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Oil and Gas Research Institute

Hydrocarbon resources (crude oil and gas) are the main source of world energy, and as the international demand increases, the technical challenges increase to meet that demand. Hydrocarbon production optimization at minimum cost and the need to serve the national petroleum industry has been the driving force behind the establishment of the Oil and Gas Research Institute (OGRI) at King Abdulaziz City for Science and Technology (KACST). OGRI is a governmental research and development entity. Its applied research activities concentrate on the upstream sector of the petroleum industry. Fields of interest cover most of the petroleum science and engineering aspects through four main divisions:

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Keynote at 9th Annual Gulf Petrochemicals & Chemicals Association Forum

By Khalid A. Al-Falih, President & CEO of Saudi Aramco

DUBAI, November 24, 2014

'Your Excellencies, distinguished guests, ladies and gentlemen: good morning. It's a pleasure to be here four years after my last address to GPCA. At that time I called this a "golden decade" for our region, and called for both greater integration and diversification, and more intensive efforts in research and development. Since then, we had a number of achievements in which you can take genuine pride.

That said, overall regional development has focused on the manufacturing of commodities for export by leveraging our strong feedstock advantage, our economies of scale and extensive industrial infrastructure. We have tended to grow horizontally rather than through vertical integration, and while primary petrochemical capacity has grown admirably,

the strengthening of functional capabilities has tended to lag. To date, our advantages have carried us through – but the existing model will not realize our full potential because the global industry landscape is changing rapidly and creating stronger competition around the world.

A Changing Landscape

On the back of growing volumes of unconventional oil and gas, North American chemicals and plastics production will virtually double over the next decade, with a substantial increase in exports to markets that we have, over the years, assumed were ours for the taking. The European petrochemical industry is closing less efficient plants, integrating assets into cross-regional networks, and altering its product portfolio. Things are changing in Asia as well, as Japan rationalizes its petrochemical sector, while China undergoes a relative

> "Collectively, we need to keep in mind that demand for chemicals is growing at a faster rate than nominal economic growth (GDP), and that not all that new demand can be met with gas light feedstocks."

economic slowdown, places an intensified emphasis on the environment, and struggles with existing overcapacity while also pursuing opportunities for coal to chemicals.

Closer to home, we see constrained gas-based feedstocks, while recent crude oil price volatility underscores yet again the value of vertical integration and greater diversification which provide greater resilience and adaptability. While our efforts are commendable and are already proving their worth, I want to outline four major opportunities where we can do much, much more.

Four Steps Forward

First, supplies of ethane are becoming tighter in our region – but supplies of alternative feedstocks such as naphtha and other liquids are plentiful. I believe that we shouldn't think of these feedstocks as mutually exclusive choices but rather view them as a mixed pool of feedstock that can be used to leverage each other. Liquids are more versatile than pure ethane and when used in mixed feed crackers, offer a broader product slate, including opportunities to produce specialty chemicals, which in turn can help spawn new industries and consequently many new jobs. Collectively, we need to keep in mind that demand for chemicals is growing at a faster rate than nominal economic growth (GDP), and that not all that new demand can be met with gas

light feedstocks. Longer term, I foresee the creation of new, groundbreaking technologies to enhance the competitive position of liquids, such as the direct conversion of oil to chemicals.

This brings me to the second opportunity: enhancing our region's existing chemicals facilities, since applying the mixed feedstock cracker strategy and other enhancements only to future projects will certainly limit their full impact and potential. Considering the massive scale of the region's petrochemical asset base built in the 1970s and 80s, it would generate enormous additional value if we pursued opportunities to restructure and upgrade these legacy assets, some of which are fully depreciated and offer limited added value to local economic development and profitability of companies in the future. This retrofit would include changes to the feedstock mix, deployment of more energy efficient technologies and the addition of high value specialty products. But to succeed in specialties, we will need to leapfrog in knowledge intensity and accelerate our innovation engines.

The third opportunity is to multiply the number of industry participants and jobs. Many of our past efforts have focused on large-scale commodity petrochemical projects, which offered the benefits of scale economies, allowed meaningful penetration into export markets

and gained for us a prominent position on the global industrial landscape. But there are tremendous advantages in combining the scale of mega-facilities with the high value addition and job creation potential of small and medium-sized enterprises, including the strengthening of an entrepreneurial ecosystem here in the region. So, rather than being content with just a handful of major players, we ought to have thousands of small and mid-size chemicals companies in the Gulf, just like we find in the US, Europe, Japan or South Korea. For example, in Europe alone the chemical industry directly employs more than a million people in about 30,000 companies, more than 95 percent of which are classified as SMEs.

All of this will be more easily accomplished if we can grab our fourth opportunity: greater regional integration. As I argued back in 2010, we should think of the entire Gulf as a unified whole rather than as a collection of individual chemical industries walled off from one another. The picture I have in mind of the future GCC is a booming, cross-connected region, buzzing with chemicals-related activity, and while we need to maintain a healthy dose of competition, we should also creatively collaborate at the regional level to create potential synergy and the essential qualitative edge in terms of innovation, education and technical excellence.

What We're Doing

At Saudi Aramco, our actions reflect our belief in the continued importance of the downstream, and we want to be as strong a downstream player as we are in the upstream. Over the coming decades we intend to become a top global refiner and a leading chemicals enterprise. This will allow us to capture commercial opportunities and profitable growth while diversifying our business portfolio, and over the next decade we will invest tens of billions of dollars around the world and all along the value chain.

We're already engaged in integrated manufacturing complexes through a series of joint ventures at home and abroad, all of which utilize liquid feedstocks and produce a diverse set of value added derivatives. Our industrial value parks at PetroRabigh, Jazan Economic City, SADARA in Jubail (our joint venture with Dow Chemical) and SATORP, our JV with Total, demonstrate our commitment to integrated downstream conversion, value addition, and the use of petrochemicals to spur new industries, and create investment opportunities for our customers. Saudi Aramco is also providing start-up SMEs, including

companies devoted to cutting edge research and innovative products, with both capital and corporate sponsorship, through our entrepreneurship center (Wa'ed) and Saudi Aramco Energy Ventures (SAEV) initiatives. And we're expanding our R&D spending and research manpower, while employing an Open Network model to make the most of in-house R&D centers, satellite research centers in the Kingdom and around the world, and strategic alliances with leading universities and research institutions, like King Abdullah University of Science and Technology (KAUST) and the Dhahran Techno Valley.

Together, these investments, initiatives and programs are transforming Saudi Aramco. But that transformation is taking place alongside a major demographic shift in the region, and we're also embracing that shift at Saudi Aramco and the tremendous opportunities it presents.

The Youth Dimension

As the region experiences a "youth bulge" of unprecedented proportions, an entirely new generation of men and women is sweeping through Saudi Aramco as well. As a result, by the end of the decade some 60 percent of our work force will be 35 years or younger. So this morning I would like to share with you some thoughts on this incredible transformation, which many other firms across the Gulf are either undergoing or will eventually experience.

