

**EFFECT OF CO<sub>2</sub> REACH ENVIRONMENT ON THE PETROPHYSICAL PROPERTIES OF ARTIFICIALLY CONSOLIDATED CORE SAMPLES**

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**Abstract:** In order to develop, maintain and deplete reservoirs economically around the globe, various measurements are needed with a high demand on natural core samples. The next stage in the life of every reservoir is a secondary or tertiary method to enhance productivity. However, to tailor the available methods and technologies to the reservoir, several screening processes, feasibility studies and pilot experiments are needed. As an aid to these, like a sensitivity analysis, continuous measurements are set up to study fluid flow, chemical reactions, additional recovery and much more, but for all of these, core samples are needed. The lack and high value of natural core samples yield that the demand cannot be satisfied from this source alone. The aim of the study was to create an artificially consolidated stone core sample, a model material, which can be suitable for being the subject of these experiments, with additional benefits in mass production and reservoir parameter-based quality control. In this article the authors wish to present partial results of a big study, this time with comparing the porosity, permeability, connate water and capillary pressure parameters of the core samples used with different after-cure techniques. The process of compaction was the same, but the overburden pressures and the effect of CO<sub>2</sub> rich curing were examined. For this, part of the samples was prone to high CO<sub>2</sub> environment for different timespans during the after treatment of the samples. The petrophysical parameters were then measured on all of the groups, including a control group and the CO<sub>2</sub> affected cores. The focus was on porosity, permeability, connate water saturation/wettability and capillary pressure measurements and the common features and differences in the yielded pore space's structure are summarized in this article.

## 1 Introduction

Natural rock core samples are pieces of rock mass taken from the reservoir in the wellbore with special core drilling devices. The cores are moved to the surface and the reason for sampling is to examine and identify the reservoir rock, its quality, and to try to get as much information as possible from the structure and characteristics of the porespace, along with the possibility of flow studies. These unique samples carry representative and useful information about the underlying geological formations, thus their inspection is crucial. One part of the conducted measurements is aimed to define the petrophysical parameters of the reservoir. In order to establish these routine measurements, plugs are drilled from the whole cores, and to get these, the completeness of the whole core section is needed with unharmed surface and uncracked interior structure. The common problems arising are the lack of these whole core samples, the insufficient quality of those existing, or often the interval cored is not the representative section of the reservoir. The quantity of the core samples is also insufficient. In practice, the main cause of these problems, is cracked and incomplete whole

core sections. As majority of the SCAL measurements are destroying the investigated samples during the measurement in terms of reproducibility, the number of possible conducted measurements is strictly finite. As the life of a reservoir proceeds, the use of secondary and tertiary depletion methods becomes inevitable. In order to tailor and screen these methods, and after selection, in order to monitor the effect of chemicals and materials used, a very high number of core samples would be needed, and their homogeneity is also a desired characteristic, increasing the overall demand much higher than the possible supply. As an answer to the challenges, the petroleum industry has developed and produced artificially consolidated core samples, thus there are multiple various methods to generate suitable cores.

Though having limited application, the generation of artificial core samples is cheaper and faster, and these cores often have even some benefits against natural core samples, in terms of uniform petrophysical parameters which can help easy monitoring of flooding chemicals. The way to the uniformity of the production is also a challenge and to achieve this the constant monitoring of the yielded

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petrophysical parameters is needed throughout the development stage. As the sample size for RCAL and SCAL measurements is often given, that is another advantage in case of artificial consolidation that the size of the generated sample can be very easily controlled despite the fact that natural core samples are seldom complete, and often the addition of some space filling material is needed. Furthermore, the fact of artificial production opens a way to develop new measurement and control devices, because a much wider variety of sample sizes are now available. The petrophysical characteristics are now a function of a production process, being not prone to reservoir heterogeneity, but in order to achieve this, several test measurements needed to find out the correct generation process to the current demand. Evaluation of the routine core analysis results is a must and the only way to tailor the

process well enough to be able to substitute the natural samples with artificial ones. As a step further, comparison also should be made between those too, and the artificial material should resemble the original one, thus if needed, the production method is altered in order to get the same petrophysical property ranges.

