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Effect of Micro-Cellulose on Mechanical Properties of Class C and H Cement at Room and Elevated Temperature

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ABSTRACT

Characterization of short- and long-term mechanical properties is the key to understand cement behavior in different scenarios. Generally, room temperature testing conditions are used as reference material properties while elevated temperature conditions to simulate downhole conditions. The understanding of the cement mechanical properties such as Unconfined Compressive Stress (UCS) will guarantee the correct selection of the cement that will meet the requirements for each specific well.

The cement characteristics of the selected cement type can be improved by additives to match the requirements for each well in terms of density, strength, curing time and so on, in this paper the effect of micro cellulose (MC) will be investigated and described.

Micro-cellulose (MC) has been reported as a great additive in geothermal well fluid loss curing solutions. Given the recent success of using Micro-Cellulose in curing loss circulation and providing Wellbore Strengthening, addition of some amount to the cement slurry could inevitably be an option for cement fluid loss cure. However, the Micro-Cellulose can change the hydration process on the cement due to its natural characteristics, decreasing the compressive strength of the cement at the early stages; this phenomenon will be further described in the paper

The samples were prepared following current API RP 10B Recommended Practice for Testing Well Cement. Samples were cured at 1, 3, 7, at room temperature, additionally, samples were cured using baths with controlled high temperature of 75°C for 1, 3, 7, 14, 21 and 28 days with few of them at 35 days, then tested and plotted to generate a comprehensive understanding of its behavior.

1. INTRODUCTION

Oil well cementing is a procedure that helps to improve several aspects of well integrity over time and works as the primary barrier to isolate crossing formations and to protect casing, also, helps to avoid excessive costs on problems related to bad cementing practices such as remedial cementation and helps to achieve a good well performance in long term, a good practice for well cementing is highly recommended although sometimes difficult to achieve due to the lack of the understanding of the mechanical properties of cement, properties that includes mix ability, stability, rheology, fluid loss, and adequate thickening time (Al-Yami et al., 2017). The cement provides zonal isolation, hydraulic seal from unwanted formations, seals loss circulation zones, protects the casing from corrosion and holds casing and completion string. (Ichim, K Saleh, Teodoriu and Sondergeld, 2019) It has been shown that mechanical properties of the annular sealant are a critical factor in the success of a well, especially in HT/HP applications (Bosma, Ravi, van Driel and Schreppers, 1999).

Analogous to oil and gas wells, geothermal wells represent some unique challenges for operations related to cement, in order to achieve proper cement placement and curing. The biggest challenge is the low fracture gradients and the considerable loss of circulation due to a great number of fractures; the design of the primary cementing is critical to ensure long-term integrity of a geothermal well and to guarantee the cement integrity in the continuous cycles when the steam is produced. Temperature design is also an important factor, since it is well known that temperature impacts massively in the curing and set time of cement, the phenomenon in which the cement is exposed to high temperatures and the mechanical properties such as strength decreases is known as strength regression,

Regularly geothermal wells targets into naturally fractured formations, in order to produce steam that injects energy into turbines and therefore generates electricity, generally the kind of formations aiming for geothermal consists in igneous rock, therefore, due to fractures in the volcanic formation, circulation losses always occur in geothermal wells. Lost circulation can be due to natural fractures, induced fractures, or even cavernous formations. In most cases, in the event of circulation losses before reaching the reservoir zone, whereas for oil and gas purposes sedimentary rock formations are more common, this important difference affects directly to the cementing should approach to meet the goal, in order to overcome strength retrogression due to high temperatures in geothermal wells, it is a common practice to add 35% to 40% of silica content, to face the high temperatures found downhole (Rachimillah, Azwar, Johri, Osman & Tanoto, 2021). In novel applications (Buelichen & Plank, 2011 and Brandl, Bray & Magelky, 2012), the use of cellulose has shown an important improvement to ameliorate the fluid loss.

Rapid dehydration during cement slurry placement has been reported when fractures are found, which leads to poor pumpability, affecting the cement hydration process. When fractures are encountered some additives (Smith 1990) are used to reduce water loss from the slurry while a porous formation is encountered. (Fink 2003; Nelson 2006). These additives are generally known as FLA (fluid loss additives)

According to previous works by Desbrières (Desbrières 1993/1; Desbrières 1993/2), three fundamental working mechanisms for polymeric FLAs are known: First, increased dynamic viscosity of the cement filtrate stemming from polymer addition can decelerate the filtration rate. Second, anionic FLAs may adsorb onto hydrating cement particles and obstruct cement filter cake pores either by polymer segments which freely protrude into the pore space, or which even bridge cement particles. this adsorptive mechanism, filter cake permeability is reduced, and low fluid loss is achieved. And third, once a certain polymer dosage is exceeded, FLAs may plug cement filter cake pores through formation of a polymer film or through associates which can bind an enormous amount of water molecules in their inner sphere and hydrate shells. This way, a large portion of the mixing water is physically bound and will not be released during the filtration process. (Buelichen & Plank, 2011)