First of all, our approach begins with the realization that these young people are a different breed than previous generations of Aramcons. Most of my colleagues and I started our careers content with a narrowly defined job working a shift at a plant or in a cubicle at the office. But today young people have what I call a "positive rebelliousness" hardwired into their worldview: they think less along the lines of organizational hierarchies and more in entrepreneurial terms, and see creative disruption as a way to produce something new and better. They have an appetite for risk; they thrive on rapid change and constant mobility; and many are simultaneously more skeptical and more hopeful than my generation was at their age.

And while they are driven to better themselves, they are equally convinced they can do that while making a difference for their community and their company. They think laterally and multi-dimensionally, and want to tackle challenges and explore the possibilities for improvement in whatever they do. It's also a generation that grew up with a baby bottle in one hand and an electronic device in the other! They are therefore a

hyper-connected generation whose lives are lived on social media and not just in their own social circles, so for many of them, a cubicle in an office isn't just small or impersonal, it's irrelevant—somewhere to recharge your smart phone or iPad rather than a place to start your career.

But that different worldview is one reason we view their potential contributions to our company as our most prized future opportunity, and why we take connecting with this new generation and directing their energies so seriously. We at Saudi Aramco enjoy a proud heritage, but we also recognize that our corporate culture must evolve with the times, and we have a number of initiatives to reinvigorate our corporate systems and streamline our processes while engaging this generation of young talent.

First of all we invest heavily in skill development through our college and advanced degree programs; apprenticeships; the three-year, on-the-job Professional Development Program; and other development and onboarding initiatives. Our veterans have embraced the mentorship of this new generation with enthusiasm, because they see their enormous potential. Our younger employees on the other hand are stretching themselves and at times their organizations as they want to set higher benchmarks and goals than their predecessors and supervisors.

We also want our newest Aramcons to be fully engaged in that evolving corporate ecosystem we are developing, so that even as we get these young people ready for the company, we are also getting the company ready for them. We are allowing their perspectives to shape Saudi Aramco, and to make our company better, stronger and more agile. For example, we want to leverage their entrepreneurial spirit, encourage them to employ their creativity towards organizational effectiveness, and utilize their love of technology to better connect across organizational and functional lines transcending professional disciplines. In my view, because they have grown up multi-tasking and want their organizations to do the same, they are best qualified to make a large corporation be just as agile and entrepreneurial as a start-up.

To maximize these benefits, we have created systems and structures to channel the inputs and harness the incredible energy of these young people. Individual company organizations have their own, specially tailored programs, but at a corporate level the best example is our Youth Leadership Advisory Board

(YLAB). This is a group of 16 young employees who during their 18-month term conduct studies on topics of significant importance both to the company and to young people; they provide advice to management, ideas and insights to senior management; and engage other young employees in the changes taking place throughout Saudi Aramco. We are now on our third YLAB cycle, and it has become a self-sustaining program that is both highly competitive and highly respected within the company. "Mini-YLABs" are taking root at various levels and locations within company organizations and professional societies—in fact, we've seen young people create their own self-directed "youth-only" groups to tackle issues of importance to them.

The participants gain a good deal of insight, experience, and maturity during their time in YLAB, but we as leaders also gain even greater insights from listening to these young men and women throughout Saudi Aramco. Likewise, I see youth as one of the Gulf chemical industry's biggest sources of competitive advantage, and many of the traits we see in these young people – a flair for entrepreneurship, a passion for technology, a desire to connect and collaborate, and a daring drive for growth and development – are the characteristics we need in our regional business.

Conclusion

That's because, just like in our incredibly talented youth, I see substantial untapped potential in the Gulf chemicals space. We have multiple advantages such as a large and diversified pool of gas and liquids feedstocks; world-scale assets; modern infrastructure; a globally competitive workforce, an ideal geographic location for exports to both East and West; and supportive government policies. This will be further bolstered by a growing degree of both vertical and regional integration as well as enormous opportunities to advance in innovation. As the global leader in petroleum, our region should be among the foremost players in petrochemicals and specialty chemicals as well.

Four years on from my last appearance at GPCA, I remain convinced that this is a pivotal decade—indeed a golden decade – for the Gulf and its chemicals sector, and that we are in the midst of a once-in-a-generation opportunity for a true transformation. So let us seize that opportunity and make the most of our enviable competitive position of considerable resources and our wealth of young talent. Thank you for your attention this morning, and let me wish each of you a rewarding and informative conference. ◊

Shale Gas Characterization and Property Determination by Digital Rock Physics

By Anas M. Al-Marzouq, Dr. Tariq M. Al-Ghamdi, Safouh Koronfol, Dr. Moustafa R. Dernaika and Dr. Joel D. Walls.

Abstract

Unconventional shale reservoirs differ largely from conventional sandstone and carbonate reservoirs in their origin, geologic evolution and current occurrence. Shale comprises a wide variety of rocks that are composed of extremely fine-grained particles with very small porosity values on the order of a few porosity units and very low permeability values in the nanodarcy (nD) range. Shale formations are very complex at the core scale: they exhibit large vertical variations in lithology and total organic carbon (TOC) at a scale so small that it renders core characterization and sweet spot detection very challenging. Shale formations are also very complex at the nano-scale level, where pores having different porosity types are detected within the kerogen volume. These complexities have led to further research and the development of an advanced application of high resolution X-ray computed tomography (XCT) scanning on full-diameter core sections to characterize shale mineralogy, porosity and rock facies so that accurate evaluation of the sweet spot locations can be made for further detailed petrophysical and petrographic studies.

In this work, argillaceous shale gas cores were imaged using high resolution dual-energy XCT scanning. This imaging technique produces continuous whole core scans at 0.5 mm spacing and derives accurate bulk density (BD) and effective atomic number (Z_{eff}) logs along the core intervals, logs that are crucial in determining lithology, porosity and rock facies. Additionally, integrated X-ray diffraction (XRD) data and energy dispersive spectroscopy (EDS) analysis results were acquired to confirm the mineral framework composition of the core. Smaller core plugs and subsamples representing the main variations in the core then were extracted for much higher resolution XCT scanning and scanning electron microscopy (SEM) analysis. Porosity, mainly found in organic matter, was determined from 2D and 3D SEM images by the image segmentation process. Horizontal fluid flow was only possible through the organic matter and the simulations of 3D focused ion beam (FIB)-SEM volumes by solving the Stokes equation using the Lattice Boltzmann method (LBM).

A clear trend was observed between porosity and permeability, correlating with identified facies in the core. Silica-rich facies gave higher porosity-permeability relationship characteristics compared to the clay-rich facies. This is mainly caused by the pressure compaction effect on the soft clay-rich samples. High percentages of organic matter were not found to be a good indication for high porosity or permeability in the clay-rich shale samples, while the depositional facies was found to have a great effect on the pore types, rock fabric and reservoir properties. The results and interpretations in this study provide further insights and enhance our understanding of the heterogeneity of the organic-rich shale reservoir rock.

Introduction

Hydrocarbon recovery factors from unconventional organic-rich shale have always been at the lower end of historic figures from conventional reservoirs[1]. The reason for this is the ultra-low permeability of the rock, which requires massive hydraulic fracturing to enhance connectivity, and therefore, permeability for the flow.

