Influence of Penetration Rate and Indenter Diameter in Strength Measurement by Indentation Testing on Small Rock Specimens

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Abstract

Indentation testing has been developed as an unconventional method to determine intact rock strength using small rock specimens within the size of drill cuttings. In previous investigations involving indentation testing, researchers have used different indenter stylus geometries, penetration rates and specimen sizes. These dissimilarities can restrict applications of this method for strength measurement and lead to non-comparable results. This paper investigates the influence of indenter diameter and penetration rate on indentation indices for carbonate rocks to provide objective comparison and application of the existing correlations.

As part of this research, several indentation tests were conducted using different indenter diameters and penetration rates. The laboratory test results showed that indentation indices can be affected by the indenter diameter while the penetration rate has only minor effect on the indentation indices. Thus, a normalizing function was presented to reduce the dependency of test results to indenter diameter. Verification of the findings with independent data confirms the suitability of the suggested normalizing function in determining the rock UCS using testing data obtained from various indenter diameters and penetration rates.

Key words: Indentation Test, Drill Cutting, Indenter Diameter, Penetration Rate, Normalized Indentation Modulus, Uniaxial Compressive Strength, Wellbore Geomechanics.

1 Introduction

Unavailability of the intact core samples, expensive and time consuming coring and logging operations restrict the usual rock mechanical testing methods. Santarelli et al. (1996) found that drill cuttings or small rock specimens can be considered as a reliable source for characterizing physical and mechanical properties of formation rocks. Over the years, several unconventional laboratory tests on rock or drill cuttings have been established to generate the UCS values required for engineering applications at marginal cost without need to core samples. These tests include measurement of P and S wave velocities (Santarelli et al. 1996; Nes et al. 1998), reconstructed core samples (Mehrabi Mazidi et al. 2012) and indentation testing (e.g. Zausa and Santarelli 1995; Santarelli et al. 1996; Ringstad et al. 1998; Mateus et al. 2007; Garcia et al. 2008).

Theoretical aspects and important factors related to indentation testing of rock specimens were investigated and the followings were found:

- The indenter geometry is an important factor that impacts the stress distribution in the rock and leads to different rock responses and failure mechanisms (Thiercelin and Cook 1988; Suarez-Rivera et al. 1991). Yue and Xu (2006) indicated that flat-end cylindrical indenters are preferred to other indenter geometries with reference to creep testing.
- Penetration rate affects the response of rock specimens and generally a higher value of stiffness is achieved as a result of a higher strain rate (Cook and Thiercelin 1989; Santarelli et al. 1991).
- Zausa and Santarelli (1995) and Ringstad et al. (1998) found that results of the indentation testing may significantly be affected by the specimen size.

Then, indentation testing technique was adapted to drill cuttings for the assessment of rock strength (Zausa and Santarelli 1995) and the results were presented as index values directly related to the rock UCS (Uboldi et al. 1999). Later, several experimental correlations were developed to predict the UCS of sandstones, limestones and shales from indentation indices. But, they used different testing procedures and indenter designs:

- Cook et al. (1984) carried out the tests by flat-end indenters with 5-20 mm diameter. Santarelli et al. (1996) and Ringstad et al. (1998) utilized a flat-end indenter with 1 mm diameter and Magnenet et al. (2009) applied flat-end indenters with diameters of 2 and 5 mm. Zausa and Santarelli (1995) used a spherical indenter of 1 mm diameter and Brooks et al. (2010) employed a diamond-tipped indenter.
- Different penetration rates were used; e.g., penetration rates of 0.05 mm/s (Shao-Quan et al. 1995) and 0.01 mm/s (Mateus et al. 2007; Garcia et al. 2008).

- Dimension of rock specimens were variable; ranging from 2 to 50 mm.

Obviously, the above investigations resulted in different correlation equations, which were affected by the different testing procedures. Thus, the mentioned dissimilarities restrict application of the existing correlations. In a research project, the authors showed the considerable effect of specimen thickness on the indentation test results and introduced size functions to normalize the indentation indices (Haftani et al., 2013). Thus, in this study, the same-sized specimens were used to investigate the effect of indenter diameter and penetration rate on the indentation indices for determining the UCS of limestone.