The aim of this study to introduce the basics of the production and to compare the effects on the same composition but different confining pressure and different after treatment methods of the artificial core samples. The two parameters were the pressure of compaction, and the treatment material, as some part of the cores were prone to vapour only and the others to CO<sub>2</sub> rich environment. The compared measurements are regarding porosity permeability, capillary pressure and relative permeability.

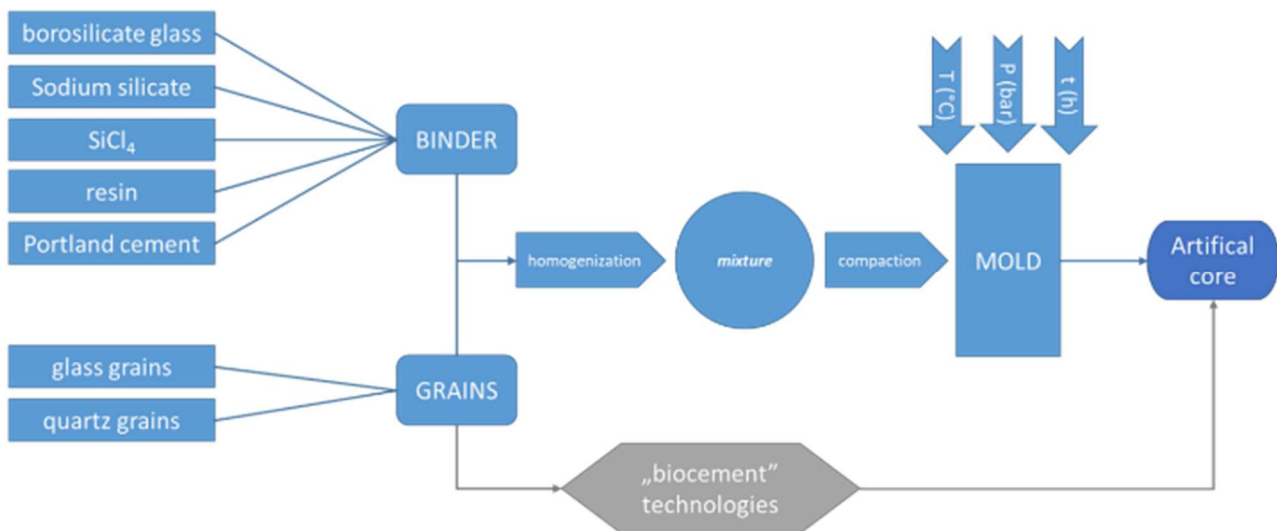


Figure 1 Possible methods for artificial samples

## 2 Main types of artificial core samples based on literature review

The history of the first attempts on creating artificially consolidated rock samples in order to substitute natural ones dates back for almost two decades [1]. As the manufacturing technology improved, new materials and methods were used on order to match the areas of interest. Based on technical literature, it is indeed possible, that using various methods (Figure 1), core samples can be generated, and these can be tuned to have similar petrophysical properties to hydrocarbon reservoirs, being suitable for conducting petroleum industry-based laboratory measurements.

To start the process the primary materials should be the same type and purpose as in the reservoir rocks. The main materials are a matrix and a bond forming material, a cement. To achieve a rock matrix, most of the methods use natural quartz particles [2-10], while some others use artificial glass beads [11], waste glass and mashed rock [12], or waste rock mud [13].

