A New Method for Correlating Rock Strength to Indentation Tests

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Abstract

In this Paper, UCS of limestone rock was determined by indentation testing on rock fragments. The size of the fragments was in the range of 2 to 5 mm, which is within the size range of drill cuttings. Here, scatter of the data was investigated by calculating the coefficient of variation that showed the size dependency of the conventional indentation parameters; Indentation Modulus (IM) and Critical Transition Force (CTF). Thus, it is recommended that the results of indentation tests should be normalized by the fragment size and presented in the form of Normalized Indentation Modulus (IM_n) and Normalized Critical Transition Force (CTF_n). Regression analysis was carried out to show the relationship between the IM_n or CTF_n and the UCS. Linear and exponential regression provided reasonable correlation coefficients of higher than 0.74 and 0.85, respectively. The proposed empirical equations for estimation of the intact rock UCS from the normalized indentation indices were verified using independent data from limestone rocks not used in developing the correlations. The outcome was a strong agreement (R~1.00) between the estimated and measured UCS. Based on statistical analysis, minimum number of indentation test was determined for random size samples. It is suggested that indentation testing be carried out using uniform fragment sizes to reduce the required number of indentation tests.

Key words: *Drill Cuttings, Indentation Test, Normalized Critical Transition Force, Normalized Indentation Modulus, Rock Fragments, Mechanical Testing, Uniaxial Compressive Strength, Wellbore Geomechanics.*

1. Introduction

Mechanical properties of rocks, especially the UCS and E, have significant impact on the design of rock engineering projects (Santarelli et al., 1996; Uboldi et al., 1999; Holt et al.,

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2005; Mateus et al., 2007 and Garcia et al., 2008). Investigations carried out by Jaramillo (2004) show that UCS of formations has the highest influence on wellbore stability compared to other factors such as azimuth, slope, exposure time and even mud weight.

Conventionally, two well-known techniques are used to obtain rock mechanical properties: a) standard laboratory tests on rock cores, e.g. ASTM 1984 or ISRM 1979, and b) acoustic well log interpretation combined with lithological analysis of formation (Fjar, 1999 and Mateus et al., 2007).

Although these methods are reliable for obtaining rock strength, practical limitations such as the costs involved, unavailability of rock cores and well logs in some intervals and the time required for delivery of results make them restricted (Zausa and Santarelli 1995; Santarelli et al., 1996; Holt et al., 2000; Chang, 2004 and Chang et al., 2006). Such limitations have led to the development of nonconventional techniques and procedures to achieve mechanical properties of formations encountered during drilling. Some of these techniques are listed below:

- Empirical correlations between rock mechanical properties and easily obtainable and reliably measured parameters such as porosity and density (Plumb, 1994 and Horsrud, 2001),
- Measurement of P and S wave velocities by Pulsed Ultrasonic on Cuttings (PUC) (Santarelli et al., 1996) and Continuous Wave Technique (CWT) (Nes et al., 1998),
- Laboratory test on reconstructed core from rock cuttings (Mehrabi Mazidi et al., 2012),
- Correlation between mineralogical or textural characteristics and mechanical properties (Tugrul and Zarif, 1999; Kekec et al., 2006 and Zorlu et al., 2008), and

- Indentation test on drill cuttings as a laboratory technique (Thiercelin and Cook, 1988; Cook and Thiercelin, 1989; Thiercelin, 1989; Suarez-Rivera et al., 1990; Suarez-Rivera et al., 1991 and Santarelli et al., 1996).

The laboratory indentation test uses small rock fragments in the range of 2 to 5 mm. In this technique, the resistance of rock fragments against the penetration of an indenter is measured and these measurements are used in empirical correlations to determine the rock UCS.

This paper presents a new method for interpretation of indentation test results that leads to a new correlation between the normalized parameters of Indentation Modulus (IM_n) and Critical Transition Force (CTF_n) with UCS. Statistical approaches are used to examine the size dependency of the indentation parameters and to determine the minimum numbers of tests required for a statistically reliable analysis. The test results showed the size dependency of the conventional indentation parameters and the higher number of tests required when using random size rock fragments compared to uniform size fragments. To minimize the size dependency and required number of tests, we recommend using normalized indentation indices for determining the UCS and doing indentation test on fragments of approximately the same size.

2. Methodology

2.1 Sample preparation

A total of 9 limestone boulders were collected from various types of limestone from outcropped rocks of Iranian oil fields. Boulders S1 through S6 (UCS from 280 MPa for S1 to 30 MPa for S6) were used to develop the correlations and boulders S7, S8 and S9 (UCS between 80 and 250 MPa) were used to verify the accuracy of the correlations.

A number of core plugs were drilled out of the samples for conventional UCS testing (see Table 1). The rest of the boulders were used for producing artificial rock cuttings in a crusher machine that produced rock fragments similar in shape and size to drill cuttings. Particles with sizes between 2 and 5 mm were selected for the indentation testing. Small rock fragments were embedded in epoxy-resin, composed of resin epoxy bisphenol and polyamine hardener, to provide the confinement needed during the testing. The samples were disk shaped with 55 mm diameter and 10 mm thickness (Fig. 1a). After curing the resin disk, the disk was trimmed and polished to expose sufficient number of rock fragments, e.g., 30-40 pieces hereafter named a specimen group, for indentation testing (Fig. 1a).

Table 1: Physical and mechanical characteristics of the samples.