2 Backgrounds on Indentation Testing

Conventionally, from the load penetration curve of the indentation testing, two main indices are derived; Critical Transition Force (CTF) and Indentation Modulus (IM).

Indentation modulus (IM) is the slope of the linear part of the load- penetration curve which is defined as the resistance of rock against penetration of the indenter (hereafter called conventional indentation modulus). This index has been used by several researchers for determination of intact rock UCS (Thiercelin 1989; Suarez-Rivera et al. 1991; Zausa and Santarelli 1995; Santarelli et al. 1996; Ringstad et al. 1998; Uboldi et al. 1999; Mateus et al. 2007; Garcia et al. 2008). Recently, an experimental correlation equation was presented to determine the rock UCS that involved the normalizing factor due to the different specimen sizes as (Haftani et al. 2013):

$$UCS = 3.48\, e^{0.0014(IM_n)} \qquad (1)$$

$$IM_n = \frac{IM}{T^{0.31 + \left(\frac{8.27}{UCS_{assu.}}\right)}} \qquad (2)$$

where, IM_n (N/mm) is the normalized indentation modulus by the size function, IM (N/mm) is conventional indentation modulus, T (mm/mm) is the ratio of specimen thickness to the unit value (i.e. 1 mm) and $UCS_{assu.}$ (MPa) is an assumed value for UCS. An iterative process should be used to estimate UCS by Eq. (1).

In this paper, testing approaches and rock specimens similar to the work by Haftani et al. 2013 were used to modify the Eq. 1 concerning the effects of indenter diameter and penetration rate on the indentation test results to be applicable for the testing with different testing procedures.

3 Experimental Work

3.1 Test design

To investigate the effect of indenter diameter and penetration rate on indentation indices, two testing procedures were implemented (Fig. 1):

- Procedure 1: Three penetration rates of 0.0025, 0.005 and 0.01 mm/s were examined. Here, a flat-end indenter with the diameter of 1 mm was used (similar to the indenter geometry used by Santarelli et al. 1996; Ringstad et al. 1998; Yue and Xu 2006; Mateus et al. 2007; Garcia et al. 2008; Haftani et al. 2013).
- Procedure 2: Using a cylindrical flat-end indenter stylus, the indenter diameters of 0.6, 1.0 and 1.5 mm were used. In this case, a constant penetration rate of 0.01 mm/s was applied during testing (similar to the penetration rate utilized by Mateus et al. 2007; Garcia et al. 2008; Haftani et al., 2013).