The fracturing technique should have the potential to lead to economical hydrocarbon production by creating a complex fracture network that is made up of many interconnected fractures in close proximity to one another. To choose the right fracturing technique, one must have a good understanding of the reservoir characteristics at multiple scales. The evaluation of shale, however, is complicated by the structurally heterogeneous nature of the fine-grained strata and their intricate pore networks, which are interdependent on many geologic factors, including total organic carbon (TOC) content, mineralogy, maturity and grain size.

In this work, full-diameter whole core samples from a shale gas reservoir in the Middle East were characterized at the core and pore scale levels. The core samples were analyzed using the dual-energy X-ray computed tomography (XCT) scanning technique to locate potentially high quality rock intervals with high porosity and high TOC. Data acquired from 2D scanning electron microscopy (SEM) and 3D focused ion beam (FIB)-SEM analysis were studied to characterize the kerogen content in the samples, together with (organic and inorganic) porosity and rock fabric. The mineral framework of the samples was determined from energy dispersive spectroscopy (EDS) analysis. The FIB-SEM images in 3D were used to determine porosity and TOC by segmentation and to determine directional permeability by the Lattice Boltzmann method (LBM). Trends were obtained among the computed data, in addition to the TOC and rock fabric values that are necessary for proper shale evaluation and completion considerations.

A clear trend was observed between porosity and permeability in relation to identified facies in the core. Silica-rich facies gave higher poroperm characteristics compared to the clay-rich facies. The depositional facies was found to have a profound effect on the pore types, rock fabric and reservoir properties.

Dual-Energy Computed Tomography Imaging

XCT imaging is a powerful nondestructive technique used in the oil industry to evaluate the internal structures of cores. The acquisition of high resolution continuous images along the core length is essential in complex reservoirs to characterize reservoir heterogeneity and optimize sample selection for further detailed analysis. Dual-energy computed tomography (CT) scanning involves imaging the core at two energy levels at the same location. This dual-energy imaging provides two distinct 3D images of the core by using a high and a low energy setting. The high energy images are slightly
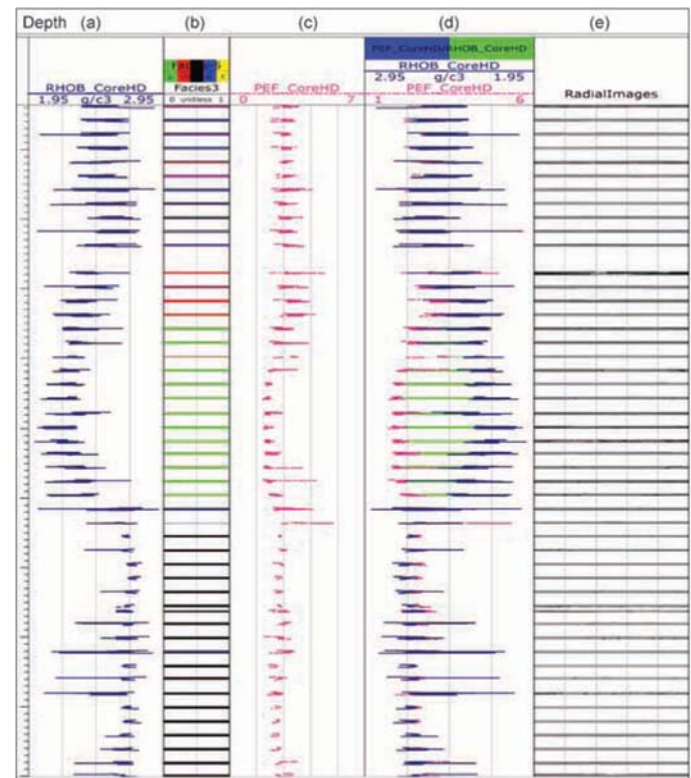
Fig. 1. Dual-energy CT data along 49 discontinuous 1 ft core sections: (a) BD, (b) identified facies, (c) PEF, (d) PEF with reversed BD, and (e) radial cross- sectional images.
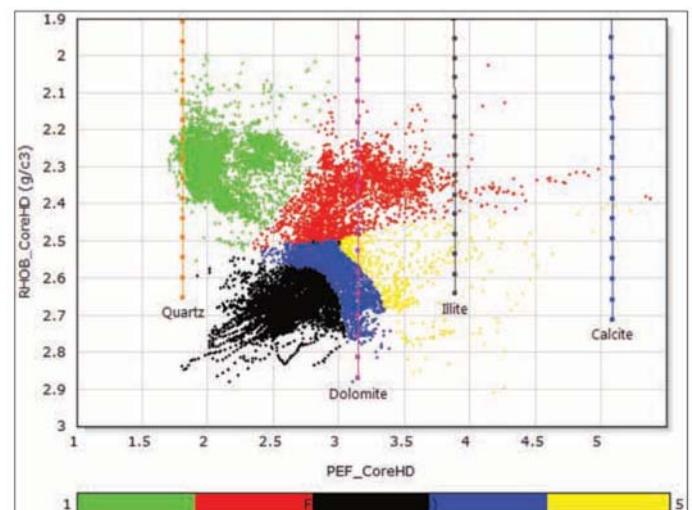
Fig. 2. BD vs. PEF for all dual-energy CT data. Color cutoffs were identified from Fig. 1d to highlight variations in shale properties. Reference lines for the main minerals are shown in the figure to indicate mineralogy variations in the core.

more sensitive to bulk density (BD) – Compton scattering effect – and the low energy images are slightly more sensitive to mineralogy – photoelectric absorption effect[2]. The high resolution computed BD values and effective atomic number (Z_{eff}), or photoelectric factor (PEF), values can be used in shale formations to interpret and quantify porosity, organic

Color Facies	Porosity	Organic Matter	Silica	Clay	Carbonate
Green	High	High	High	Low	Low
Red	Low	High	Low	High	Low
Black	Very low	Low	High	Low	Low
Blue/Yellow	Very low	Low	Low	High	Low

Table 1. Potential description of each identified facies in the cores based on PEF and BD values from dual-energy CT data

content (for identifying sweet spots) and mineralogy. When combined with other commonly available information, such as core spectral gamma data, more complex analyses can be performed. For example, the elastic properties and brittleness index can be determined[3]. Recently, the technique has been used in complex carbonate and sandstone reservoirs in the Middle East to characterize reservoir heterogeneity and optimize the sample selection for special core analysis testing[4-6]. In cases of poor core recovery and drilling mud invasion, it becomes more practical to correlate the CT data to density logs or photoelectric logs instead of the natural gamma ray logs.

Core Characterization and Sample Selection

Dual-energy CT scanning was performed on a total of 49 ft of core (49 discontinuous 1 ft sections) from a shale source rock reservoir in the Middle East. The dual-energy logs in Figs. 1a and 1c provided accurate BD and PEF data, respectively, along core lengths that were used to characterize the core sections and to efficiently identify sweet spots for the representative selection of plug sampling locations. Shale formations are often composed of stacked para-sequences[7] that are quite thin and difficult to detect from well logs. This high resolution data from the whole core therefore provides a powerful tool to define these para-sequences.

