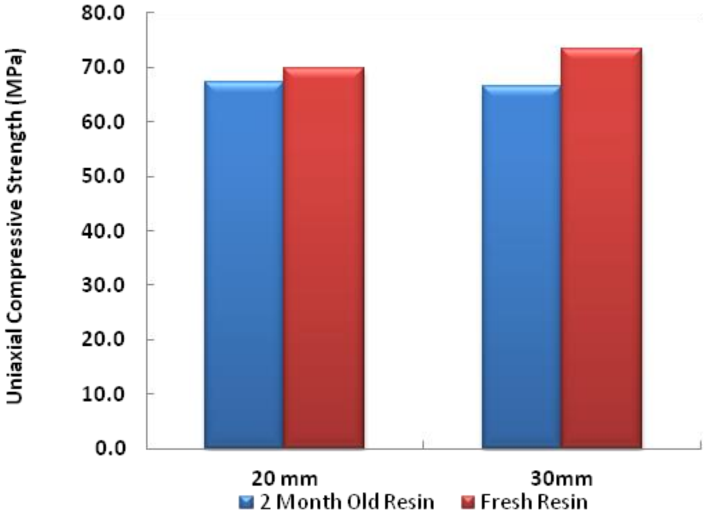


Figure 12 - Variation in resin UCS values between new supplied and stored (old) resins, for both 20 mm and 30 mm diameter samples, L/D=2

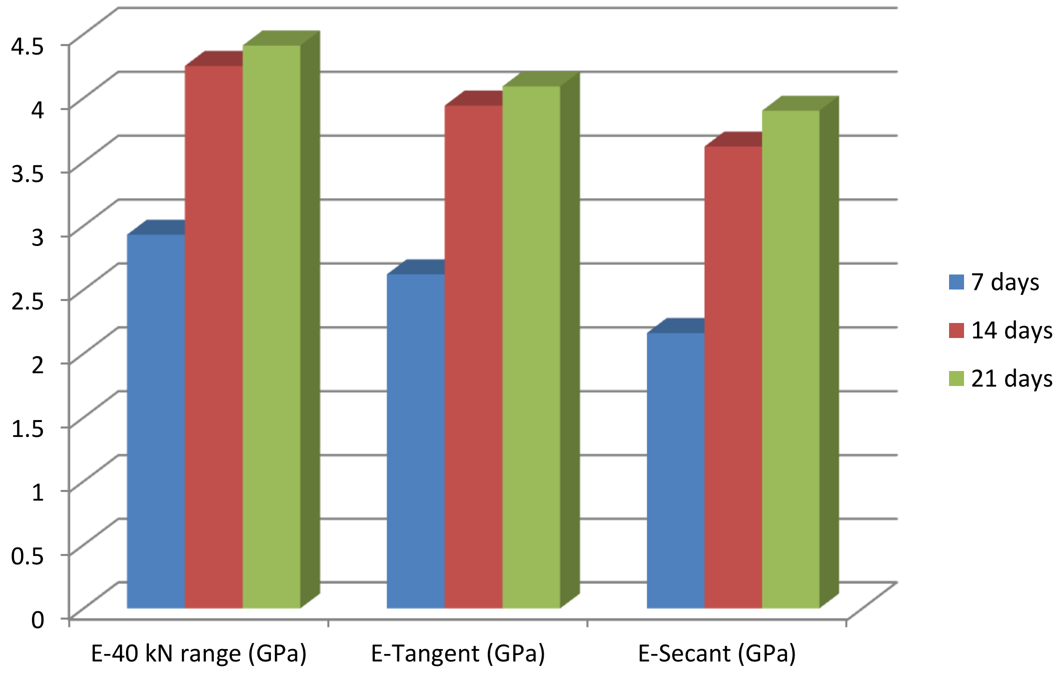


Figure 13 - Comparison between the E-values obtained through different ways for resin samples with various curing time ranging from 7 to 21 days