2. MATERIALS AND METHODS

This section will describe the materials and methodology used throughout the experiments, sample preparation methods and the equipment used to test the samples. The proposed methodology is following the same steps that has been used in the past for preparation of other cement recipes some of which have been also presented (Ichim et al., 2017, Teodoriu et al. 2014a, 2014b)

2.1 **Mold preparation**

Samples were prepared in stainless steel molds with 2 inches for each side in accordance with ASTM C109, molds were greased prior cement pouring. Same grease and preparation procedure was used for all samples. Cubes are used as per API RP 10B-2.

2.2 **Sample preparation**

The sample preparation was conducted by *API RP 10B-2: Recommended Practice for Testing Well Cements*, Distilled water was used in the mixing process in order to maintain consistency, especially since it has been shown that the cement properties are directly affected by the water quality (Saleh et al. 2018). The water cement ratio and the amount of additive are shown in Table 1. GRC consultants have reported that 2 to 4% by volume of added MC to drilling muds have shown the best efficiency in curing mud losses. Thus 4% MC by Weight of Cement has been proposed for the current experiments.

2.3 **Mixing procedure**

Digital scales were used to measure the correct amount of water and cement used in the mixture and mixed at a constant speed using OFITE-20 Constant Speed Blender which satisfies the requirements of API specifications. The amount of cement, water and micro cellulose is described in the Table 1. The amount of water was increased by 5% to compensate for the MC water needs.

Table 1: Mixing ratios for cements used in the paper

Type	Cement (gr)	Water (gr)	Micro-Cellulose (gr)
Class H + 10% Mc	860.26	343.245	86.02
Class H + 4% Mc	860.26	343.345	34.41
Class C + 10% Mc	683	401.604	68.3
Class C + 4% Mc	683	401.604	27.32

Following API recommendations, the cement was poured into the cup with the correspondent distilled water in the first 15 seconds, it is important to remind that the blender runs at constant speed of 4000 rpm within the first 15 seconds, after that the blend increase and maintains the speed of 12,000 rpm for the next 35 seconds.

2.4 **Curing process**

The mix coming from the mixing cup is poured into previously greased molds until half full, then the air is removed from the samples by shaking the mold to avoid any air contained in the mixture, then the other half of the molds is filled.

The samples are then submerged into a container with pure distilled water for the samples at room temperature, for the high temperature samples, special baths with regulated temperature are used. The curing time for room temperature was 1,3 and 7 days, whereas for high temperature 1, 3, 7, 14, 21 and 28 days test were performed. Some samples were tested after 35 days.

2.5 **UCS measurements**

Destructive and non-destructive tests were performed at the same time, having priority for the non-destructive test, the Ultrasonic Pulse Velocity test was performed using Proceq™ Ultrasonic Device with an accuracy of $\pm 2\%$. The UPV data was only use as a prescreening of the samples prior destructive tests. Destructive test is conducted by Test Mark Compressive Strength test machine (Figure 1) after the non-destructive test. The sample is placed between the plates and a uniaxial load applied to the sample until failure, the values are directly screened and recorded.

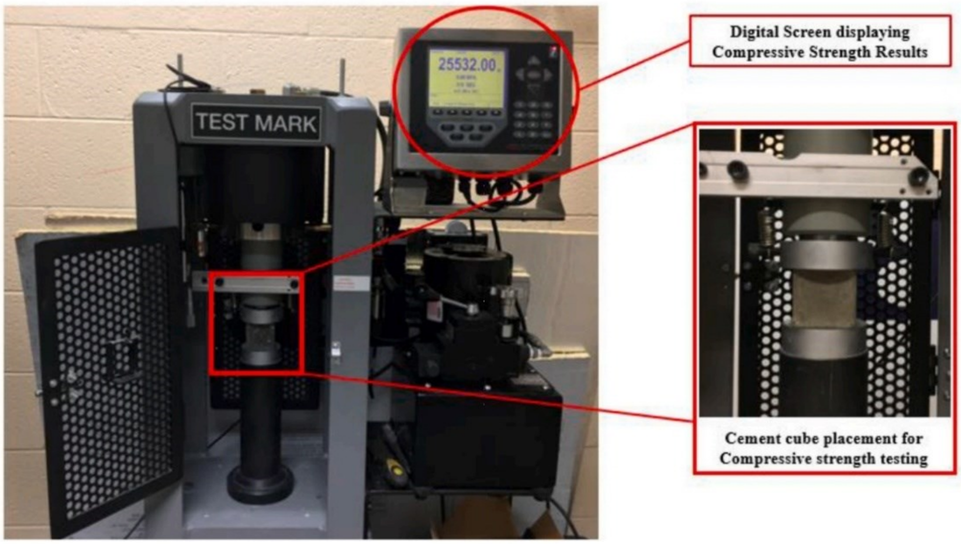

Fig. 1.- Test mark Compressive Strength test machine **(Arbad, Rincon, Teodoriu and Amani, 2021)**