Figure 1d plots the PEF data with reversed scale BD to highlight the best quality shale intervals, with the largest gap presented in green. In this perspective, low PEF values (around 1.8) and low BD (<2.4) would indicate silica-rich shale with low clay content and high porosity. Five different facies were detected and highlighted in Fig. 1b. Figure 2 plots the BD data vs. PEF with the highlighted facies. Reference lines for the main minerals are shown in the figure to indicate mineralogy variations in the core. It is clear from Fig. 2 that this core contains no calcite minerals. The five different color facies were identified as follows and summarized in Table 1:
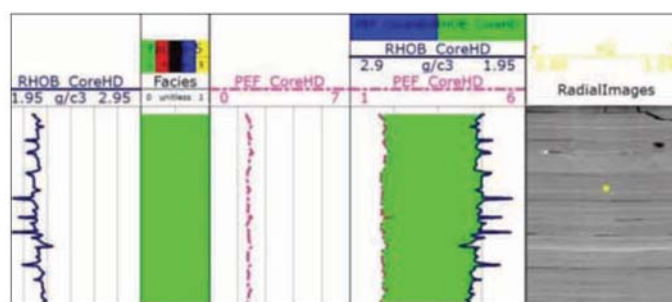
(a) green facies, large green fill (low PEF, low BD)
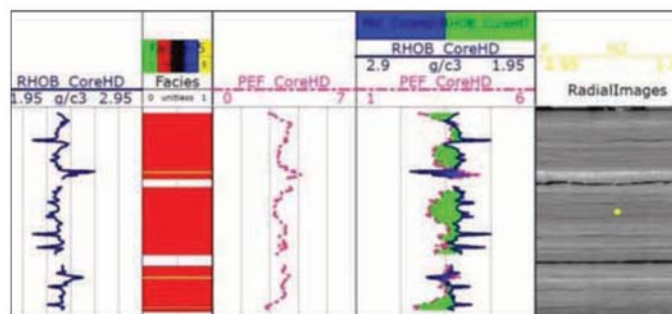
(b) red facies, small green fill (medium PEF, medium BD)
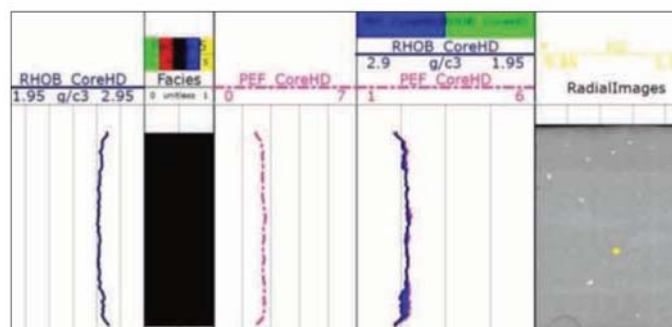
(c) black facies, small blue fill (medium PEF, high BD)
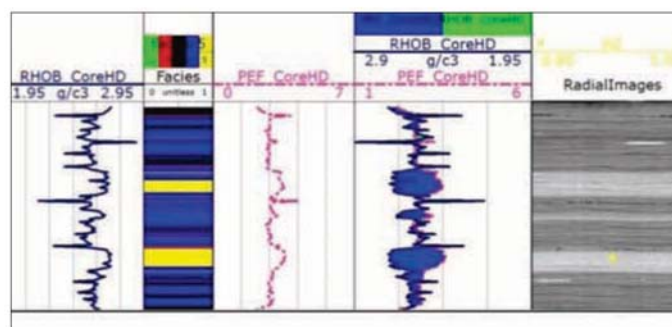
(d) blue/yellow facies, large blue fill (medium PEF, high BD)

Fig. 3. Example of color facies based on the dual-energy CT data from Fig. 1d.

• Green facies: Data with low density and low PEF. When BD is reversed as in Fig. 1d, it creates the largest gap between the PEF and ROHB curves. This identifies the potential regions for silica-rich shale with high porosity/organic matter and low clay content (sweet spots). This behavior is clearly shown in one of the zoomed intervals as represented by Fig. 3a.
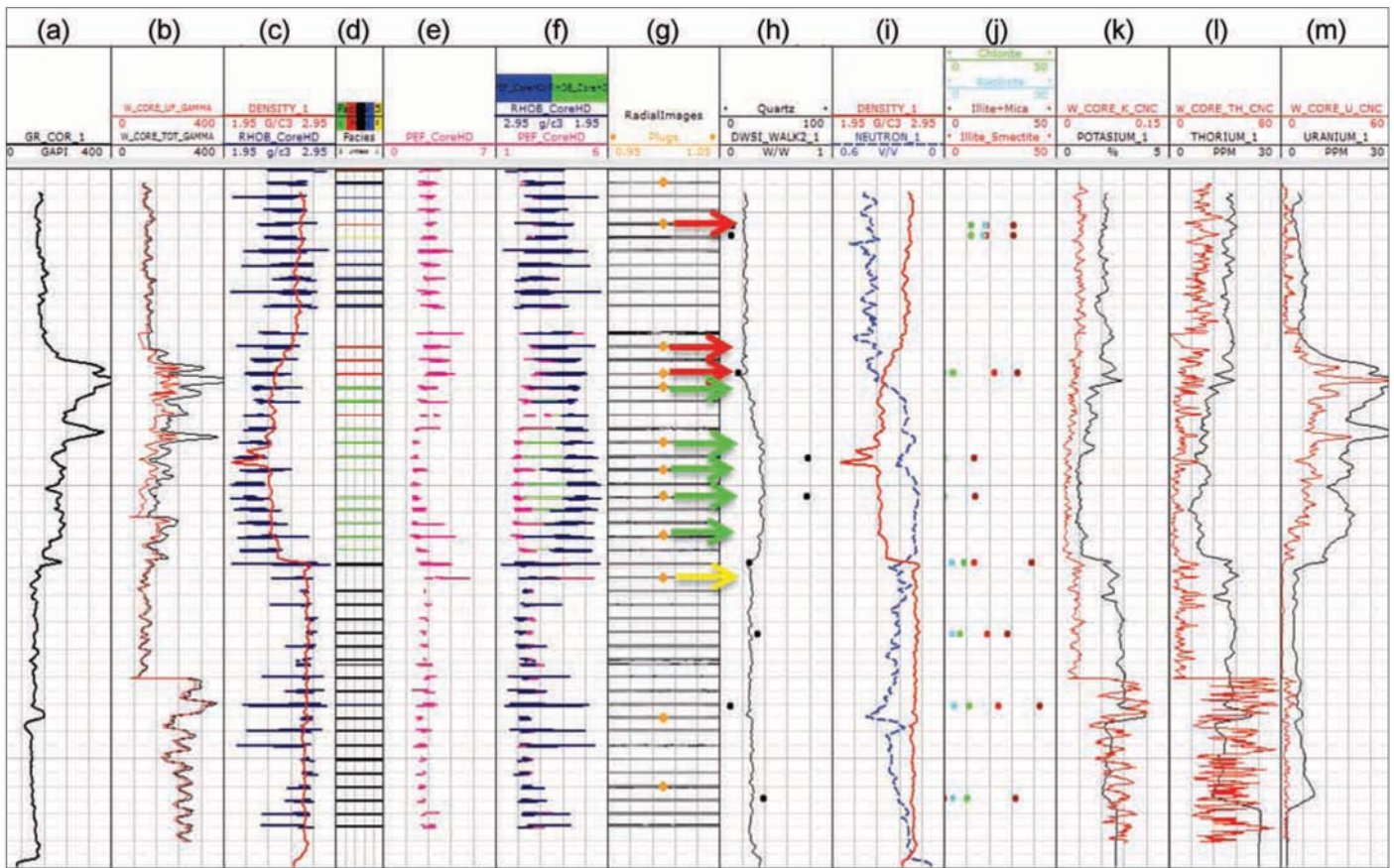
Fig. 4. (a) wireline gamma ray, (b) total gamma, (c) wireline density vs. duel-energy density, (d) identified facies from dual-energy CT, (e) PEF from dual-energy CT, (f) PEF with reversed BD from dual-energy CT, (g) recommended sampling locations, (h) % quartz from XRD data, (i) wireline neutron/density, (j) % clay minerals from XRD data, (k) potassium log, (l) thorium log, and (m) uranium log. Arrows in column (g) indicate selected plug locations for further porosity, permeability and TOC characterization. Arrow colors refer to identified facies from dual-energy CT.