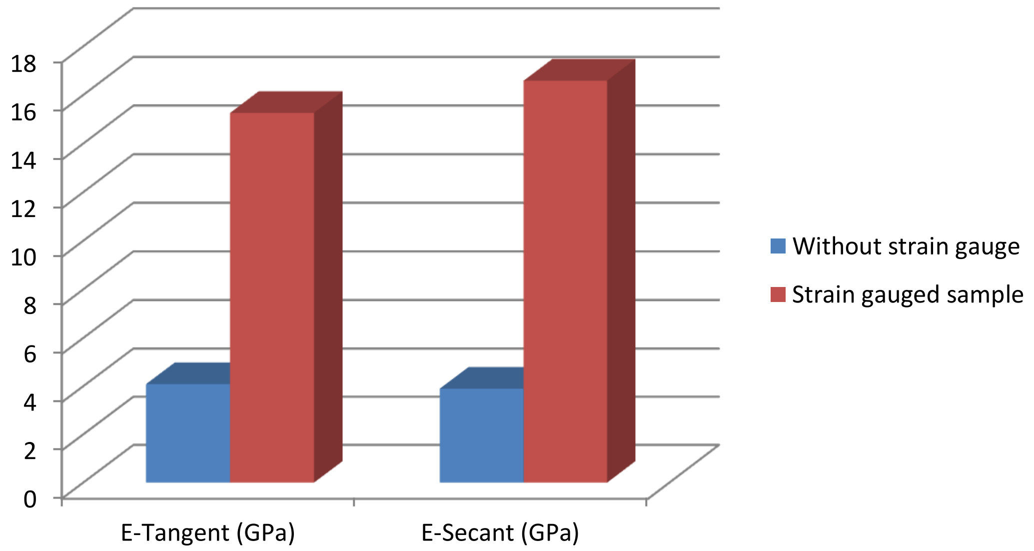


Figure 14 - Comparison between the E-values determined from the strain gauged samples and specimens without strain gauges

The comparison between the E- values of cubic and cylindrical samples for different curing time is shown in Figure 15. It is concluded that the cubic samples exhibit higher elastic modulus values in comparison to cylindrical specimens various curing time. However, this aspect involves further study.

Punch shear test results

Using the punch shear box shown in Figure 2 a series of punch shear tests were undertaken to study the shear strength of a particular resin. Each 65 mm diameter, 3 mm thick disc was cast using the new resin casting mould shown in Figure 2. Four shear tests were obtained from each disc cast. Table 2 shows typical results of punch tests carried out on several segments of one disc sample of the Orica fast setting resin, which is scraped from the resincapsulessupplied to a designated mine. A number of tests from a single or several large samples prepared using the newly designed casting moulds demonstrated the ease with which several tests can be carried out over a short time and with consistency of the results. Figure 16 shows the bar chart of variations in the average values, indicating the increase in average shear values with sample cure time, similar to UCS values. Figure 17 shows the variation of shear strength values between Mix and Pour and scraped slow setting resins respectively.

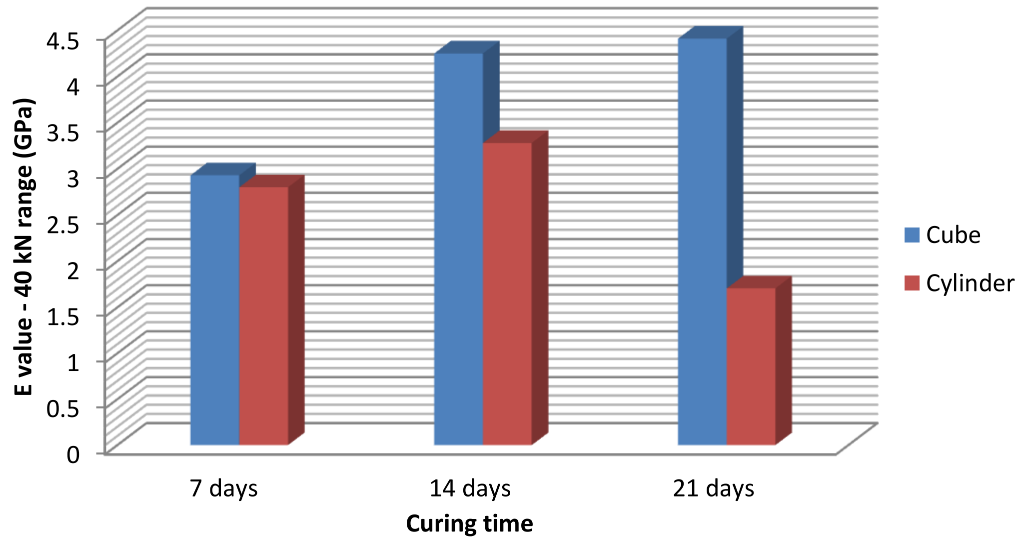


Figure 15 - Comparison between the E- values of cubic and cylindrical samples for different curing time