The secondary component is the cementing material, which connects the particles and solidifies the structure. These components can vary based on the different future application areas of the samples. With the application of sodium silicate or any alkaline silicate, the resulting cement material and sample is rigid, thus having quite low mechanical resistance [2,4,6,14,15]. Besides having its own advantages, one must conclude that these materials can easily devitrification [15] in water-based environments, thus rendering their application in permeability measurements insufficient. As an improvement, the aim was the strengthen the bonds by [16], who added kaolinite-based additives to the cementing material. As per rigidity, other materials can be used such as borosilicate glass [3,17] or the strongly toxic silica tetrachloride [18]. Following the same ideas, to strengthen bonds, Portland's cement or other industrial cements can be used as a bonding material, also resulting in a rigid but suitable pore structure [8,19,20-22]. The usage of cement has another advantage in petroleum engineering, which is that the resulting pore structure will be more of a water wet

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system, thus mimicking the reservoir rock structure more efficiently. Still on the rigid side, heat treatment of clay van also yields porous artificial samples [23-25]. When applying epoxy resin as a bonding material in the samples [26-30], the resulting artificial core is more plastic, and mechanically resistant, but, on the other hand, all of these cores exhibit moderate or strongly oil wet behaviour, which renders them inapplicable in measurements, or serious correction is needed in the evaluation process [30]. Their plasticity alone is also a big difference compared to conventional reservoir rocks.

As a third, rather independent method, microbial carbonate emission can also be used to generate artificial porous material [31-33]. First the construction industry applications were studied [31-37], then it was adopted as a trial to generate petroleum industry core samples, too [9,10,38].

If the components are finalised, a recipe is created. Once that part is ready, the next step is some sort of consolidation, solidification etc. As a start a thorough homogenisation is done, and the resulting material is a rock pulp/mud/dollop, which is put into a sample holder. To achieve sufficient density and consistence, several different devices can be used, which are based on the vibration and compaction principles [2,6,12,13]. According to the different methodologies, then this mixture is often put under certain pressure and temperature conditions in order to finalise the bond, structure and any reaction present within the sample. Pressure itself is a good analogue to the generation of sedimentary reservoir rocks, thus is a very important parameter to set up correctly. When a natural core is taken from the wellbore, even the pressure release is the same type compared to the release of the samples from the machine [5].

### 3 Design process, experiment plan

Based on our recent studies, there is possibility to alter the porosity and permeability values of the samples [39] based on the methodology, across a quite wide range (from reservoir engineering point of view). Based on the origin of the matrix material, there are two main sources of artificial core samples. One source is regarded as natural, which is the crushed shells of real whole core remnants, or any residue but from the given reservoir rock. The mining and riverbank sand sources are regarded as non-natural, but yet these are the preferred ones, as the representative recipe can be much more easily generated, and there are numerous cheap sources, which are having the same parameters as the typical Hungarian sedimentary reservoirs. The particles creating the matrix were between 110-150 micrometres in size. As a bonding material, industrial cement based gluing material was used.

In the first part of the experimental study, the matrix-cement ratio and the composition were the same in all of the samples. The parameters varying were the added water (12,1-13,19 g) and the pressure used during the 24-hour

compaction period. The curing was 14 days in both cases at 10 bars and 45 °C (113 °F).

The difference in the second set of experiments was the curing process (Table 1). In the technical literature there is precedent of using the artificial samples to study CO<sub>2</sub> injection EOR methods [40], but the effect of that CO<sub>2</sub> to the resulting pore structure and permeability lacks proper documentation, thus becoming the aim of these measurements. The main parameters of the samples can be found in Table 2. There were 4 groups, part of the groups was prone to CO<sub>2</sub> rich environment, whereas the other parts were prone only to water vapour rich environment.

Table 1 The curing and types of the groups within the samples

2 weeks of CO <sub>2</sub>	Group 1	Sample 1-6
1-1 week CO <sub>2</sub> and vapour	Group 2	Sample 7-12
1-1 week vapour and CO <sub>2</sub>	Group 3	Sample 13-18
2 weeks of vapour	Group 4	Sample 19-24

After the core samples were ready and stable, the next step was the design of the measurements of comparison. As for the natural core samples, the idea was to recreate a typical RCAL routine, starting with porosity and effective and absolute permeability measurements. To identify the wettability and to categorise the resemblance to natural core samples, connate water saturation, capillary pressure curve and relative permeability were also examined on the artificial samples. In order to physically fit into the CoreTest URC-628 rock centrifuge, these samples were intentionally made shorter than the normal plugs, thus already exhibiting on of the benefits of the process (that one can easily choose the output size and form).