• Red facies: Data with medium density and medium PEF. When BD is reversed, it creates a small gap between the curves. This identifies potential regions for clay-rich shale with low porosity/high organic matter and low silica content. This behavior is clearly shown in one of the zoomed intervals as represented by Fig. 3b.

• Black facies: Data with high density and low-to-medium PEF. When BD is reversed, it creates no gap between the curves. This identifies potential regions for silica-rich shale with very low porosity/low organic matter and low clay content. This behavior is clearly shown in one of the zoomed intervals as represented by Fig. 3c.

• Blue/yellow facies: Data with high density and medium PEF. When BD is reversed, it creates a large gap between the curves filled with blue. This identifies potential regions for clay-rich shale with very low porosity/low or ganic matter and low silica content. The larger gap in this group indicates denser layers, which are indicated with yellow; the layers are otherwise blue,

as can be clearly seen in the "facies" and "radial image" columns in Fig. 3d.

Table 1 provides only qualitative indications for the facies variations in the cores and should be confirmed by further detailed analysis using X-ray diffraction (XRD), EDS and SEM. It should also be noted that (in this analysis) each color facies has a range of dual-energy data that allows for shale property variations within the same facies. Therefore, the description in Table 1 should be used only to locate potentially high quality shale for sampling and further analysis.

Facies-based Sample Selection
Figure 4 combines wireline log data with the dual-energy XCT derived data. In column (c) the BD data from dual-energy CT shows a reasonable match with the wireline density log. Column (h) shows the percentage of quartz obtained from the XRD analysis performed in selected locations in the core to confirm the facies distribution determined from dual-energy CT and described in Table 1. High quartz percentages from the XRD data confirmed the green facies in the
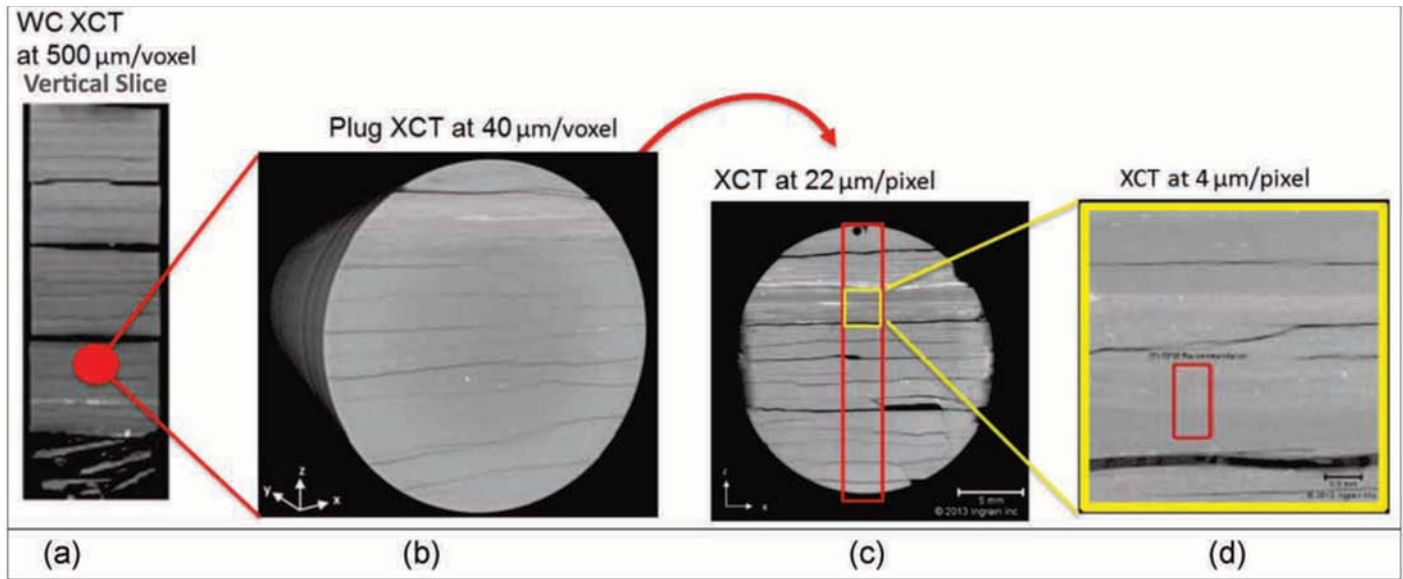
Fig. 5. Multiresolution XCT images from whole core at (a) 500 microns/voxel down to (d) XCT at 4 microns/voxel.
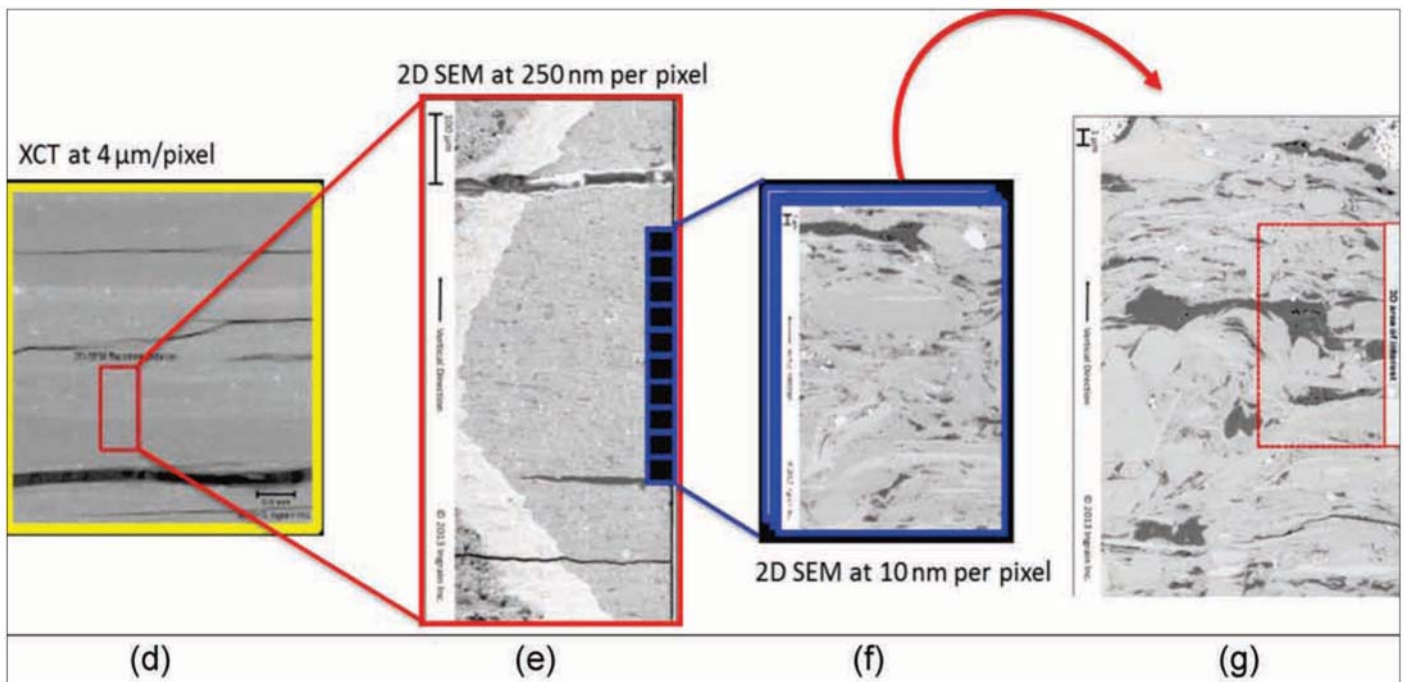
Fig. 6. 2D SEM images from plug Sample #1: (e) 2D SEM overview image at 250 nm/pixel selected from the XCT image at 4 microns/pixel (d); (f) a set of 10 high resolution 2D SEM images at 10 nm/pixel; (g) one representative high resolution 2D SEM image chosen for 3D FIB-SEM (the 3D area of interest is outlined in red).