Table 2 - Shear strength values of resin samples tested using punch shear test. The test results are with respect to the samples cure time of 1, 7 and 14 days

	MN	T (m)	D (m)	π	τ (MPa)
1 Day Samples					
A	0.003493	0.00325	0.01258	3.142	27.1912
B	0.003455	0.00351	0.01266	3.142	24.74577
C	0.003408	0.0036	0.01264	3.142	23.83657
D	0.003706	0.00339	0.01256	3.142	27.70192
E	0.003192	0.00337	0.01261	3.142	23.90626
F	0.003493	0.00338	0.01256	3.142	26.18702
				Average	25.59479

	MN	T (m)	D (m)	π	τ (MPa)
7 Day Samples					
A	0.003983	0.00328	0.01255	3.142	30.79545
B	0.004337	0.00343	0.0126	3.142	31.9388
C	0.003447	0.0034	0.0126	3.142	25.60859
D	0.004387	0.00339	0.01264	3.142	32.58477
F	0.003821	0.00366	0.01262	3.142	26.32876
				Average	27.66446

	MN	T (m)	D (m)	π	τ (MPa)
14 Day Samples					
A	0.004148	0.0033	0.01261	3.142	31.72514
B	0.004652	0.0034	0.01264	3.142	34.45145
C	0.005442	0.00409	0.01256	3.142	33.71626
D	0.004138	0.00338	0.01263	3.142	30.85065
E	0.00344	0.0032	0.01257	3.142	27.21868
F	0.004091	0.00388	0.01254	3.142	26.76049
				Average	30.78711

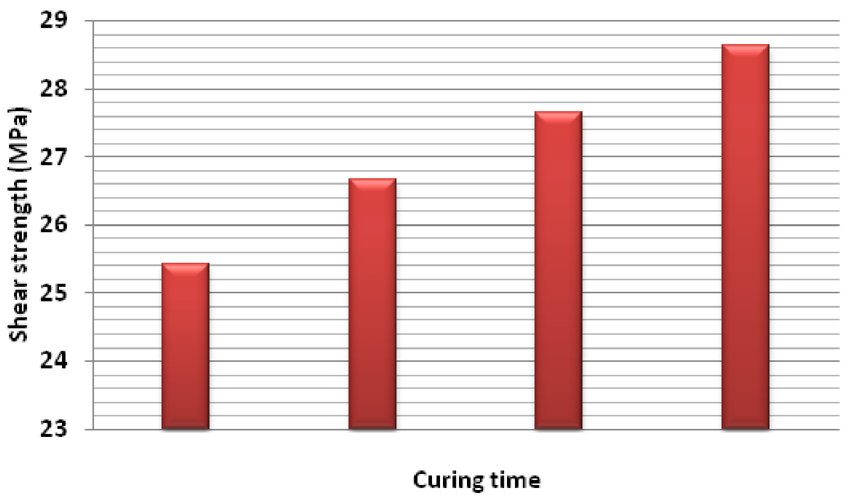


Figure 16 - Average shear strength values for various cast samples cure time

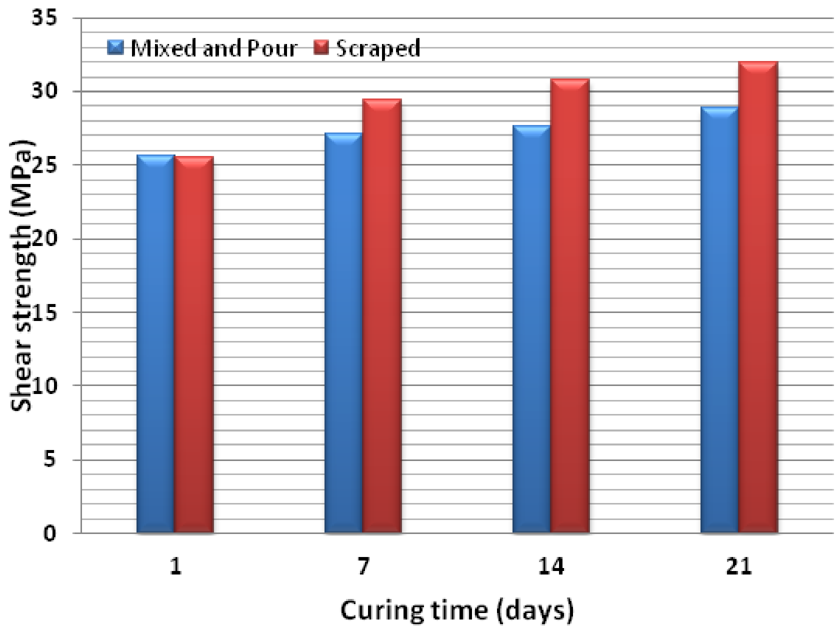


Figure 17 - Variation in resin shear strength values between new supplied and scraped resins for various curing periods

Creep tests

No creep tests have been carried out at this stage and will be the subject of study as part of the resin mechanical strength properties study programme for an ACARP report.