Table 2 Composition characteristics of the artificial samples

Sand (g)	178,80
Cement (g)	30,00
Water (g)	12,10
Sand: (Cement + Water) ratio	4,25
Cement-Water ratio	2,48
Sand-Cement ratio	5,96
110 µm - 150 µm	100,0%
Applied pressure (bar)	400
Duration under pressure (hr)	24:00:00
Temperature (°C)	~23
Number of strikes (Marshall)	6x
Curing period (hr)	~336:00

In order to prevent anomalies caused by end effects and to be in accordance with normal lab routine, the other

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groups size was set with removing material from the two ends of the samples. If the ends are not removed, based on previous experience, the structures can be deviant near the end zones, which are affecting the results of the flooding-based experiments.

#### 4 Porosity and permeability measurements

The porosity values of the artificial core samples were measure with Helium porosimeter and are displayed on Figure 2. The permeability values vs. the porosity results are displayed on Figure 3. The results of the measurements are presented in Table 3 The application of different water quantities did not create any major change in permeability, but the trend is that less water during production yields higher permeability. This phenomenon is much stronger in the artificial core samples which were created under high pressure values of 300-400 bars. On the pore-perm plot the distinctiveness of the different pressures can be easily seen. In terms of the created porespace, more than 10 percent decrease can be yielded with increasing the confining pressure from 200 to 400 bars. In these scenarios, the permeability values can also be varied utilising different pressures.

In the described particle size, the minimum permeability values were around 170 mD, while the maximum values scaled at around 1412 mD. According to the porosity measurements the minimum was 20,02% at 400 bars of confining pressure and 32,82% at lower pressures. The previous values are for the water sensitivity analysis.

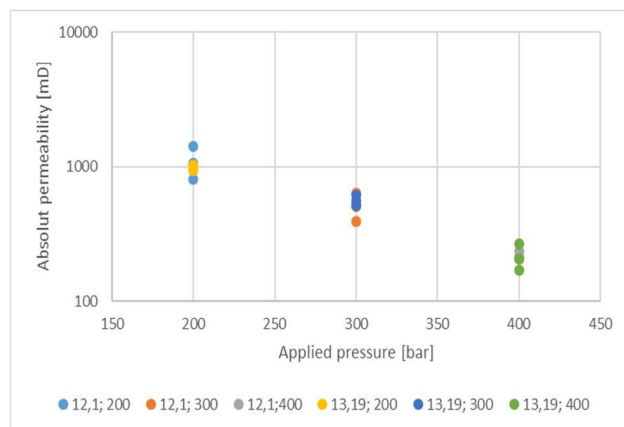


Figure 2 Absolute permeability values vs applied pressure of the 110-150 μm particle size artificial core samples

In the second part of the experiments, the difference was in the curing process. There were 4 groups as mentioned, one with 2 weeks in vapour only, one with 2 weeks of CO<sub>2</sub> only and two groups of 1 week in environment 1 and the other week of curing in environment 2, respectively, in accordance with Table 1. To clarify, within group 1 was the difference in added water only, as a sub-experiment. As the samples were created with a local developed method, the first part was also the determination

of porosity. (All of the results are summarized in Table 3) The average porosity in the group which had 14 days of CO<sub>2</sub> exposure was 26,68% with the deviation of 1,73%. For the group which was cured with CO<sub>2</sub> for 7 days then with vapour for the second 7 days, the average porosity was 25,64% with a deviation of only 0,5%. In this group the reproductivity is the highest, from the 6 samples 3 exhibit differences in porosity only in the magnitude of 0,01-0,02%, and only one of the sets yielded a porosity above 26%. The third group (vapour curing for one week, then CO<sub>2</sub> curing for one week) three samples are the same type in terms of result such as in the previous group, overall porosity decreased a little bit to 25,99%, with the deviation of 1,47%. The last/control group (14 days of curing in vapour only) yielded a decrease in porosity, at an average of 24,44%. From the total of 24 samples, the 3 most dense are all in this group, porosity is below 24% for all of those ones.