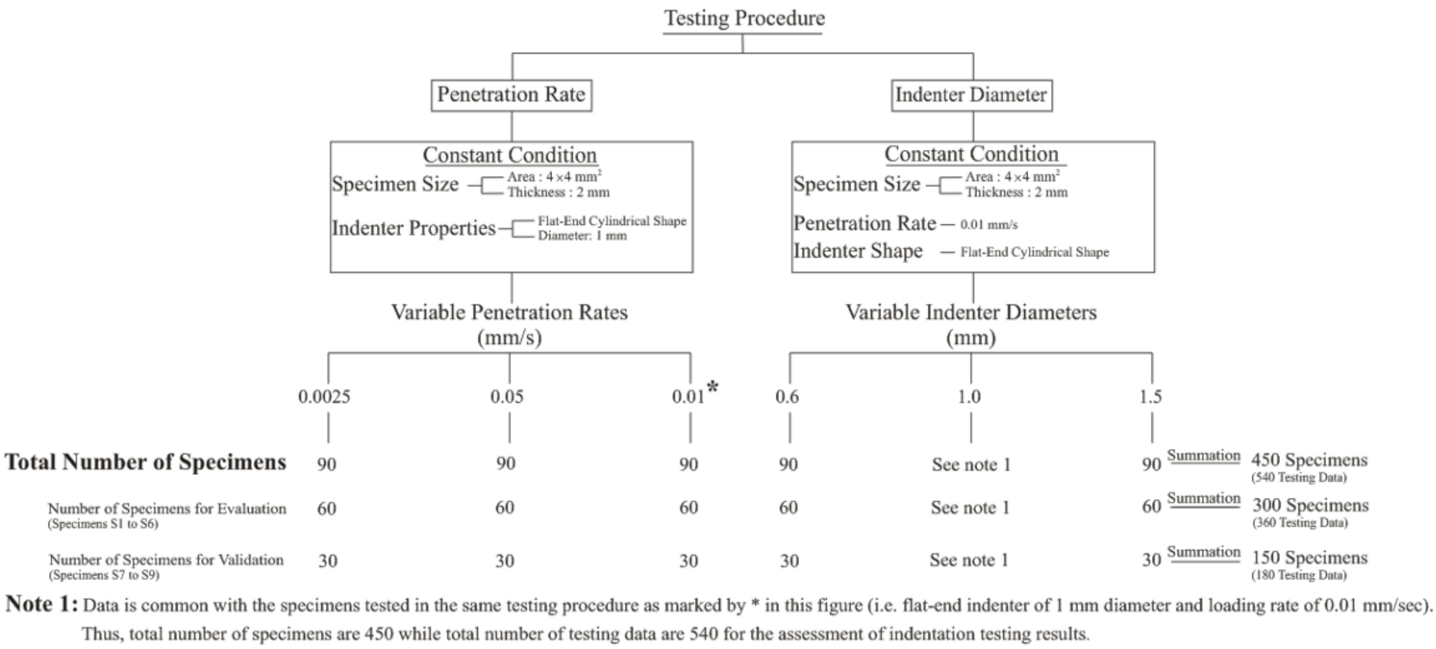


Fig. 1: a) Testing procedure showing constant and variable testing conditions as well as number of specimens in each testing procedure.

In both testing procedures, the laboratory tests were carried out on small rock specimens with the same dimensions to factor out the effect of different specimen size. The specimen cross section was 4×4 mm2 and the thickness was 2 mm. The indenters used for this study were cylindrical indenters made of tungsten carbide with ASTM hardness of 91 Rockwell B to apply load on the rock specimens. During testing, the applied load and penetration rate were recorded by a load cell and a displacement sensor, respectively (Fig. 2).