core and the facies description in Table 1. Similarly, column (j) shows high clay concentrations from the XRD data for the red and yellow facies, which are characterized by medium-to-high PEF values, thereby confirming the descriptions in Table 1. The nine arrows in Fig. 4g indicate the selected plug sampling locations in the core for further shale characterization. Five samples were cut from the green facies (identified sweet spot), three from the red facies and one from the yellow facies.

The goal of this facies analysis and sample selection is to explore the possible links between shale depositional facies and pore types in shale rocks. This will enhance our understanding of the overall reservoir quality. It is also our goal to quantify the relationship between porosity and matrix permeability for each identified facies in the core. Identifying such trends of poroperm data and facies would facilitate upscaling, reserves estimation and well-to-well correlation.

Plug Sample #	Core Facies	Porosity (%)	Organic Matter (%)	Porosity in Organic Matter (%)	High Density Material (%)
1	Red	1.58	8.16	1.33	2.06
2	Red	1.51	9.91	1.34	1.74
3	Red	1.17	16.37	0.88	4.24
4	Green	3.09	13.48	2.73	2.99
5	Green	5.36	8.75	4.05	0.57
6	Green	3.95	6.02	2.66	0.53
7	Green	4.80	13.56	3.92	0.82
8	Green	2.63	5.01	1.84	2.46
9	Yellow	0.29	0.75	0.08	0.37

Table 2. Average values from the 10 2D SEM images for each plug sample

Sample #1 (red facies)   Sample #6 (green facies)   Sample #9 (yellow facies)
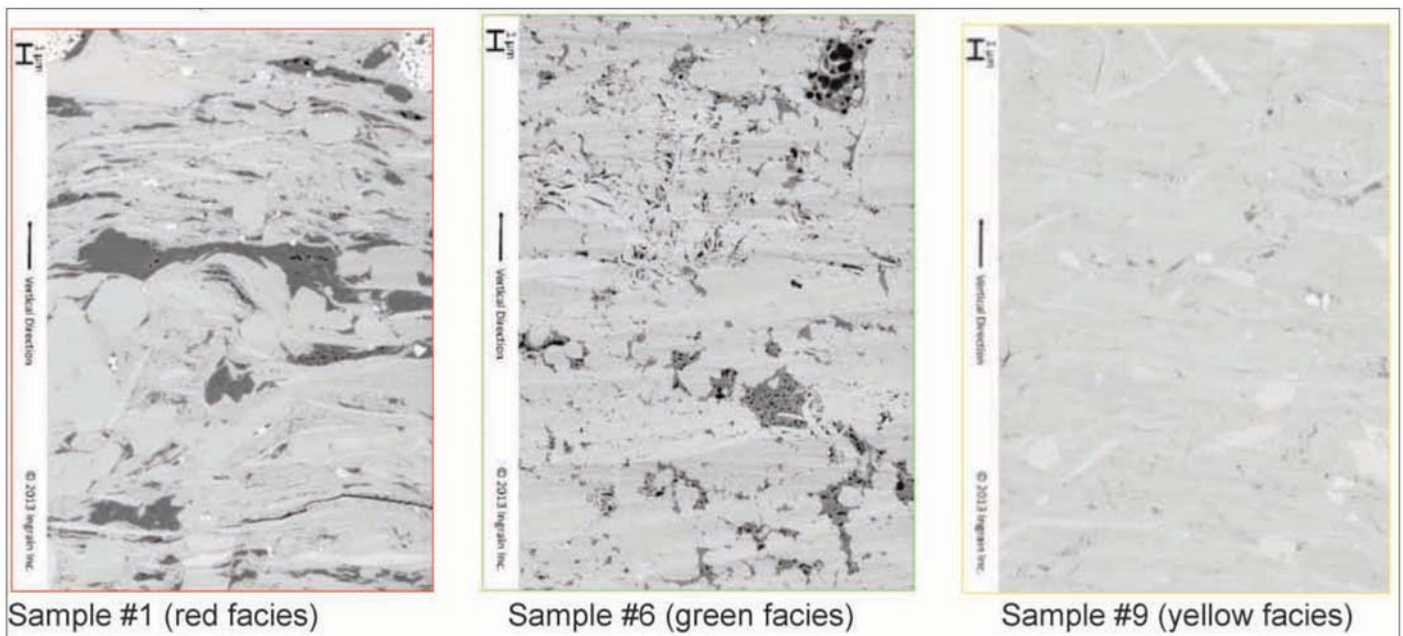
Fig. 7. Example 2D SEM images (at 10 nm/pixel) representing different facies that were identified at core scale.

Petrophysical Properties

Laboratory-based core analysis data on shale rocks are very difficult to obtain due to the tight nature of these rocks. Traditional laboratory evaluation methods may not be applicable to shale, and therefore the continued development of laboratory methods is required to help characterize and understand challenging shale reservoir behaviors. In recent years, digital imaging technology has been extensively used in the petroleum industry, including in shale formations[8], to obtain fast and reliable core data such as porosity and permeability. The new emerging technology has been called digital rock physics (DRP) and has contributed reliably to the computations of reservoir properties through image segmentation in 3D and direct simulation[4, 9-11].