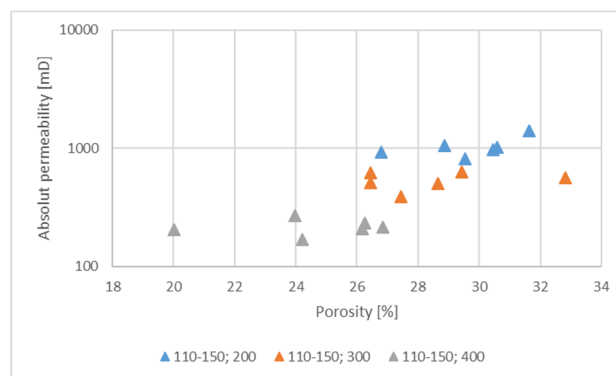


Figure 3 Porosity values vs absolute permeability values of the 110-150 μm particle size artificial core samples

The next parameter to measure and compare was absolute permeability, which was measured with Nitrogen gas. For the two weeks of CO<sub>2</sub> curing group, the values ranged between 15 and 89 mD. One sample is anomalous amongst these six, but the rest exhibit uniform behaviour. For the one-week CO<sub>2</sub> one week vapour curing group, the range of permeability is more or less the same, if trend needs to be stated, it is increasing in terms of permeability by a small amount. For group 3, treated with vapour first then CO<sub>2</sub>, there is a significant increase in absolute permeability, with an average of 221 mD. For the two weeks of CO<sub>2</sub> only group this behaviour was the same, resulting in permeability values with the average of 205 mD.

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Table 3 Results of the porosity and permeability measurements in the groups of samples with different curing

		Number of sample	Porosity (%)	Absolut permeability (mD)	Effective permeability (mD)
Curing type	2 weeks CO <sub>2</sub>	Sample 1	27,41	89	22
		Sample 2	28,36	35	21
		Sample 3	26,60	37	20
		Sample 4	26,44	18	12
		Sample 5	24,95	15	7
		Sample 6	26,30	17	9
	1 week CO <sub>2</sub> + 1 week water vapour	Sample 7	25,11	82	64
		Sample 8	26,14	36	28
		Sample 9	25,72	57	38
		Sample 10	25,71	38	32
		Sample 11	25,73	12	9
		Sample 12	25,42	13	10
	1 week water vapour + 1 week CO <sub>2</sub>	Sample 13	25,65	196	99
		Sample 14	26,67	258	148
		Sample 15	26,25	275	167
		Sample 16	24,52	89	70
		Sample 17	25,97	252	66
		Sample 18	26,86	257	226
	2 weeks water vapour	Sample 19	26,47	231	98
		Sample 20	25,75	188	105
		Sample 21	22,73	162	77
		Sample 22	23,12	190	167
		Sample 23	23,73	217	88
		Sample 24	24,81	239	122

The third measurement in the study was the effective permeability measurement. using synthetical pore water, the composition of that is described in Table 4. The speed of the flood was 50ml/hour, and the key concept was to displace at least ten porespace equivalent water through each and every sample, and then take the average permeability of each measurement. For Group 1 the values are within a narrow range, with an average of 15 mD. For Group 2, despite having roughly 1 percent less porosity, this value was increased, with an average of 30 mD. For Group 3 there was a significant change in terms of an increase, the average effective permeability values were at around 129 mD. As per Group 4 the average was similar, being at around 110 mD, though the porosity values were the lowest in this group.

Table 4 Composition of the artificial pore water

Component	Quantity (g/l)
NaCl	0,5
NaHCO <sub>3</sub>	2,6
CH <sub>3</sub> COONa	1,6
Total	4,7

As a secondary result of the experiments using flooding, it can be conducted that after further evaluation of the expelled fluids, there were no solid content or any residue washed out from the sample, thus rendering the artificial core samples stable enough for the previously stated expectations.