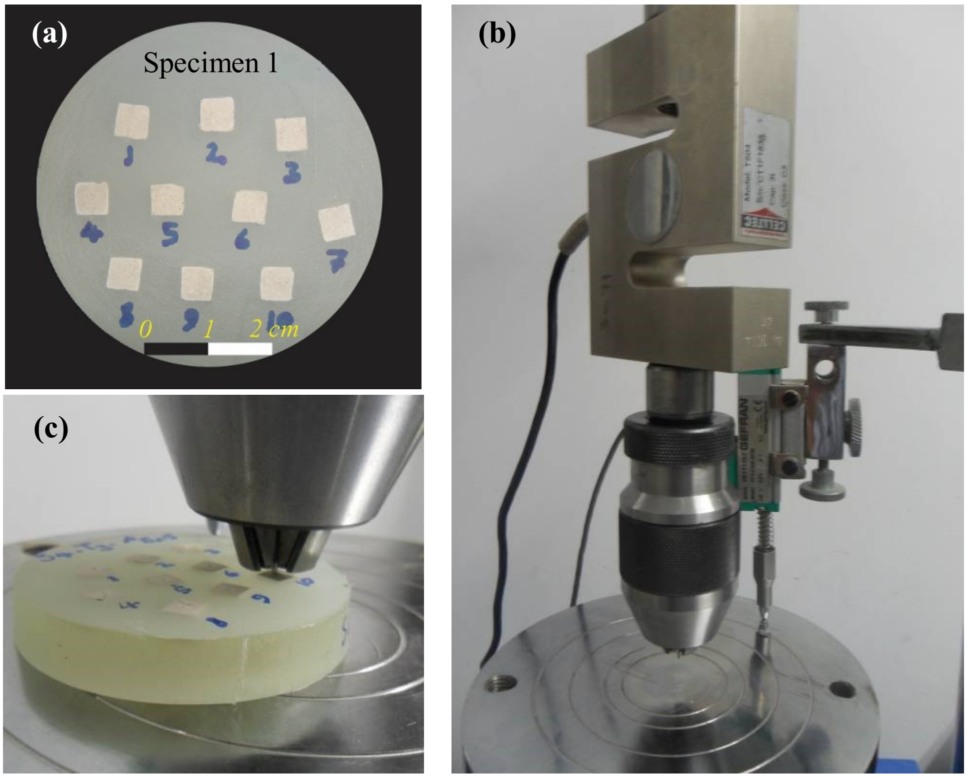


Fig. 2: a) Artificial rock cuttings embedded in epoxy; each epoxy pool contained 10 rock specimens, b) indentation equipment, and c) cylindrical flat-end indenter stylus applying normal loads to rock specimens.

For determining the required minimum number of tests (N) to provide a statistically acceptable representative mean value, the small-sampling theory was used (Gill et al. 2005).

$$N = \left[\left(\frac{p+1}{p-1} \right) t_{\beta} \frac{CV_{ob}}{100} \right]^2 + 1 \tag{3}$$

where, CV_{ob} is the calculated coefficient of variation after testing, 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), and p corresponds to the reasonable precision index. Generally, different p values were suggested depending on the investigation/project importance; e.g. 1.50 to 1.20 for the projects with fewer and higher importance, respectively (Protodyakonov 1969; Vutukuri et al. 1974 and Gill et al. 2005). Here, based on the guidelines of Gill et al. (2005) for research work in a project with high importance, $p \leq 1.20$ was used.

3.2 Specimen preparation

In this study, nine types of limestone from different locations throughout Iran with the UCS values of 30 MPa to 280 MPa were utilized. Rock specimens were generated from six boulders (numbered S1 to S6) for the assessments and from three others (S7, S8 and S9) for validation purposes. Using standard laboratory testing methods, physical and mechanical properties of the intact rocks were determined (Table 1).

Table 1: Physical and mechanical properties of the rock blocks used for indentation testing.

Specimen 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

Due to the size dependency of the indentation test results, the tests should be carried out on specimens with the same dimensions. For this reason, rock blocks were cut by a diamond saw machine into rectangular cuboids and then ground at the faces to the dimensions of $4\times4\ m^2$ and thickness of 2 mm.

In order to contain the specimens during the test and to provide a flat surface for load application, every 10 rock specimens were embedded in a disk shaped epoxy-resin. The diameter of the disk was 55 mm and its thickness was 10 mm (Fig. 2). Once the resin cured, the disk was trimmed and polished to expose the square face of rock specimens for indentation testing (Fig. 2).

Correspondingly, a total number of 450 specimens were produced for the indentation testing. Data relevant to the indentation testing of 90 Specimens tested with the same testing procedure (i.e. flat-end indenter of 1 mm diameter and loading rate of 0.01 mm/sec) were used in both the penetration rate and indenter diameter procedures as common/shared data (Fig. 1).

The rock blocks used in this investigation were composed of homogeneous calcareous particles of less than 4 μm, which can be classified as micritic limestones using the carbonate rock classification (Flugel, 2004). Consequently, effect of the grain size can be neglected.

4 Evaluations of Test Results

4.1 Test results

A total of 360 indentation test results were used to assess the effect of the penetration rate (180 tests) and the indenter diameter (180 tests) on the indentation index. The conventional indentation modulus was determined from the load-penetration curve. Average values of conventional indentation modulus were normalized using Eq. (2) (see Section 2) for testing with different penetration rates and indenter diameters and presented in the form of Normalized Indentation Modulus (IM_n) in Table 2. To define IM_n, $UCS_{assu.}$ is the UCS value measured using the suggested method, by ISRM 1979. Also, the pertinent coefficients of variation were calculated for each specimen group comprised of about 10 rock specimens in each disk (Table 2).

Table 2: Normalized indentation modulus and the coefficient of variation from indentation testing at several penetration rates and indenter diameters.