Sample No.	Lithology	Rock Density (g/cm^3)	Porosity (%)	UCS (MPa)
S1	Micritic Limestone	2.59	2.25	279.76
S2	Micritic Limestone	2.64	2.62	230.39
S3	Micritic Limestone	2.73	< 0.1	182.49
S4	Siliceous limestone	2.65	0.91	129.73
S5	Siliceous limestone	2.64	0.93	124.39
S6	Marly Limestone	2.43	9.64	31.50
S7	Micritic Limestone	2.72	1.16	246.32
S8	Micritic Limestone	2.63	6.06	178.45
S9	Siliceous limestone	2.64	1.07	82.89

Note: Samples S1 to S6 were utilized to evaluate the indentation test results and samples S7 to S9 were used to verify the accuracy of the proposed equations.

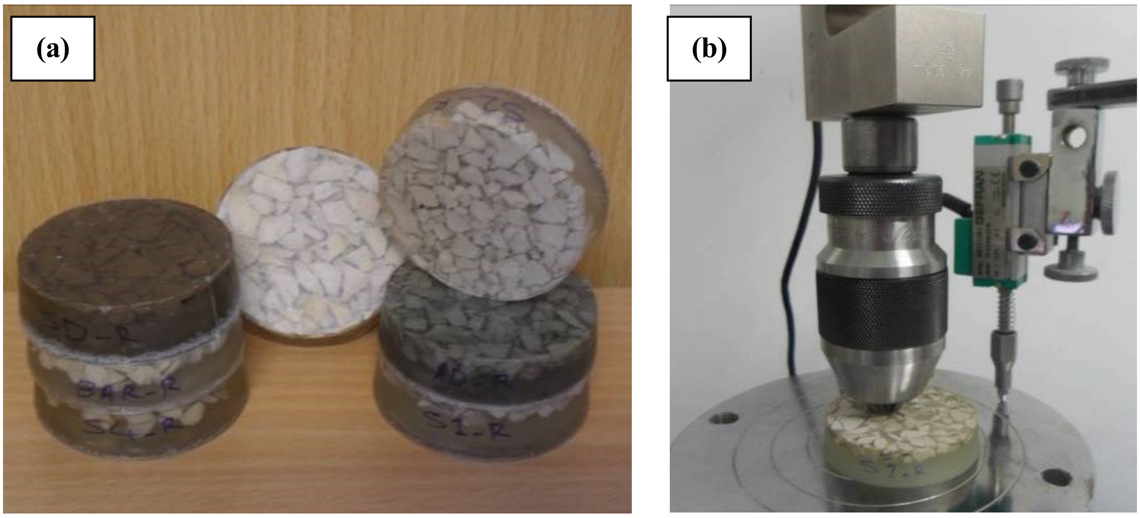

Fig. 1: (a) Rock fragments embedded in epoxy-resin and shaped in disk form with a polished face to expose the rock fragments on surface, and (b) indentation machine and the cylindrical flat-end indenter used for this study.

2.2 Testing method and analysis

Indentation test measures the force and displacement of an indenter while penetrating into a rock fragment until it breaks apart. A cylindrical flat-end indenter stylus with the diameter of 1 mm was used in this work. It was made of tungsten carbide with ASTM hardness of 91 Rockwell B. The applied force and penetration rate were measured during the testing by a load cell and a displacement sensor, respectively (Fig. 1b). A constant penetration rate of 0.01 mm/sec was applied on rock fragment until failure occurred (similar to the rate applied by Mateus et al., 2007; Garcia et al., 2008).

The results are shown in the form of load versus penetration, similar to the conventional stress-strain curves. A typical curve, illustrated by Garcia et al. (2008), is presented in Fig. 2. Conventionally, two main indices are derived from the indentation curve: Indentation Modulus (IM) and Critical Transition Force (CTF). Indentation modulus (analogous to elastic modulus) is obtained from the slope of the linear part of the curve and is defined as the resistance of rock against penetration of the indenter. The critical transition force is the load level beyond which softening behavior is observed.

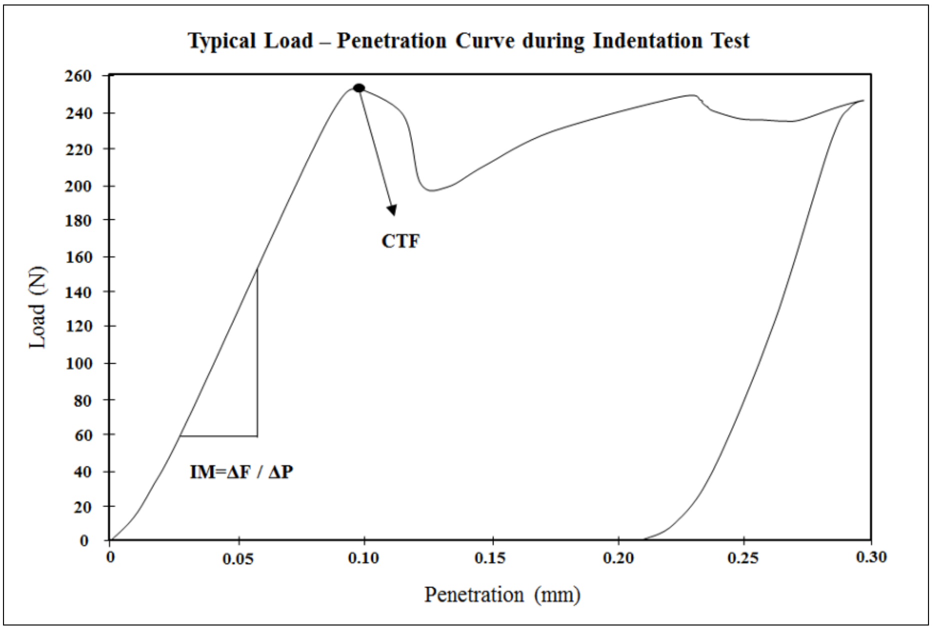

Fig. 2: Typical load-penetration curve from indentation testing with IM and CTF identified on the curve. After Garcia et al. (2008).