3. RESULTS

The results for class C cement HT, class C + 4% MC HT, class H HT and class H + 4% MC are presented as following for which UCS is in the Y axis and Days in the X axis, in this way turns easier to identify when the compressive strength stabilizes, the beginning of strength degradation starts and to observe the difference between neat cement and cement with 4% MC, leading into information about the impact of MC in the cement hydration process. The data shown is based on a minimum of 3 samples for each curing time stage. If large variation was observed additional samples were prepared and tested. Figure 2 shows the data for Class C with 4%MC for room temperature (RT) and elevated temperature (75°C). It is observed from Fig. 2 that the trend for high temperature is to stabilize at 14 days, starting to observe strength regression from day 28, the data for high temperature shows a lower UCS than room temperature in the first week, which is caused by the hydration of MC

Figure 2 and 3 shows the measured UCS for class H and Class C cements at room and high temperature. Additionally control samples of the neat cement were prepared and matched with previous data to compare with future work to show the effect of MC. It is shown that the effect of high temperature in the hydration process gets accelerated, resulting in a higher compressive strength in a shorter time lapse.

In Fig. 4 a comparison between class C cement with 4% MC at room and high temperature, is shown. A decrease in the total UCS of the samples at room and high temperature were observed when compared to neat samples, also, the strength of the cement at high temperatures is diminished due to the change in the hydration process due to natural effects of micro cellulose. Since the main objective of this study is to show the impact of MC at elevated temperatures, RT was only investigated up to 14 days.

Figure 5 shows that H neat cement at high temperature has a higher compressive strength compared to H cement + 4% MC at high temperature, with a variation up to 45 MPa at 21 days, which shows the great impact in the hydration process due to micro cellulose.