Different Penetration Rates						
Specimen No.	0.0025 mm/s		0.005 mm/s		0.01 mm/s	
	IM_n	CV_{ob}	IM_n	CV_{ob}	IM_n	CV_{ob}
S1	2458.60	6.87	2893.74	9.98	2533.01	8.69
S2	2550.88	3.80	2803.35	7.94	3060.71	7.03
S3	2319.59	2.95	2720.35	6.08	2815.56	5.71
S4	2710.20	9.78	2691.84	10.08	2496.87	5.86
S5	2650.77	6.94	2332.08	6.34	2751.21	6.80
S6	1746.20	8.34	1835.42	13.82	1863.20	5.29
Different Indenter Diameters						
Specimen No.	0.6 mm		1.0 mm		1.5 mm	
	IM_n	CV_{ob}	IM_n	CV_{ob}	IM_n	CV_{ob}
S1	2040.95	5.54	2533.01	8.69	3586.11	5.74
S2	2143.48	9.56	3060.71	7.03	3341.08	5.03
S3	2204.76	5.32	2815.56	5.71	3543.33	3.79
S4	1876.05	7.86	2496.87	5.86	3483.75	3.48
S5	1754.67	6.62	2751.21	6.80	3353.68	9.04
S6	1299.78	8.34	1863.20	5.29	2319.86	8.81

Normalized Indentation Modulus (IM_n) in N/mm.
Coefficient of Variation (CV_{ob}) expressed in %.

Average values of the coefficient of variation for the IM were used to find the required minimum number of specimens using Eq. (3): 7.35% and 6.58% for testing of different penetration rates and indenter diameters, respectively. Considering a precision index of 1.2 with confidence interval of 95% (recommended by Gill et al. 2005), the required minimum number of specimens was computed, consisting of 8 to 10 indentation tests.

4.2 Effect of the penetration rate

Three different penetration rates (PR) of 0.0025, 0.005 and 0.01 mm/s were used for indentation testing of 180 rock specimens. The Conventional indentation modulus was normalized using Eq. (2) and the results are presented in Table 2. The normalized indentation moduli for the different penetration rates are plotted in Fig. 3. It shows that variation of the penetration rate in the range of 0.0025 to 0.01 mm/s has no considerable effect on the test results. Deviation of the normalized indentation modulus of each rock specimen from the mean value is less than 7% for various penetration rates.

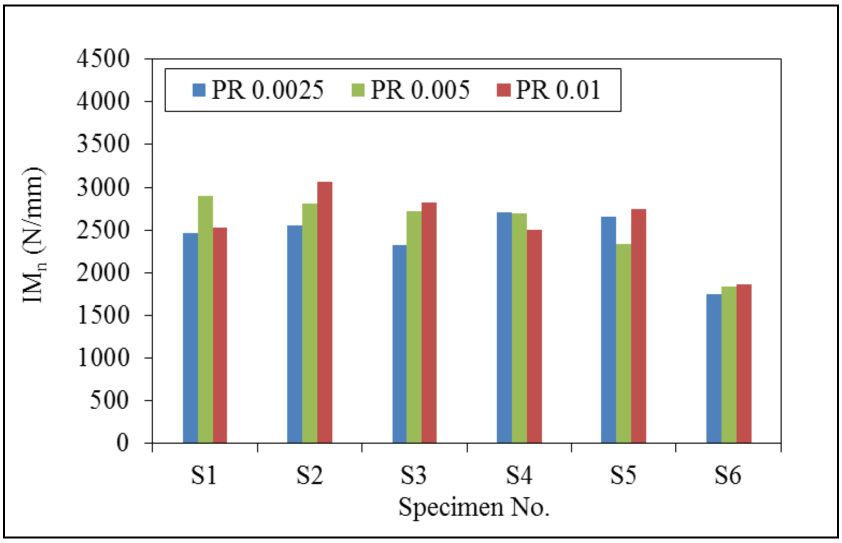


Fig. 3: Relationship between the conventional indentation modulus and the penetration rates.

4.3 Effect of the indenter diameter

Indentation testing results on 180 rock specimens with different indenter diameters (ID) (see Table 2) showed significant differences between the normalized indentation modulus from various diameters (Fig. 4). The larger the diameter of indenter, the greater is the value of normalized indentation modulus (IM_n).