In Section 3.1, the results of the conventional indentation indices are presented which indicate size dependency of the results. The indentation indices were normalized to decrease the size dependency of the indices when testing samples containing fragments of different sizes.

An arithmetical average value for normalized indentation indices (IM_n and CTF_n) was calculated for each specimen group, which consisted of a total of N fragments (30-40 fragments in our tests) as follows:

$$IM_n = \frac{\sum_{i=1}^{N} \frac{IM_i}{OC_i/IC_i}}{N} \quad (1)$$

$$CTF_n = \frac{\sum_{i=1}^{N} \frac{CTF_i}{OC_i/IC_i}}{N} \quad (2)$$

Where IM_i and CTF_i are the conventional indentation indices for fragment i, OC_i is the diameter of the smallest circle circumscribed about Fragment i and IC_i is the diameter of the circle inscribed in the same fragment. Finally, normalized indentation indices of the samples were correlated with the uniaxial compressive strength from core testing of the same rock type.

2.3 Statistical concepts

A statistical analysis was performed to determine the minimum number of rock fragments required for a reliable assessment of the indentation indices and to allow the size dependency analysis.

The number of data points required for determining a mean representative value in rock mechanical problems is a function of sample heterogeneity, sample sizes and scatter of the results. Gill et al. (2005) utilized the small sampling theory and presented a statistical procedure to obtain the minimum number of specimens as observation points for determination of the representative values of the rock mechanical characteristics. Based on the small sampling theory, the observed coefficient of variation after testing (CV_{ob}), the reasonable precision index and the confidence interval have major effect on the minimum number of specimens. The CV_{ob} is calculated as the ratio of sample standard deviation relative to the arithmetic mean value (S) to the average value of the arithmetic sample mean $\left(\overline{X}\right)$, expressed in percentage:

$$CV_{ob} = \frac{S}{\overline{X}} \times 100 \qquad (3)$$

An advantage of the coefficient of variation is its independency on the units of measurement. The other important factor for determining the minimum number of samples is the precision index (p) which is defined as the ratio of the upper to lower bound of the population mean as estimated for a specimen group:

$$p = \frac{\overline{X} + t_\beta \frac{S}{\sqrt{N-1}}}{\overline{X} - t_\beta \frac{S}{\sqrt{N-1}}}, \quad p \geq 1 \qquad (4)$$

where S is standard deviation, $\overline{X}$ is arithmetic average value and t_β represents the confidence coefficient obtained from the Student *t* distribution which is a function of the number of degrees of freedom (N-1) that in turn is a function of sample size N in the final calculation. Combination of Eqs. (3) and (4) gives the required number of tests (N) for a statistically significant mean value which will guarantee a given precision index (p) for a specified confidence interval:

$$N = \left[\left(\frac{p+1}{p-1}\right) t_\beta \frac{CV_{ob}}{100}\right]^2 + 1 \qquad (5)$$

The numbers of required specimens for determination of the representative value as a function of precision indices ($1.20 \leq p \leq 1.60$) and coefficients of variation for a 95% confidence interval are presented in Table 2.

Table 2: The number of required samples based on the precision index and coefficient of variation with 95% confidence interval (Gill et al., 2005).

CV_{ob} (%)	$p \leq$					
	1.6	**1.5**	**1.4**	**1.35**	**1.3**	**1.2**
35	13	16	21	25	31	61
30	10	12	16	19	24	46
25	8	10	12	15	18	33
20	6	7	9	11	13	22
15	5	6	7	8	9	14
10	4	4	5	5	6	8
5	3	3	3	4	4	5

Gill et al. (2005) suggested different p values depending on the investigation/project importance; $p \leq 1.35$ for long-life mining structures and civil engineering works and $p \leq 1.20$ for important underground excavation and research work. Protodyakonov (1960 and 1969) and Vutukuri et al. (1974) recommended $p \leq 1.50$ as the best value for determining the number of required samples for laboratory investigations. Generally, Spiegel (1961) suggested 30 observation points for finding a reliable mean value for laboratory investigations.

For the indentation test, different researchers suggested different number of indentation tests in order to compute an average value; Santarelli et al. (1996) recommended a series of five indentation tests, Mateus et al. (2007) suggested an average value of 10-15 for sandstone samples and Garcia et al. (2008) showed that a minimum of 25 indentation tests were necessary for shale samples.

3. Results and Discussion

3.1 Test results and statistical analysis

A total of about 220 indentation tests were carried out on 6 specimen groups of limestones, each containing around 35 rock fragments. Macroscopic and microscopic residual imprints of the indenter and post failure fractures are presented in Fig. 3. The indentation modulus (IM) and critical transition force (CTF) were extracted from the load-penetration curves of the indentation tests. The average conventional indentation indices were calculated from the results of all indentation tests for each sample and the corresponding coefficient of variations (CV_{ob}) were calculated to determine the dispersion of the indentation parameter in each specimen group (Table 3). The results showed that:

- IM varied from 2425 N/mm for Sample S6 to 4433 N/mm for Sample S2,
- CTF was between 290 N and 990 N for Samples S6 and S3, respectively,
- CV_{ob} of IM and CTF were in the range of 17.61% - 28.11% and 28.62% - 49.97%, respectively.
- CV_{ob} of the IM was less than that of the CTF (see Fig. 4).