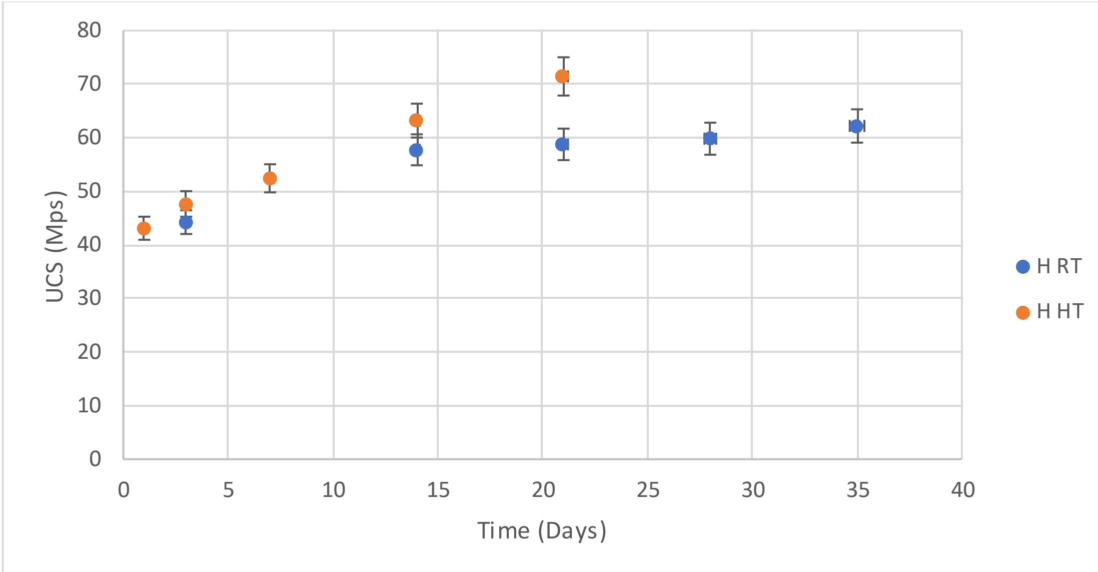

Fig. 2.- Class H t neat cement at high and room temperature

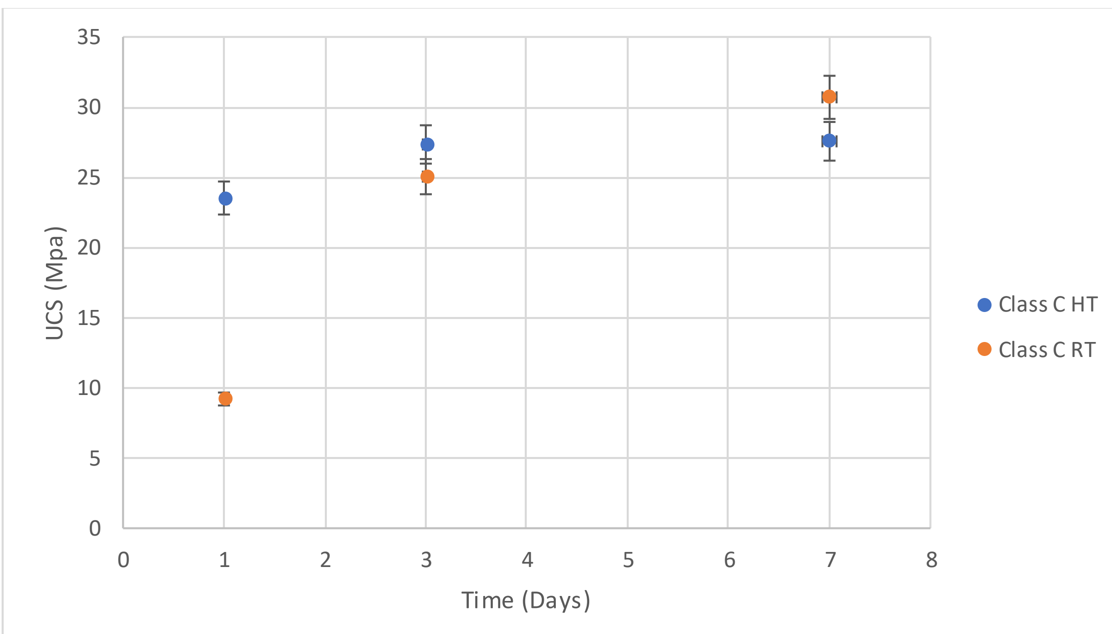

Fig. 3.- Class C neat cement at room and high temperature

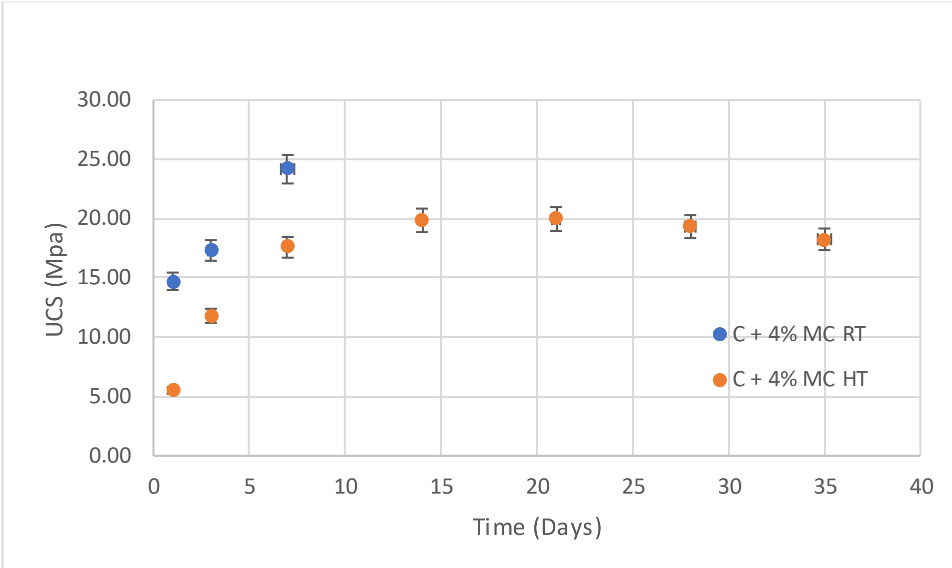

Fig. 4.- Class C cement with MC 4% at room vs high temperature

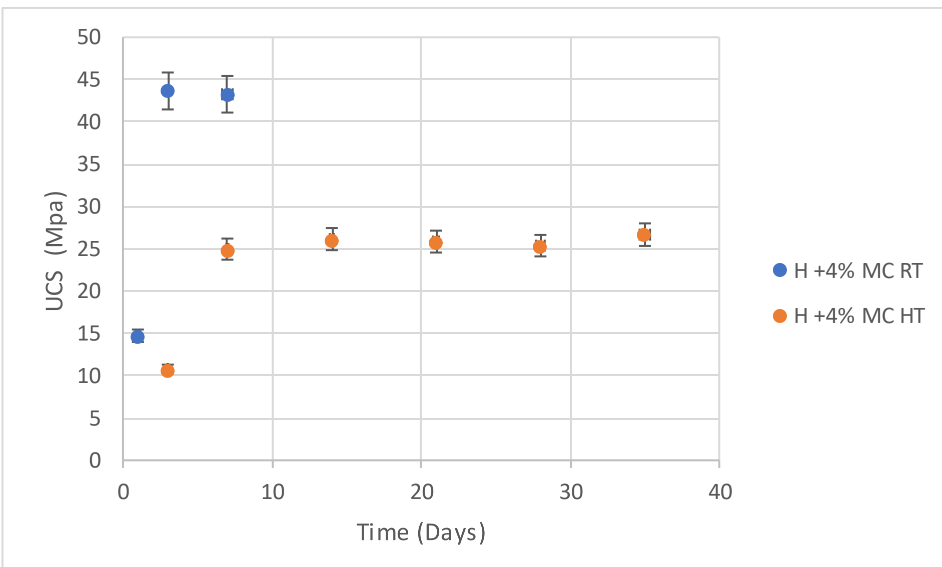

Fig. 5.- Class H cement with 4% MC at room vs. high temperature

4. DISCUSSIONS

In what follow we will compare the neat cement properties with the resulted properties after adding 4% of MC to the cement. Overall, the addition of MC to the cement leads to a reduced UCS at room temperature and at high temperatures. Moreover, although not measured for this paper we have noticed that cement behavior was more plastic compared to neat cement.

Figure 6 shows the comparison of class C neat at room temperature versus class C cement + 4% MC at room temperature, demonstrating the direct effect of MC on the cement hydration process.

Fig 7 shows a comparison between class C cement neat at HT vs C + 4% MC at HT, showing a considerable change in the UCS for each defined time of around 30%.

Similarly figure 8 and 9 shows the comparison between class H neat cement and the UCS for class H with 4% MC added.

Fig. 10 shows that the current selection of 4% of MC was selected when compared to 10% content of MC, showing that the effect of such amount of MC will affect the hydration process considerably, directly decreasing the UCS of the samples, whereas 4% MC showed a better compressive strength with less amount of MC. For example, after 24 hours (1 Day) of curing, samples with 10% MC could not be tested (very soft).