Normalized indentation modulus of specimens was plotted against the pertinent indenter diameter to find out the amount of indenter diameter effect on test results. The best lines were drawn for each set of specimens and the following formula was proposed to relate the indentation modulus to the indenter diameter, ID (Fig. 5):

$$IM_n = d\,(ID)^k \tag{4}$$

where, IM_n is the indentation modulus normalized by specimen size and k is the power in the correlation equation measured from each line in Fig. 5. In this way, the k value is considered as the factor affecting the indentation modulus due to the different indenter diameters.

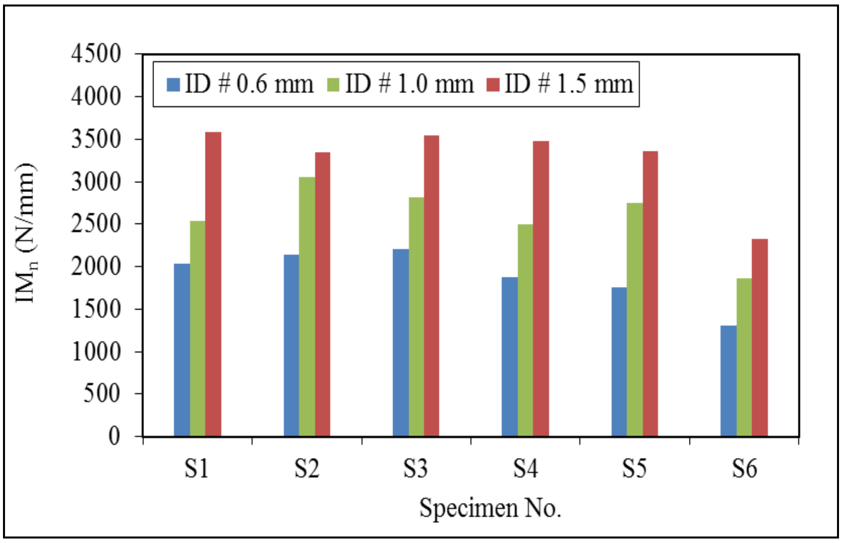
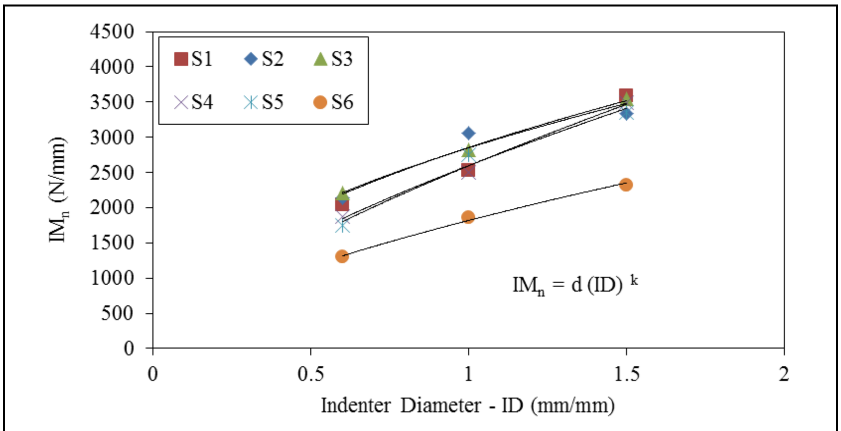


Fig. 4: Relationship between the conventional indentation modulus and the indenter diameters.



Specimen No.	S1	S2	S3	S4	S5	S6
k - value	0.61	0.49	0.52	0.67	0.71	0.63

Fig. 5: Indentation modulus variation with indenter diameter.

From the similarity of this value in different specimens, the average value of k was used for normalizing the indenter diameter effect, which is 0.60. Thus, a normalizing function was suggested as:

$$IM_{n-ID} = \frac{IMn}{ID^{0.60}} \quad (5)$$

where, $IM_{n\text{-}ID}$ is the indentation modulus normalized by size and indenter diameter.