The CV_{ob} represents the dispersion of the test results for the specimen groups. The high value of CV_{ob} indicates a wide range of variation of the indentation parameter for different size fragments. Higher values of CV_{ob} for the CTF compared to the IM indicate stronger sensitivity of this parameter to the rock fragment size.

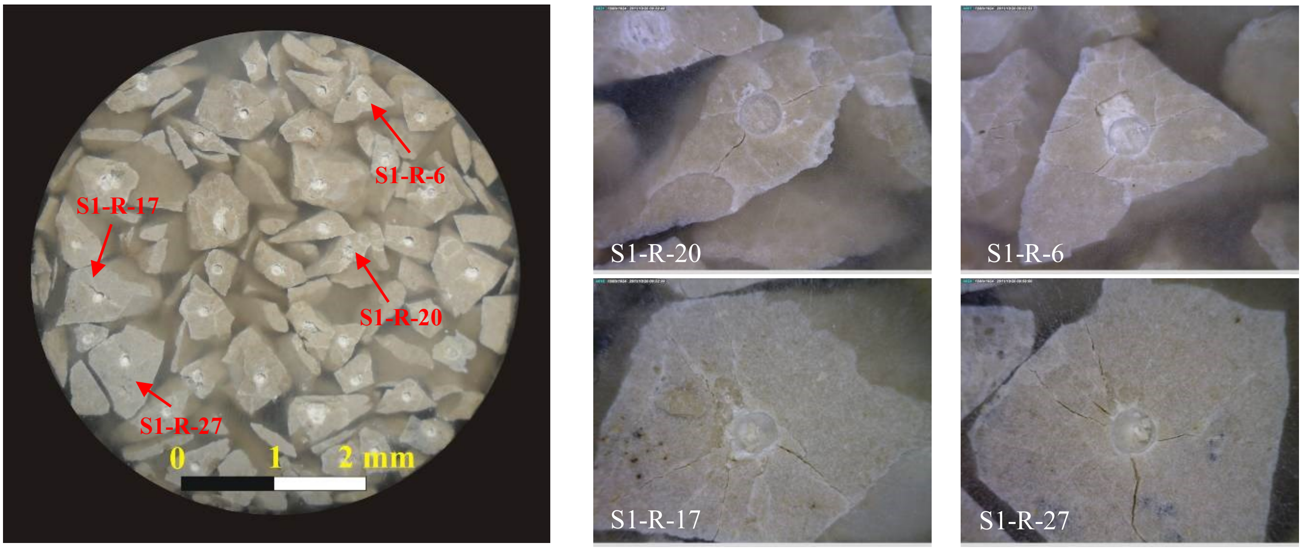

Fig. 3: Macroscopic residual imprint after indentation test on Sample S1 and close-up of four tested fragments.

Table 3: Results of the conventional indentation tests on rock fragments.

Sample No.	Number of Tested Samples	Indentation Modulus		Critical Transition Force	
		IM (N/mm)	CV_{ob} (%)	CTF (N)	CV_{ob} (%)
S1	39	4265.02	26.88	825.27	48.23
S2	37	4433.25	17.61	869.53	41.47
S3	40	4102.52	20.70	990.03	33.94
S4	35	3869.82	26.21	673.30	49.97
S5	39	4034.61	18.09	887.77	42.38
S6	30	2425.65	28.11	290.57	28.62

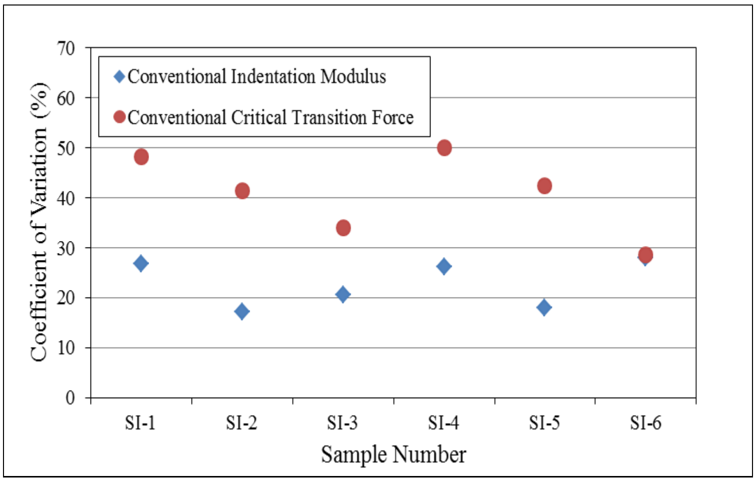

Fig. 4: Distribution of the observed coefficient of variation (CV_{ob}) for IM and CTF for different samples (red circles show the conventional critical transition force and the blue diamonds illustrate the conventional indentation modulus).

To illustrate the effect of specimen size, a total of 40 load-penetration curves of Sample S1 are presented in Fig. 5a (Fragments S1-R-1 through S1-R-40). The figure shows curves with various slopes and thus different indentation indices. The dependency of IM on the sample size is also evident from the curves in Fig. 5b for the four different rock fragments of Sample S1. The size of each rock fragment (area and ratio of OC/IC) and related indentation indices are also presented in this figure. As shown in Fig. 6, the greater the sample size, the higher are the indentation indices.

The sizes of all fragments in each specimen group were measured and plotted against the corresponding IM and CTF, which confirmed the size dependency of the indentation indices (Fig. 6). Linear regressions were developed resulting in correlation coefficients of 0.72 and 0.64 for CTF and IM, respectively. Higher correlation coefficient of the CTF indicates its higher size sensitivity compared to the IM.