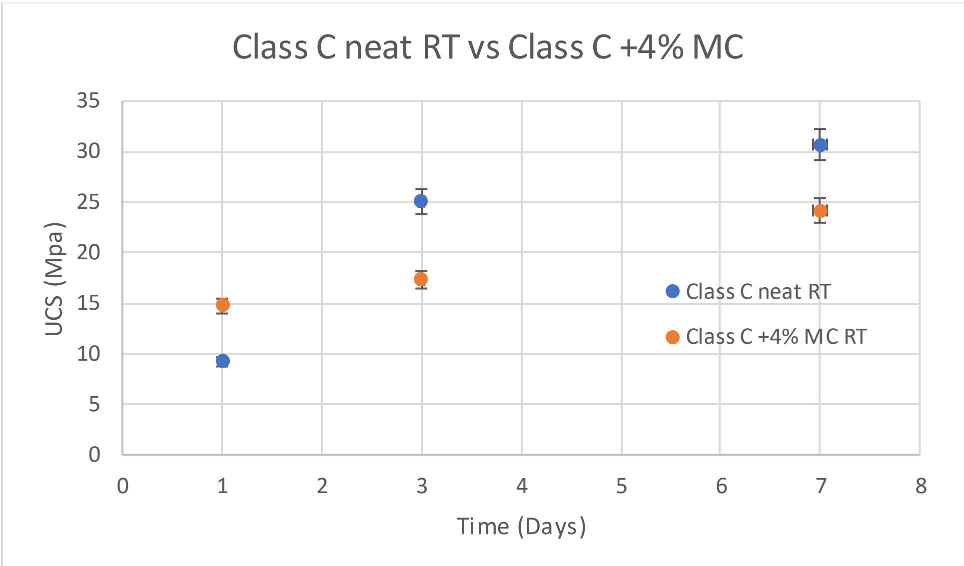

Fig. 6. Class C neat cement vs C with 4% MC at room temperature

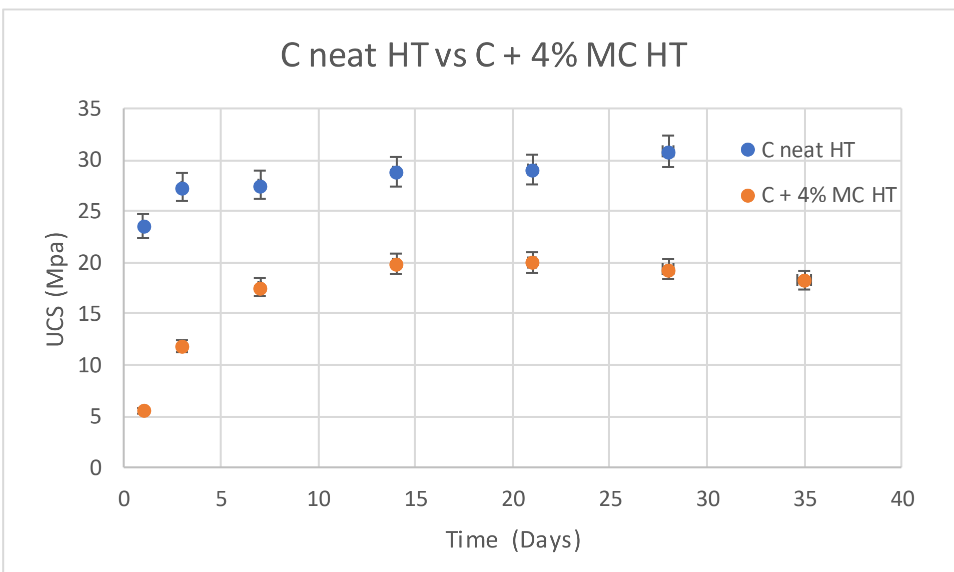

Fig. 7. Class C cement neat HT vs C + 4% MC at HT

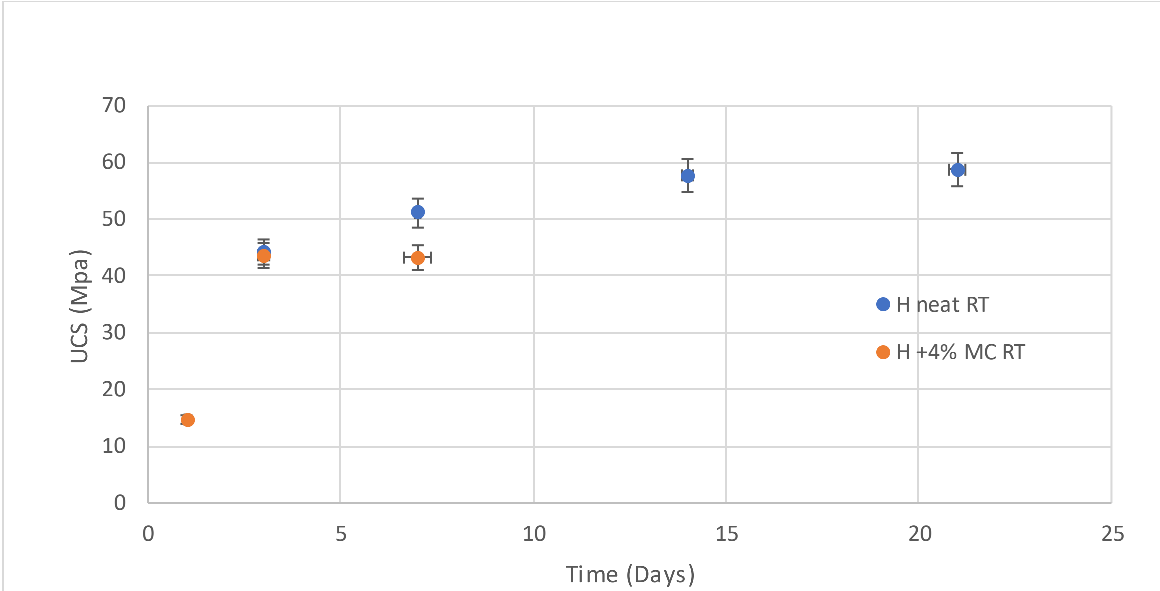

Fig. 8. Class H cement neat HT vs H + 4% MC at RT

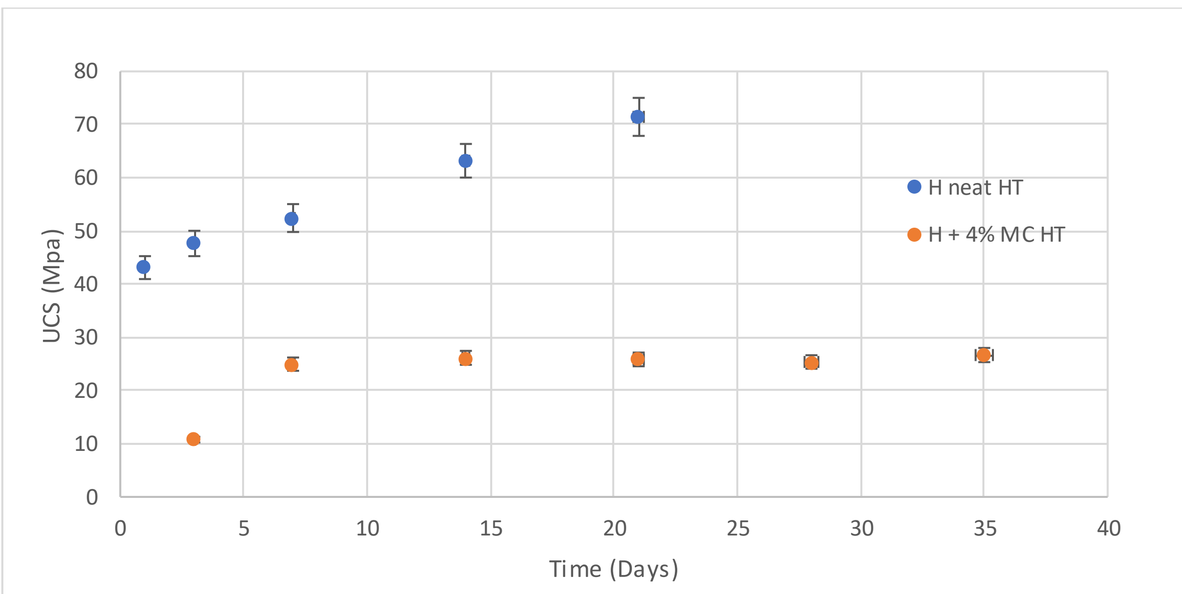

Fig. 9. Class C cement neat HT vs C + 4% MC at HT

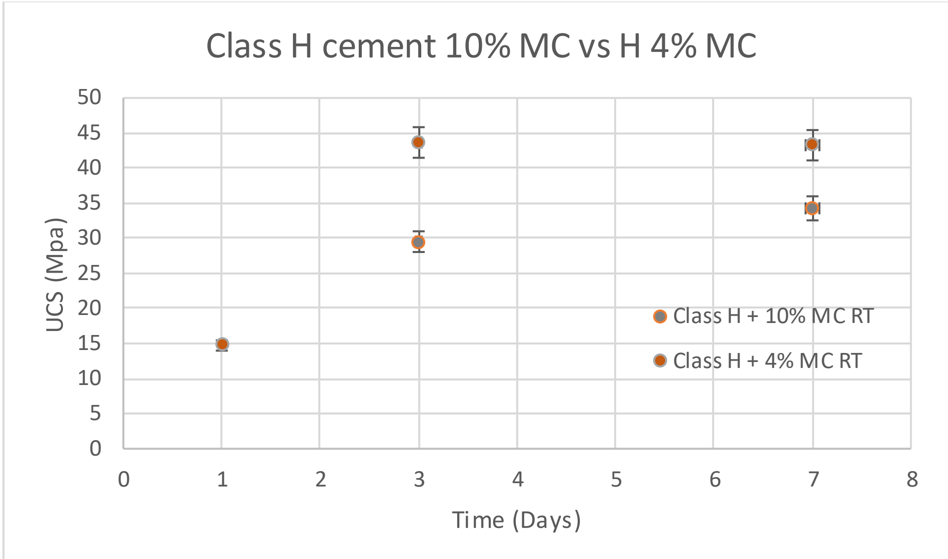

Fig. 10. Class H cement 10% MC RT vs class H cement +4% MC RT

As a comparison Table 2 shows the averaged UCS values for the 4% MC samples both at room temperature and elevated temperature. While neat class H and class C are showing an increased UCS when exposed to elevated temperature all our 4%MC samples have clearly shown that the addition of MC will reduce the elevated temperature UCS values when compared with same slurry composition at room temperature, lower than those at room temperature and obviously lower than the neat cement. The reason for this behavior could be attributed to the hydration process of the MC itself which may absorb additional water from the cement hydration process as well as the swelling behavior of the slurry. This observation is also in line with Hoyos et al (2013) who mentioned that their experiments lead to the conclusion that MC will delay the hydration process especially the hydration rate because of its own -OH products. For room temperature testing we have noticed a similar trend when we added bentonite the cement mix.