Micro XCT Imaging

Each selected plug sample from the nine whole cores was scanned with a micro XCT scanner at a resolution of 40 microns per voxel. A series of multiresolution scans was then acquired, down to 4 microns per voxel, to evaluate the microscale heterogeneity and to scout for an optimal location in the sample for further SEM analysis. These micro XCT scans were combined with X-ray fluorescence readings to characterize the elemental composition of the sample and to locate a region that could adequately represent the sample. Figure 5 presents an example of such images from Sample #1.

2D SEM

In Fig. 5d, a representative region (outlined in red) was

Plug Sample #	Core Facies	Porosity (%)	Non-Connected Porosity (%)	Organic Matter (%)	Porosity in Organic Matter (%)	Absolute Permeability (Kh) (nD)	Absolute Permeability (Kv) (nD)	Conversion Ratio (%)	Porosity in OM/ Total Porosity (%)
1	Red	2.2	0.9	14.8	2.1	40	0	12.4	95.5
2	Red	2.5	0.8	9.3	2.2	19	0	19.1	88.0
3	Red	3.2	0.8	18.5	3.0	50	0	14.0	93.8
4	Green	6.1	1.5	11.4	5.1	102	0	30.9	83.6
5	Green	5.0	0.8	7.1	4.2	131	32	37.2	84.0
6	Green	6.4	1.2	9.3	5.6	348	21	37.6	87.5
7	Green	5.3	0.6	9.2	4.9	786	0	34.8	922.5
8	Green	7.7	1.2	10.1	6.8	6,111	0	40.2	88.3

Table 3. Values from 3D FIB-SEM volumes for each plug sample
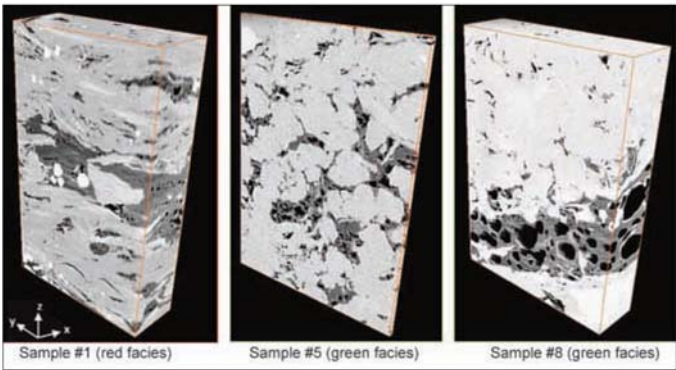
Fig. 8. Example 3D FIB-SEM video snapshots (at 15 nm/pixel) representing directional flow for different samples.
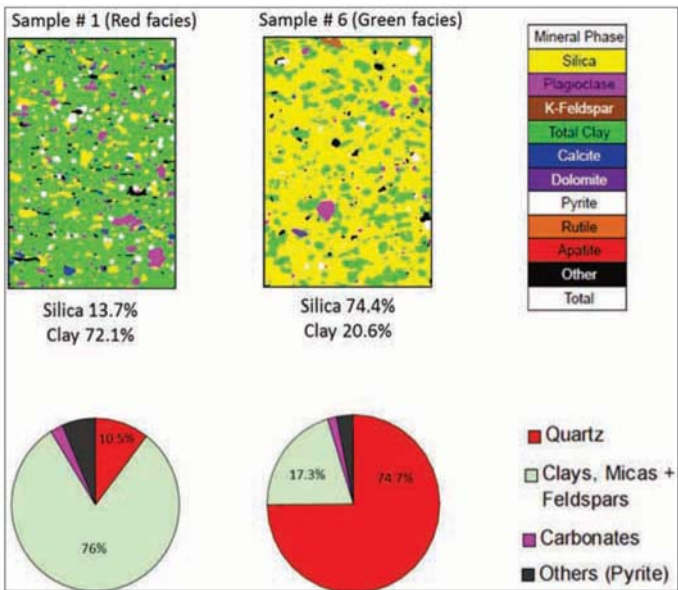
Fig. 9. Comparisons between EDS (top) and XRD (bottom) analyses for Sample #1 (red facies) and Sample #6 (green facies). EDS is represented by the mineral distribution map and XRD by pie chart. Reference mineral phase is given for the EDS mineral maps, and legend is given for the XRD pie charts.

selected for 2D SEM overview. The 2D SEM area was extracted and polished with a broad ion beam, resulting in a smooth surface of approximately 1,000 by 500 microns. That surface was imaged at a resolution of approximately 250 nanometers (nm) per pixel. Then a series of high resolution SEM images was acquired perpendicular to the lamination at a resolution of 10 nm per pixel. It is at this resolution that we were able to observe and quantify porosity and organic matter content. Figure 6 shows a representation of this analysis. Images were segmented for total porosity, porosity in organic matter, organic matter and high density. These results were used to choose one representative image with high porosity and high organic matter for 3D FIB-SEM. The segmented data for all the nine plug samples are shown in Table 2. The identified facies from the nine samples link very well with the segmented porosity and organic matter percentages. As described in Table 1 from the dual-energy CT data in the core, Table 2 shows that the green facies has the highest porosity, the red facies has low porosity, and the yellow facies has very low porosity. The pictures of selected 2D SEM images from the different facies shown in Fig. 7 confirm the obtained data in Table 2.

3D FIB-SEM

The area of interest in Fig. 6g was imaged in 3D at a resolution of 15 nm per voxel using FIB-SEM imaging and digital reconstruction techniques. Rock matrix materials, organic matter and porosity were individually identifiable via their unique gray scale signatures. Each of the 3D volumes from the plug samples was digitally analyzed, and volumetric percentages of organic matter and total porosity were determined. The porosity was further analyzed and quantified as connected, non-

Plug #	Core Facies	Silica	Pla-gio-clase	K-Feld-spar	Clay	Cal-cite	Dolo-mite	Sider-ite	Anhy-drite	Pyrite	Rutile	Apa-tite	Other	Total
1	Red	13.7	5.0	0.0	72.1	1.0	0.2	0.0	0.0	4.7	0.5	0.0	2.9	100
2	Red	21.3	5.4	0.3	62.4	0.1	2.5	0.0	0.0	4.7	0.5	0.0	2.8	100
3	Red	25.6	9.8	0.0	44.5	0.0	6.1	0.0	0.0	7.6	0.8	0.2	5.5	100
4	Green	57.5	5.6	0.0	29.1	0.0	1.7	0.0	0.0	3.2	0.3	0.9	1.6	100
5	Green	64.8	2.3	0.0	18.7	0.1	3.9	0.0	0.0	1.3	0.1	0.0	8.8	100
6	Green	74.4	1.6	0.5	20.6	0.1	0.2	0.0	0.0	1.0	0.1	0.0	1.6	100
7	Green	73.0	5.0	0.0	19.1	0.1	0.0	0.0	0.0	1.3	0.4	0.0	1.1	100
8	Green	63.4	4.4	0.0	25.3	0.1	2.1	0.0	0.0	1.3	0.5	0.3	2.7	100
9	Yellow	20.2	8.3	0.0	67.0	0.0	0.0	0.0	0.0	0.5	0.5	0.3	3.1	100