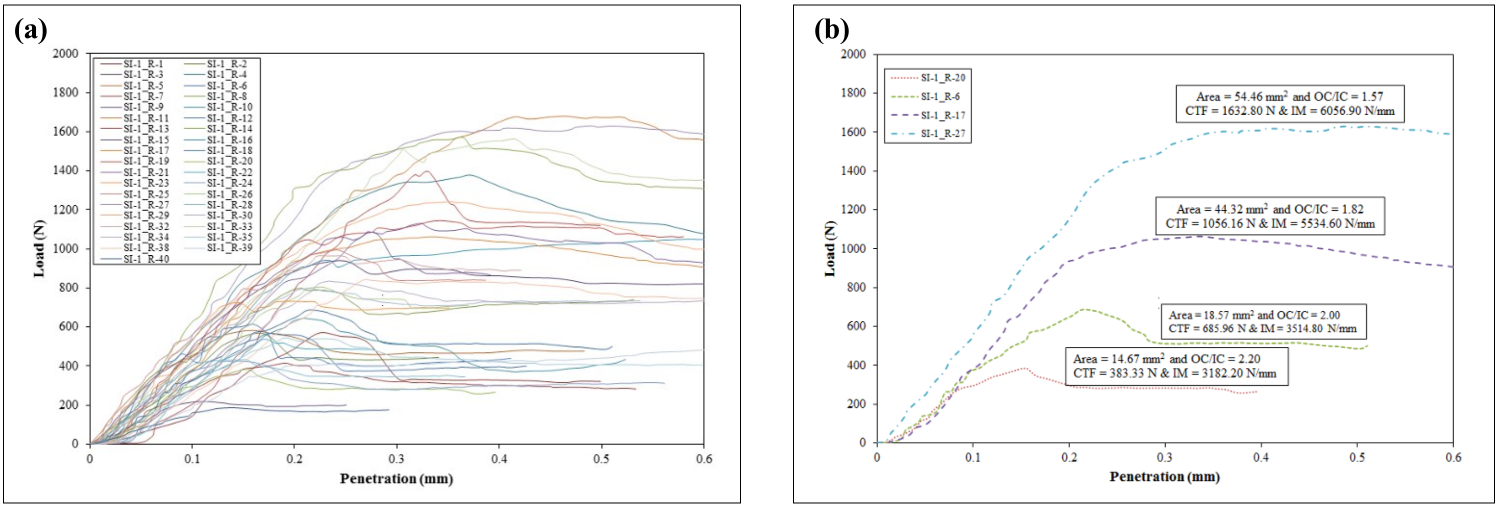

Fig. 5: (a) Load-penetration curves measured during the indentation tests on fragments of Sample S1, and (b) Load-penetration curves of the rock fragments shown in Fig. 3.

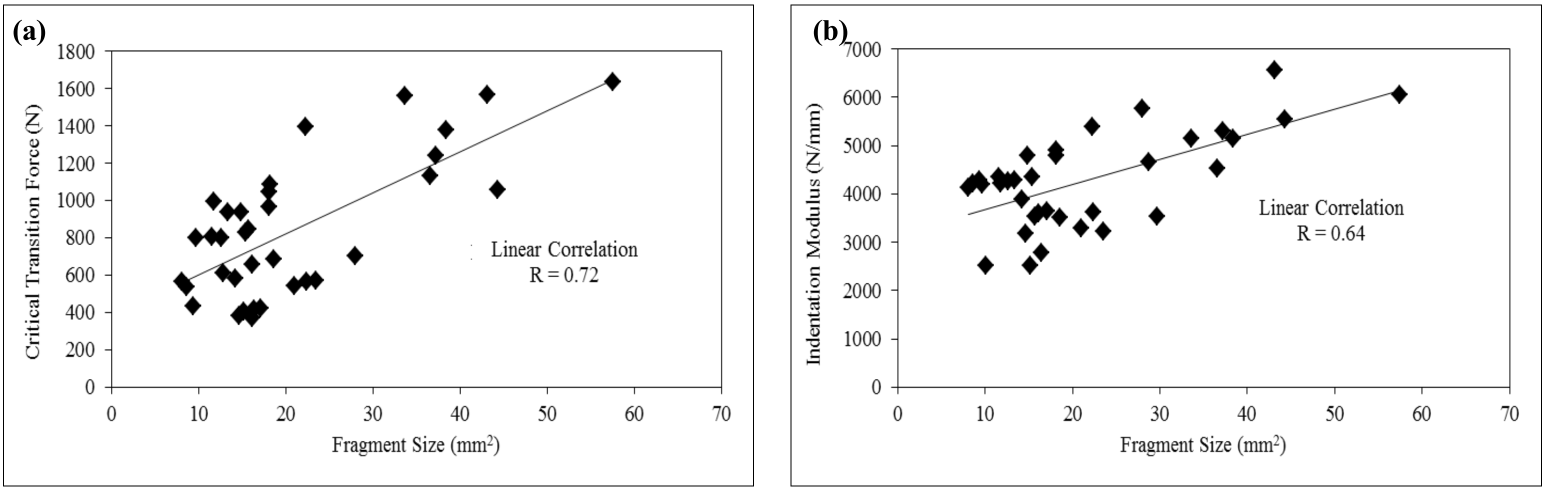

Fig. 6: Sensitivity of the conventional indentation indices to the fragment size for Sample S1.