To check the performance of the normalizing function in reducing the dependency to indenter diameter, the IM_{n-ID} values were plotted on the same graph (Fig. 6). The values are quite close in each specimen.

Combining the Eqs. (2) and (5) gives:

$$IM_{n-ID} = \frac{IM}{T^{0.31+\left(\frac{8.27}{UCS_{assu.}}\right)} \times ID^{0.60}} \tag{6}$$

So, it is essential to add this normalizing factor to the original equation (i.e., Eq. 3) as:

$$UCS = 3.48\, e^{0.0014(IM_{n-ID})} \tag{7}$$

Substitution of Eq. (6) in Eq. (7) gives:

$$UCS = 3.48\, e^{0.0014\left(\frac{IM}{T^{0.31+\left(\frac{8.27}{UCS_{assu.}}\right)} \times ID^{0.60}}\right)} \tag{8}$$

An iterative progression should be used for estimation of UCS from this equation as followings:

- First, assume a value for UCS and insert into the right side of the equation (UCS_{assu}) and calculate UCS.
- Next, the calculated UCS is considered as the UCS_{assu} in the right side and recalculate the equation.
- Again back to first step and continue this sequence until the assumed UCS (UCS_{assu}) and the calculated one become identical.

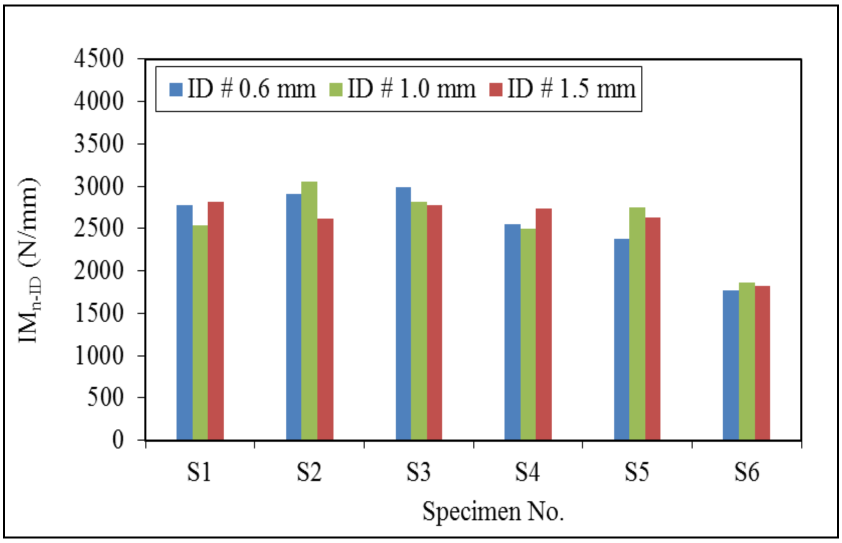


Fig. 6: Variation of normalized indentation modulus with indenter diameter.

5 Verification of the correlations

Independent test data from rocks that were not used in above assessments (Blocks S7 through S9) were utilized to validate the modified correlation Eq. (8). Physical properties and strength (UCS) of the rock blocks were measured, as presented in Table 1.

Totally, 180 indentation tests (90 tests to verify the effect of indenter diameter and 90 tests to verify the effect of penetration rate) were carried out on rock specimens and the conventional indentation modulus was determined from the load-penetration curves. Calculated average values of the coefficients of variation for the IM were generally less than 10% for both penetration rate and indenter diameter. Using the average value of CV_{ob} and considering a precision index of 1.2 with confidence interval of 95%, the adequately of 8-10 indentation tests to calculate the representative mean value was confirmed.

Using Eq. (8), UCS values were predicted and compared to the measured value (Table 3). In Fig. 7, the scatter of measured and estimated UCS values for specimens S7-S9 are shown relative to the line X=Y. The estimated UCS values are reasonably close to the measured UCS values.