DISCUSSION

Cube tests are used as the normal compression test of resin and cementitious grouts in Great Britain, in Germany and in some other parts of Europe. The British standard (BS 7861-Part 1:1996) now favours 50 mm cubes for slow setting resins and much smaller sizes for fast setting resins. Similarly ASTM-C759 uses cubes and only South African standard (SANS1534) uses cylinders of H/D ratio of 2:1. In Australia the two major resin suppliers used different shapes and it was not until recently that the use of 40 mm cube has prevailed. With current methodology of resin mixing that is being described in this paper and previously by Aziz, *et al.,* (2013a), the consistency of test results for the determination of the UCS and E values and other properties can be achievable for both cube and cylindrical samples.

Normally the cube is tested at right angles to the position at which are cast; this means that the faces of the cube in contact with the bearing platens are cast against the sides of a rigid steel mould, which is an advantage.

Also the relation between the directions as cast and as tested has, however, no influence on the results since resin grout does not segregate when vibrated. Similarly, the direction away from as cast has shown not to affect appreciably the strength of cubes made with unsegregated and homogenous concrete and grouts as reported by Neville (1969). It should be remembered that the stress distribution in any compression test is such that the test is only a comparative one and the strength values obtained is dependent of the shape and size of the tested sample.The main drawback of the cube test is that the friction between the platens of the compression machine and the specimen ends creates much more confinement (triaxial compression) than cylindrical specimen of the H/D ratio of two and greater. This leads to higher strength values when measured on cubes rather than cylinders as demonstrated with the test results shown in Figures 7, 8 and 9 respectively. The ratio between cube strength and cylinder strength as tested ranges between 1.1 to 1.3. The decision on deciding whether to use cubes or cylinders for evaluating strength properties of a chemical resin relies on the following:

- How easily and consistently resin samples can be prepared and tested with a minimum of effort and extra preparation after casting, identification of the factors that contribute to the simplicity of the sample preparation and testing,
- Recognising that the UCS values obtained from testing samples will yield a relative strength and not true strength,
- Cylindrical sample ends invariably require machining to ensure the ends are smooth and perpendicular to the sample axis. Hence, labour and additional equipment for sample end preparation are required.
- The current practice adopted by the resin manufacturers, which in general use cubes is that the adoption of cubes is internationally recognised by various mining companies.

Given that the current methodology of preparation of resin mixes in bulk as described in this paper, which allow multiple samples to be readily cast irrespective of the sample shape, it is thus propitious and advantageous to use cubes, as cube samples can be easily prepared and tested without additional effort. It is thus become easier for the mine operators and other professionals that may not have access to additional sample end preparation facilities to readily conduct strength property tests. This will enable the product suppliers and end users to maintain trust and avoid unnecessary delays.

CONCLUSIONS

The study of the suggested methods of composite material preparation and testing for strength has led to the following conclusions:

- The methodology of resin mixing and sample casting is an important aspect of evaluating the strength properties of resin. The new double wall container for mixing resin provides a practical way of thorough mixing of mastic and catalyst enabling casting of samples with minimum of unmixed patches and air bubbles.
- Cubes are the ideal and practical way of sample casting and testing as they require a minimum of time and effort of sample ends preparation after casting.
- The determination of the modulus of elasticity can best be determined by using 40 mm x 40 mm x 80 mm prisms (H/D :2) instrumented with strain gauges. This approach will also allow the determination of Poisson's ratio, cohesion, angle of internal friction and others to be determined for analytical modelling studies for effective strata load transfer mechanism studies.
- The punch shear box test is an easy, economical and fast method of determining the shear strength of the resin. The use of 3 mm thick, large size (65 mm in diameter) sample discs enables multiple samples to be tested for repeatability and for test quality assurance. The use of vibrator during resin casting helps to produce homogeneous composition resin mix.
- More studies are currently undertaken been to examine the creep properties of the cast resin.

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