Statistical approach was used to determine the required number of tests to obtain an acceptable CV_{ob} and a reasonable precision value. Table 3 and Fig. 4 show the CV_{ob} ranges from 29-50% for the critical transition force and from 17-28% for indentation modulus. The average values of CV_{ob} for CTF and IM are 40.8% and 22.9%, respectively. The required number of tests can then be estimated as a function of precision indices and coefficients of variation for a 95% confidence interval using Eq. 5; the results are shown in Table 4.

Table 4: Required number of tests from Eq. (5) according to *p* value for the investigation importance.

Sample No.	Number of Tested Samples	Indentation Modulus				Critical Transition Force			
		CV_{ob} (%)	Required Test Number			CV_{ob} (%)	Required Test Number		
			$p \leq 1.50$ [a]	$p \leq 1.35$ [b]	$p \leq 1.20$ [c]		$p \leq 1.50$ [a]	$p \leq 1.35$ [b]	$p \leq 1.20$ [c]
S1	39	26.88	8	14	37	48.23	25	44	116
S2	37	17.61	4	7	16	41.47	19	33	87
S3	40	20.70	5	9	22	33.94	13	22	58
S4	35	26.21	8	14	35	49.97	24	42	111
S5	39	18.09	4	7	17	42.38	19	34	90
S6	30	28.11	9	16	41	28.62	10	16	42

a: Protodyakonov (1960, 1969) and Vutukuri et al. (1974) for laboratory investigations.

b: Gill et al. (2005) for long life mining structures and civil engineering works.

c: Gill et al. (2005) for vital underground excavation and research work.

Note that the required number of tests varies in relation to the importance of the investigations. Larger number of tests is required to achieve a higher precision index. The number of fragments in this investigation satisfies the $p \leq 1.20$ and $p \leq 1.35$ for IM and CTF, respectively. Thus, the number of test specimens in this study, i.e. 35-40 for each specimen group, provided a representative mean value and guaranteed the accuracy of correlation between indentation indices and UCS based on small sampling theory (see Table 4).

Regarding to the number of tests carried out in this investigation and guides on p-value, field applications of the correlation equations were recommended; correlation equations between IM_n and UCS can be applied for all engineering applications while those of the CTF_n and UCS can be used for long-life mining structures and civil engineering projects. All of the suggested equations are applicable for the laboratory investigations.

Generally, possible factors causing scattering of results in a specimen group can be the size of fragments, heterogeneity, fragment microstructure, diameter and thickness of disk, number of fragments in a specimen group, and the mechanical properties of resin. The last three parameters were kept constant in all these tests to minimize their influence on the test results. The number of fragments in a specimen group can directly influence the indentation parameters. However, their effects can be minimized when considering a reasonable precision index based on the project importance. Using statistical methods for determining the required number of tests, results in a representative population and reliable assessment of the mean value of indentation parameters. Based on the findings in this investigation, the fragment size had the greatest effect. Therefore, we suggest normalizing the indentation indices by the fragment size to reduce the size effect.

3.2 Normalized indentation indices

The obtained IM and CTF were normalized using Eqs. (1) and (2), see Table 5. The mean normalized indices for each sample were plotted against the corresponding UCS to examine possible correlations. Different types of regression curves were used and their correlation coefficients were determined. Ringstad et al. (1998) and Mateus et al. (2007) used a linear regression while Santarelli (1996) and Zausa et al. (1997) employed exponential function for similar correlations. We examined both linear and exponential regression types (Fig. 7) and the correlation equations were obtained as follows:

Linear regression for:

IM_n and UCS $\quad UCS = 0.20\ (IM_n) - 226.21 \quad R = 0.91 \quad (6)$

CTF_n and UCS $\quad UCS = 0.48\ (CTF_n) - 19.36 \quad R = 0.74 \quad (7)$

Exponential regressions for:

IM_n and UCS $\quad UCS = 3.32\ e^{0.0019\ (IMn)} \quad R = 0.97 \quad (8)$

CTF_n and UCS $\quad UCS = 20.62\ e^{0.0049\ (CTFn)} \quad R = 0.85 \quad (9)$

The correlations of IM_n and CTF_n with UCS were reasonable as evidenced by the correlation coefficients of greater than 0.74. The correlation coefficients obtained from both regression types are close, although that of exponential is slightly higher (greater than 0.85). Also, correlation of the IM_n with UCS shows higher correlation coefficients, which confirms less dependency of this parameter on the sample size.

Table 5: Results of the normalized indentation indices using Eqs. (1) and (2).

Sample No.	Normalized Indentation Indices	
	IM_n (N/mm)	CTF_n (N)
S1	2242.57	434.60
S2	2234.90	436.50
S3	2207.17	531.53
S4	1786.12	303.71
S5	1979.72	440.38
S6	1220.60	149.93