## 5 Capillary pressure and relative permeability measurements

In the last chapter of presenting the results, the authors wish to evaluate the results of the RCAL measurements regarding capillary pressure, relative permeability and connate water saturation. The measurements were conducted in every group of artificial cores with a CoreTest URC-628 rock centrifuge. After screening the characteristics for porosity and permeability, and having good results, the next step is to monitor the artificial cores as a hydrodynamic system, when wettability is present, and when two phases exist together, to find out its applicability in flooding experiments. On the other hand, if any difference is present compared to natural core samples, that still does not make any problem if a correlation is found and the values can be corrected.

During the RCAL measurements it was found that the cores are suitable for substitution, but the nature of the pore structures yielded a challenge to solve through the evaluation process. The problem was that the rock centrifuge gave reliable values on connate water saturation and capillary pressure, but the relative permeability curves were shifted to the right, to higher saturations. This can be caused by spontaneous imbibition and microfractures in the samples. The idea is that if microfractures are present, the water is expelled from the samples at much lower rotational speeds, thus much lower pressure, and the high-speed camera can detect the change, but arranges it to the wrong saturation, as the software thinks the samples are still almost fully saturated in the first stage of rotation. The mobile saturation range, however can be extracted from the capillary pressure curves, and using a normalisation, the relative permeability curves can be transformed to this range.

In terms of results, the 1 week one environment, 1-week other environment cores (Group 2,3) exhibited same behaviour, and the values scaled in between the 2 weeks of CO<sub>2</sub> and 2 weeks of pure vapour cured groups (Group 1-4). Thus, only the two "extremums" are shown in this article, groups 1 and 4, through one-one representative examples. In all of the groups, one common fact is, that all of the artificial cores generated exhibit water wet behaviour, which is desired, if the aim is to mimic cores from sandstone reservoirs. The magnitude is a little bit higher than in average reservoir conditions, but if this fact is known during the evaluation of the planned measurements using these samples, no problem is generated. One must state, that compared to epoxy resin artificial samples, these scenarios are much more

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favourable, as correlation is easy, compared to the unreliability of the oil wet artificial cores.

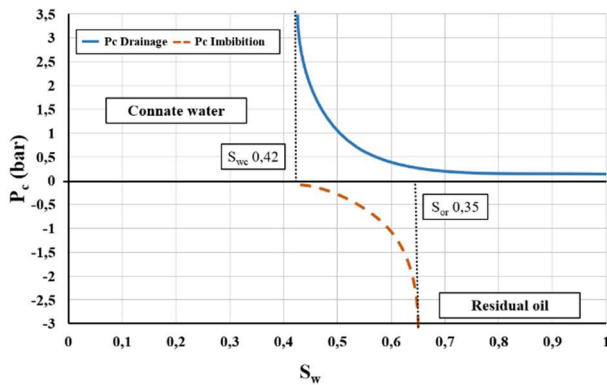


Figure 4 Capillary pressure curves of Sample 4 taken from the CO<sub>2</sub> cured group

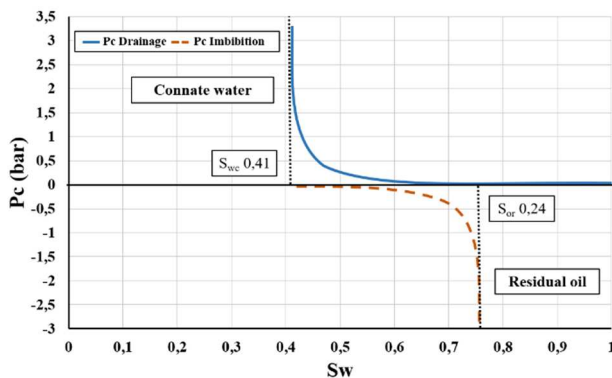


Figure 5 Capillary Pressure curves of Sample-20 taken from the vapour cured group

On Figures 4-5, the Capillary pressure curves are presented for Group 1 and 4. The connate water saturation of the samples ranged between 30-45%, being rational for sandstone rock samples, and being in the expected range, as even the bonding material type predicts water wet behaviour. The residual oil saturations ranged between 25-35%, being also in an expected and suitable range.