Table 2: Summary of the UCS experimental data (average values)

Type	UCS (RT) Class H + 4% MC	UCS (HT) Class H + 4% MC	UCS (RT) Class C + 4% MC	UCS (HT) Class C + 4% MC	UCS ratio to Neat H cement (RT)	UCS ratio to Neat H cement (HT)	UCS ratio to Neat C cement (RT)	UCS ratio to Neat C cement (HT)
1 Day	14.72	N/A	14.72	5.51	N/A	N/A	1.59	0.23
3 Days	43.65	10.76	17.32	11.82	0.98	.56	.69	0.43
7 Days	43.26	24.96	24.17	17.59	0.75	.51	.78	0.64
14 Days	43.11	26.14	N/A	19.85	0.73	N/A	N/A	0.68

Although our current long-term data covers only 35 days, additional work is needed to define the long-term stability of the cement sheath under elevated temperature conditions. Additional temperature levels could also reveal the applicability of this product for high temperature geothermal applications. The drop in UCS for MC cements is high at elevated temperature for the short term, indicating a retarding effect of the MC but this seems to be smaller for long term data. This UCS drop, may seem irrelevant when compared to the alternative cements, but it is compensated by the fact that curing fluid loss will allow the well to be fully cemented.

Future work will involve testing at various temperatures and changing the water ratio available for both cement and MC. These experiments are at the initial phase of qualifying the MC as cement additive, but the results are promising, and thus long-term investigations are also currently under investigation.