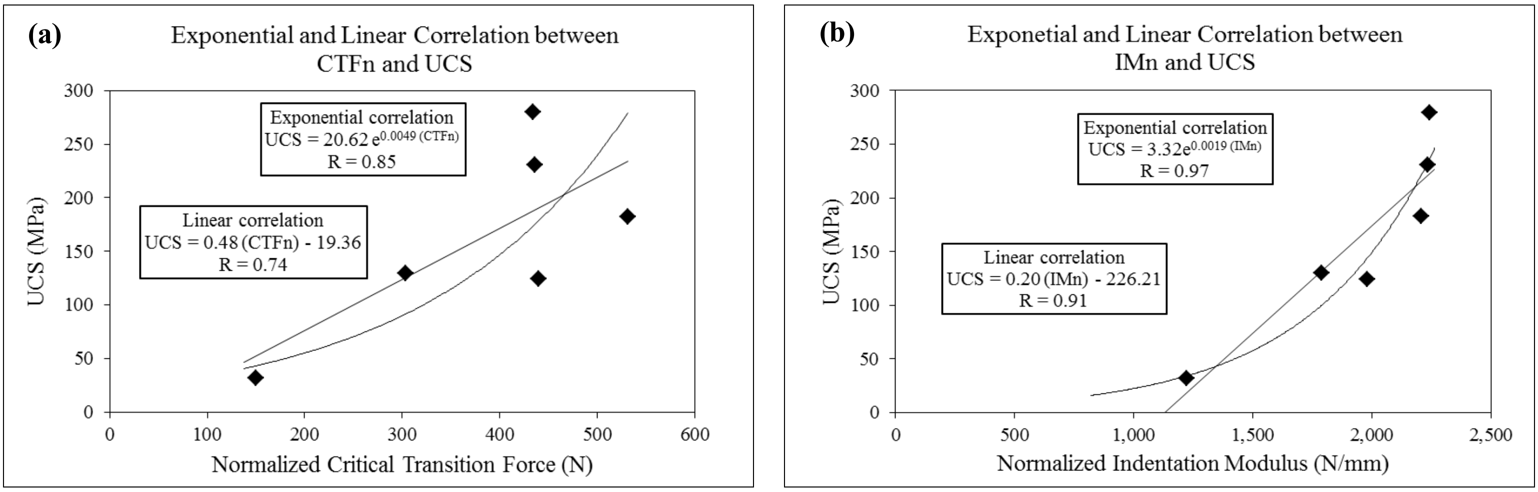

Fig. 7: Correlation of the UCS with the normalized indentation indices for linear and exponential regressions for Samples S1 to S6.

3.3 Verification of the correlation equations

To verify Eqs. (6) through (9), the UCS of three limestone samples (Blocks S7, S8 and S9) were predicted using the exponential and linear equations and were compared to the measured UCS of intact rock core samples. Totally, 106 indentation tests were carried out on rock fragments of Samples S7-S9 and the indentation indices were determined from the load-penetration curves (see the average values in Table 6). Next, the indentation indices were normalized using Eqs. (1) and (2) (see Table 6). The normalized indentation indices (IM_n and CTF_n) were utilized to estimate the UCS of the rocks using the linear (Eqs. (6) and (7) and exponential (Eqs. (8) and (9)) correlations. The estimated UCS values were plotted against the measured UCS and the correlation coefficient in the linear regression type was obtained using the least square method (Fig. 8). A strong agreement ($R \approx 1.00$) between the estimated and measured UCS resulted. The UCS estimated from both IM_n and CTF_n were accurate, but the estimated value by IM_n in the exponential regression type was closer to the measured value.

The laboratory UCS of Sample S7 to S9 was plotted along with the correlation curves of Fig. 7 to examine the agreement with the correlations (see Fig. 9). The estimated UCS values were reasonably close to both linear and exponential curves, but the estimated values were closer to the exponential correlation curves for IM_n and CTF_n.

It is to be noted that the UCS of samples used for developing the correlation equations was in the range of 30-280 MPa. Further the UCS of the verification samples was between 83 and 245 MPa. Extending the UCS range of the samples used in the correlation development can further refine the correlation.

Table 6: Results of the indentation tests on Sample S7, S8 and S9 for correlation verification. Conventional indentation indices were normalized using Eqs. (1) and (2) and the UCS values were estimated by Eqs. (6) to (9).

Sample No.	No. of Tests	Conventional Indentation Indices		Normalized Indentation Indices		Measured UCS by Lab Test (MPa)	Estimated UCS (MPa)			
							Linear Eqs. (6) and (7)		Exponential Eqs. (8) and (9)	
		IM (N/mm)	CTF (N)	IM_n (N/mm)	CTF_n (N)		UCS from IM_n	UCS from CTF_n	UCS from IM_n	UCS from CTF_n
S7	40	4113.35	936.96	2262.47	515.44	246.32	226.51	226.25	244.56	257.74
S8	32	4089.38	844.99	2108.73	432.47	178.45	195.75	186.72	182.61	171.65
S9	34	3788.02	745.83	1685.44	331.90	82.89	111.05	138.79	81.70	104.86