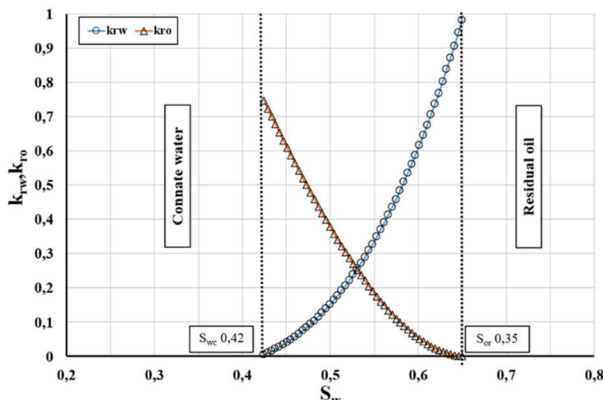


Figure 6 Relative permeability curves of Sample-4 taken from the CO<sub>2</sub> cured group

For the sample cured with CO<sub>2</sub> for two weeks, the mobile saturation range is between 42 and 65 percent. The pure vapour cured sample exhibit same behaviour setting the mobile saturation between 41 and 76 percent.

The relative permeability curves are also presented for Group 1 and 4. These were the curves which needed transformation based on the mobile saturation ranges shown by the capillary pressure curves. Figures 6-7 display the transformed relative permeability curves for the groups. The shape and form of the curves is realistic, having great resemblance to the natural sandstone core materials. Equilibrium saturation and endpoint saturation values are normal.

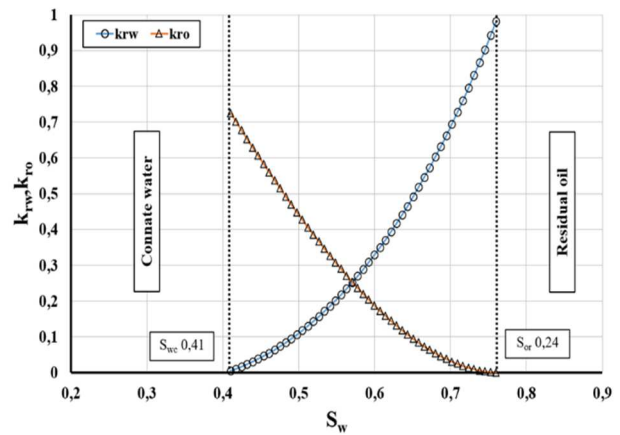


Figure 7 Relative permeability curves of Sample-20 taken from the vapour cured group

## 6 Conclusions

The aim of the study was to present a possible production method for artificially generated sandstone rock core samples, and to study the effects of different curing techniques applied to the yielded samples. Four groups were declared among the samples. Group 1 had curing in CO<sub>2</sub> rich environment only, for two weeks. Group 2 had one week in CO<sub>2</sub> and one week in water vapour. Group 3 had one week in water vapour and then one week in CO<sub>2</sub> rich environment. Group 4 had two weeks of pure water vapour curing, and in this group two types of samples were made, having difference in the added water to the cement material.

The differences in porosity and permeabilities were evaluated. According to added water, the smaller amount yielded higher permeability values. Based on curing, the vapour cured samples exhibited higher permeability, but relatively lower porosity values.

As for special core analysis, every group exhibited appropriate behaviour, in terms of connate water saturation, capillary pressure and relative permeability, which means that these samples can be suitable for substituting real natural core samples in high sample demand monitoring type petroleum industry measurements.

Plans for the future include geomechanically measurements, and further experimentation on additives at

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the bonding stage. The cores were stated to be suitable for substituting real cores in EOR measurements, and are now used in one such project, but this was not introduced in this article due to corporate secrecy. Another company is already having request for production of these types of cores to utilise them at their RCAL laboratory.

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**EFFECT OF CO<sub>2</sub> REACH ENVIRONMENT ON THE PETROPHYSICAL PROPERTIES OF ARTIFICIALLY CONSOLIDATED CORE SAMPLES**

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