5. CONCLUSIONS

This paper shows some preliminary tests on the use of Micronized Cellulose as cement additive with application to geothermal wells.

MC is currently used successfully in curing fluid loss situations in geothermal drilling, and the addition to cement could enhance the ability to cement geothermal wells with some fluid loss issues. Based on the experience in the area of the fluid loos, we decided to use 4% MC BWOC to prepare the slurry.

The data have shown as expected a decrease of the UCS of the cement when compared with neat cement at both room temperature and elevated (75°C) temperature.

Our tests have shown that the high temperature UCS is lower than the UCS at room temperature for both class C and class H slurries, which is induced by the hydration rate reduction induced by the MC at elevated temperatures.

REFERENCES

Arbad, N., Rincon, F., Teodoriu, C. and Amani, M., 2021. Experimental investigation of deterioration in mechanical properties of oil-based mud (OBM) contaminated API cement slurries & correlations for ultrasonic cement analysis. *Journal of Petroleum Science and Engineering*, 205, p.108909.

Rachimillah, Allam Putra, Azwar, Cinto , Johri, Ambuj , Osman, Ahmed , and Eric Tanoto. "Obtaining Zonal Isolation in Geothermal Wells." Paper presented at the IADC/SPE Asia Pacific Drilling Technology Conference, Virtual, June 2021. doi: https://doi-org.ezproxy.lib.ou.edu/10.2118/201068-MS

Buelichen, D.. , and J.. Plank. "Formation Of Colloidal Polymer Associates From Hydroxyethyl Cellulose (HEC) And Their Role To Achieve Fluid Loss Control In Oil Well Cement." Paper presented at the SPE International Symposium on Oilfield Chemistry, The Woodlands, Texas, USA, April 2011. doi: https://doi-org.ezproxy.lib.ou.edu/10.2118/141182-MS

Bosma, M., Ravi, K., van Driel, W. and Schreppers, G., 1999. Design Approach to Sealant Selection for the Life of the Well. *SPE Annual Technical Conference and Exhibition*,.

Brandl, Andreas , Bray, Windal Scott, and Christina Magelky. "Improving Well Cementing Quality with an Environmentally Preferred Multifunctional Polymer." Paper presented at the SPE Europec/EAGE Annual Conference, Copenhagen, Denmark, June 2012. doi: https://doi-org.ezproxy.lib.ou.edu/10.2118/154498-MS

Desbrieres, J. 1993/1. Cement cake properties in static filtration: Influence of polymeric additives on cement filter cake permeability. Cem Concr Res 23: 347.

Desbrieres, J. 1993/2. Cement cake properties in static filtration: On the role of fluid loss control additives on the cake porosity, Cem Concr Res 23: 1431.

Fink, J.K. 2003. Cement Additives. In Oil Field Chemicals, ed. J.K. Fink, Chap. 10, 125. Burlington, MA: Gulf Professional Publishing.

Hoyos, C.G., Cristia, E., Vázquez, A., 2013, Effect of cellulose microcrystalline particles on properties of cement-based composites, Materials & Design, Volume 51, 2013, Pages 810-818, ISSN 0261-3069

Ichim, A., K Saleh, F., Teodoriu, C. and Sondergeld, C., 2019. The Need of NMR Cement Properties Characterization and Lesson Learned. *Day 2 Wed, April 10, 2019*,.

Ichim, A.C., Teodoriu, C., Falcone, G. 2017. The influence of remedial cementing on thermal well design with applications to wellbore integrity, 42nd Stanford Geothermal Workshop, Stanford, California, USA, February 13–14.

Nelson, E.B. ed. 2006. Well Cementing; Sugar Land, TX: Schlumberger Dowell.

Saleh, F. K., Rivera, R.. , Salehi, S.. , Teodoriu, C.. , and A.. Ghalambor. "How Does Mixing Water Quality Affect Cement Properties." Paper presented at the SPE International Conference and Exhibition on Formation Damage Control, Lafayette, Louisiana, USA, February 2018. doi: https://doi-org.ezproxy.lib.ou.edu/10.2118/189505-MS

Teodoriu, C., Okolieocha, P.A. 2014. A Study of Cementing Techniques for Gas Hydrate Exploitation, DGMK/ÖGEW Spring Meeting (Frühjahrstagung), Celle, Germany, April 24-25.

Teodoriu, C., Asambe, P. 2014. A Study of Salt Content Effect on Class G Cement Properties with Application to Gas Hydrate Exploitation, DGMK/ÖGEW Spring Meeting (Frühjahrstagung), Celle, Germany, April 24-25.

API, RP. "10B, Recommended Practice for Testing Well Cements, 22nd." Washington, DC: API 2.BS EN

API Spec 10, "Specification for Materials and Testing for Well Cements" 22nd Edition, API, (Jan 1, 1995)