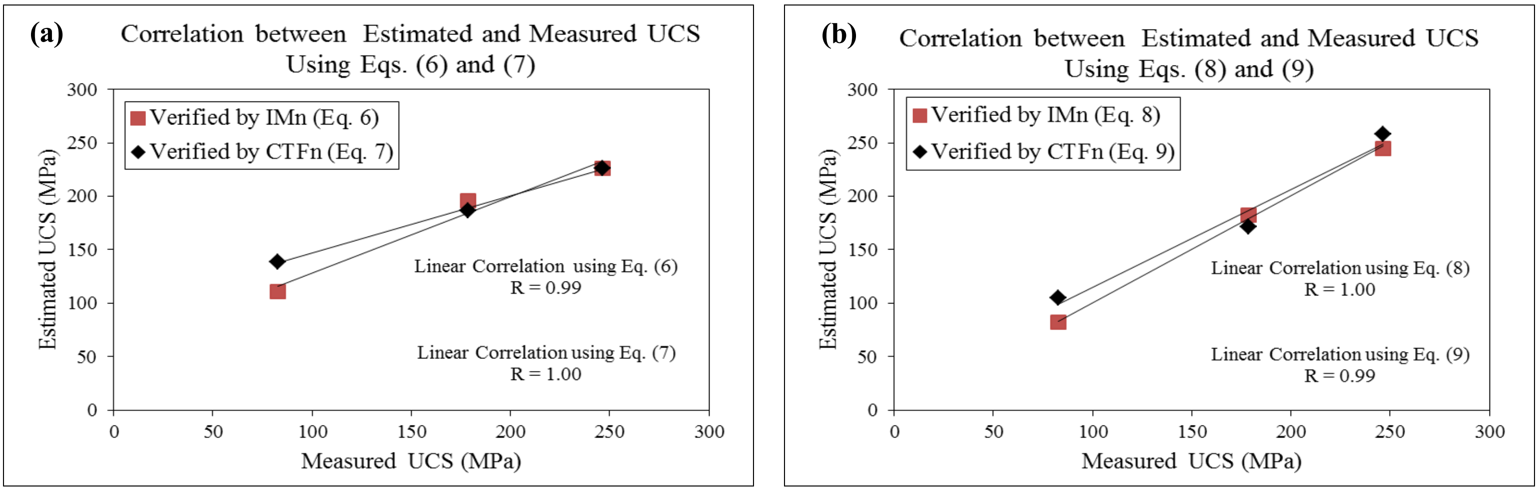

Fig. 8: Comparison of estimated and measured UCS of Samples S7 to S9 using a) linear correlation, Eqs. (6) and (7) and b) exponential correlation, Eqs. (8) and (9).

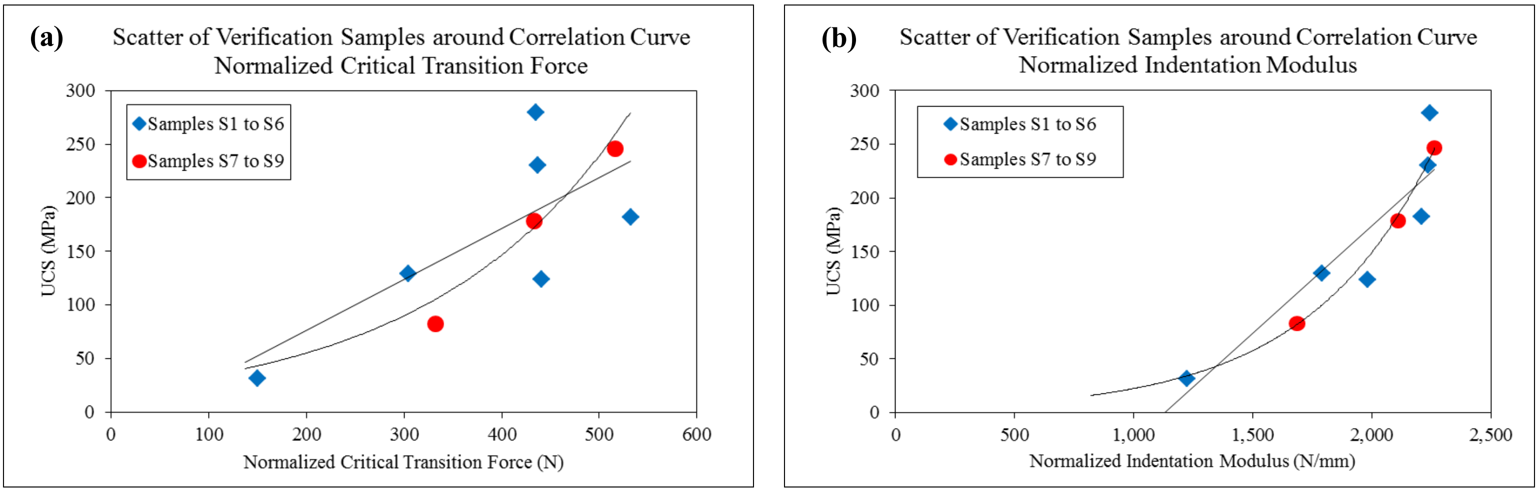

Fig. 9: Comparison of estimated UCS of Samples S7 to S9 with the correlation curves of Fig. 7 which shows a higher agreement with the exponential correlation curves (the blue diamonds present Samples S1 to S6 and the red circles Samples S7 to S9).

4. Conclusions

We used the indentation testing of rock fragments for determining indentation indices that can be employed to estimate the uniaxial compressive strength (UCS) of rocks.

The results of indentation indices are size dependent and thus we recommend using the normalized indentation indices for determining the UCS and doing indentation test on fragments of approximately the same size. Correlation coefficient of the normalized indentation modulus (IM_n) and the normalized critical transition force (CTF_n) with UCS was generally greater than 0.74 and 0.85 in linear and exponential regression, respectively.

Between the two normalized indices mentioned above, the IM_n showed less scattered results and stronger correlation coefficient.

The proposed correlations were verified using the normalized indentation indices of three other limestone samples that had not been used in developing the correlations. Results showed that the correlation coefficient between the estimated and measured UCS was near 1.00. Obviously, the near perfect match in this case is accidental and such a close match should not be generally expected.

The strong correlation between indentation indices and UCS, verification results and simplicity of indentation testing indicate great promise for real-time rock strength and borehole stability assessments.

It should be noted that the proposed correlations are valid only for the limestone rocks with rock fragment sizes between 2-5 mm using the same sample preparation method and testing manner as was presented in this paper. Further investigations are required to study the impact of the rock fragment sizes (particularly thickness and area), penetration rate and indenter diameter on the indentation indices as well as refining the correlations by including data from other geographical locations and different lithologies.

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