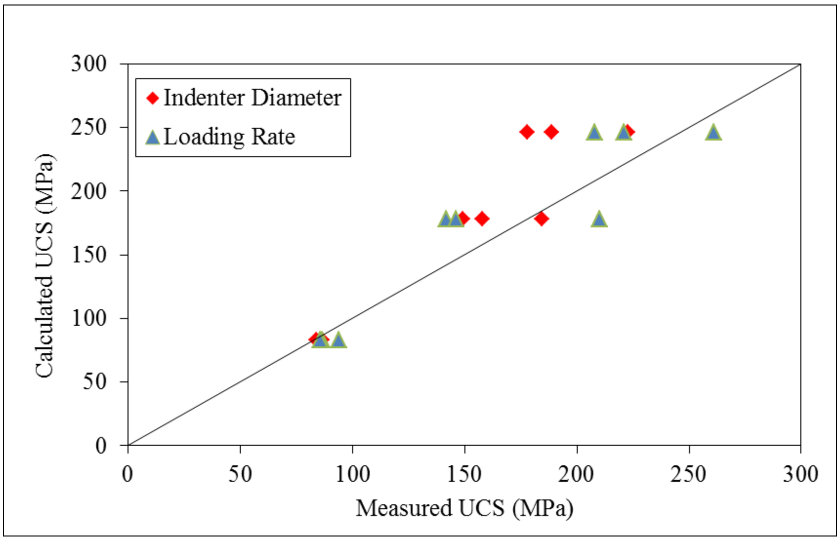


Fig. 7: Investigating the scatter of the predicted and measured UCS values of rock blocks S7 to S9 around the linear relation (i.e. X=Y).

Table 3 provides additional information on the performance of these correlations. The correlation against $IM_{n\text{-}ID}$ indicates a maximum prediction error of less than 20%, which may be considered a reasonable accuracy where no cores are available for UCS testing.

Table 3: Measured versus predicted UCS from the modified correlation equation associated with the difference between the measured and predicted UCS in percent.

Specimen No.	Measured UCS (MPa) Standard ISRM Testing on Core Sample	Calculated UCS (MPa)			Difference between the measured and predicted UCS (%)		
		Penetration Rate (mm/s)					
		0.0025	**0.005**	**0.01**	**0.0025**	**0.005**	**0.01**
S7	246.32	192.52	182.59	222.74	10.35	15.61	5.91
S8	178.45	130.67	134.32	184.49	20.53	17.99	17.87
S9	82.89	91.07	84.77	84.07	13.24	4.34	3.35
		Indenter Diameter (mm)					
		0.6	**1.0**	**1.5**	**0.6**	**1.0**	**1.5**
S7	246.32	188.66	222.74	177.95	23.41	9.57	27.76
S8	178.45	157.96	184.49	149.06	11.48	3.39	16.47
S9	82.89	89.95	84.07	86.63	3.69	1.42	4.52

6 Conclusions

Different designs of the indentation testing apparatus and procedure limit the application of the indentation parameters for predicting the rock UCS. In this study, limestone rock blocks with various UCS (in the range of 30-280 MPa) were used to investigate the influence of the indenter penetration rate (0.0025, 0.005 and 0.01mm/s) and indenter diameter (0.6, 1.0 and 1.5 mm) on the results of indentation testing. From laboratory testing on artificially made small rock specimens, the conventional indentation modulus was extracted and normalized using a specimen size function. The normalized indentation modulus is affected by the indenter diameter but it is not susceptible to the variation of the penetration rate in the range of 0.0025 to 0.01 mm/s. Higher normalized indentation modulus is obtained when increasing the indenter diameter. So, due to the dependency to indenter diameter, a normalizing factor was proposed to modify the experimental correlation equation for strength measurements. The modified correlation equation predicts the UCS of limestone rocks with an error of less than 20%, which may be considered a reasonable accuracy for UCS assessments where no cores are available for direct UCS testing.

The proposed correlations are valid only for limestone rocks using the same specimen preparation method as presented in this paper. Further investigations using rock blocks from other geographical locations and lithologies are required to refine the correlations. In this paper, a flat-end cylindrical indenter was used, but this work will be further continued and improved by investigating the potential differences on the predictive results for pointed and rounded (conical, ogee, hemispherical, etc.) indenter shapes.

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