Table 4. Volume percent mineralogy from EDS analysis

Sample	Core Facies	Illite/Smectite	Illite+Mica	Kaolin-ite	Chlo-rite	Chert	Quartz	K Feld-spar	Pla-gio-clase	Cal-cite	Dolo-mite	Sider-ite	Py-rite	Total
1	Red	19.9	31.6	15.8	11.2	0.0	10.5	TR	2.1	1.2	1.2	0.0	6.4	100
2	Red	20.2	31.9	12.1	10.0	0.0	12.7	TR	2.9	0.0	1.8	0.0	8.6	100
3	Red	23.9	27.5	0.0	3.5	0.0	21.0	TR	3.6	0.0	2.0	0.0	18.5	100
4	Green	14.8	25.4	0.0	0.2	0.0	42.0	TR	4.2	0.0	4.5	0.0	8.9	100
5	Green	0.0	15.8	0.0	0.0	0.0	76.4	TR	2.3	0.0	1.5	0.0	4.1	100
6	Green	0.4	17.3	0.0	0.0	0.0	74.7	TR	2.8	0.0	1.6	0.0	3.1	100
7	Green	0.2	20.2	0.0	2.6	0.0	69.4	TR	2.0	0.0	0.8	0.0	4.8	100
8	Green	0.2	25.1	0.0	4.0	0.0	58.9	TR	2.3	TR	3.1	2.3	4.0	100
9	Yellow	25.7	35.5	0.0	10.4	0.0	19.3	TR	3.2	0.0	0.0	5.9	0.0	100

Table 5. XRD analysis

connected and associated with organic matter. The connected porosity was used to compute absolute permeability directly in the 3D digital rocks in the horizontal and (whenever possible) vertical directions using the LBM[12]. Porosity associated with organic matter can be an indicator of organic matter maturity and flow potential. Table 3 gives the segmented values from the 3D FIB-SEM volumes. The table also gives calculations of the conversion ratio, and the organic porosity and total porosity in percentages. The conversion ratio percent would represent the porosity within the organic matter with respect to the organic matter volume, while the organic-to-total porosity percent would represent the percentage of pores in the organic matter with respect to the total porosity

in the 3D volume. The 3D volume data in Table 3 is a clear confirmation of the potential relationship among facies, pore type, porosity and flow characteristics in shale. Both the red and green facies have high percentages of organic matter, but the red facies are at the lower range of porosity, which influenced the flow properties and thereby yielded much lower matrix permeabilities than the green facies samples. This can be quantified in Table 3 by the conversion ratio values, which show higher than 30% for the green facies and lower than 20% for the red facies. These findings suggest that further analysis of the organic matter and mineral framework in the red facies samples is required to determine the reasons behind the lower conversion ratios. Sample #9 was excluded from the 3D FIB-SEM
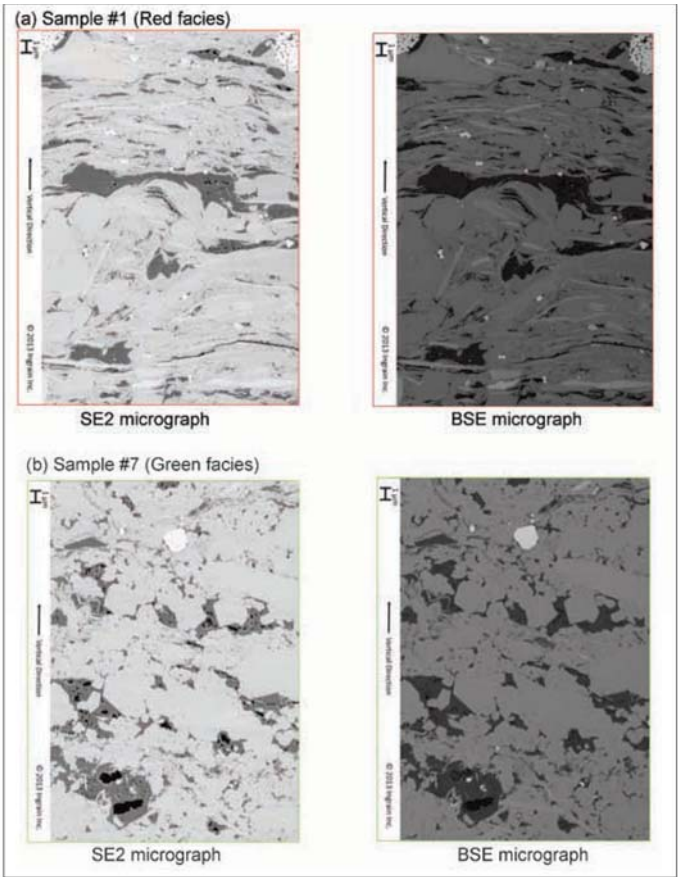
Fig. 10. Different pore types detected in different facies.

Fig. 11. Poroperm characteristics of the red and green facies with Eagle Ford upper and lower bounds.

analysis because the sample showed no flow potential due to the very low porosity in the 2D SEM image in Fig. 7 and Table 2.

Figure 8 shows video snapshots from the different facies with their different permeability values. This figure serves as a good visual means to evaluate the simulated directional permeability values in Table 3.

Fig. 12. Organic matter vs. porosity for the red and green facies: The green facies have larger porosity but less organic matter.

Sample #1 has low horizontal permeability with low porosity in the organic matter. Sample #5 has higher horizontal permeability and gave rise to flow in the vertical direction as well. Sample #8 has the highest horizontal permeability value, and the reason is clearly seen to be an unrepresentative streak of organic matter with relatively large pore sizes. The permeability in these shale facies seem to be controlled by the organic matter distribution and the porosity associated with the organic matter.

Mineralogy

Areas of interest for the EDS analysis were selected to include the analyzed 2D SEM images and the 3D area of interest. The SEM-EDS area of interest is imaged at a resolution of approximately 200 nm per pixel and covers an area of approximately 200 by 150 microns. Table 4 gives the mineral volume percentages for all plug samples analyzed by EDS. The EDS results confirm a clear link between mineralogy and the core facies as analyzed from dual-energy CT data on the whole cores and as previously described in Table 1. The red and yellow facies are clay-rich shale with less than 25% silica, while the green facies are silica-rich shale with less than 25% clay. One would then be tempted to think of a link between mineralogy and porosity when comparing the red and green facies. These two facies have similar fractions of organic matter but different porosity. The reason for this could be either maturation of kerogen or the mineral framework of the samples.

XRD analysis was performed on all nine samples to confirm the EDS mineralogy results. Table 5 gives the XRD data and confirms the EDS analysis. Figure 9 presents schematic comparisons between the EDS and

Fig. 13. Comparison between the data obtained from the selected 2D SEM image for 3D FIB-SEM and the 3D volume data: porosity (left) and organic matter (right) — heterogeneity effect.

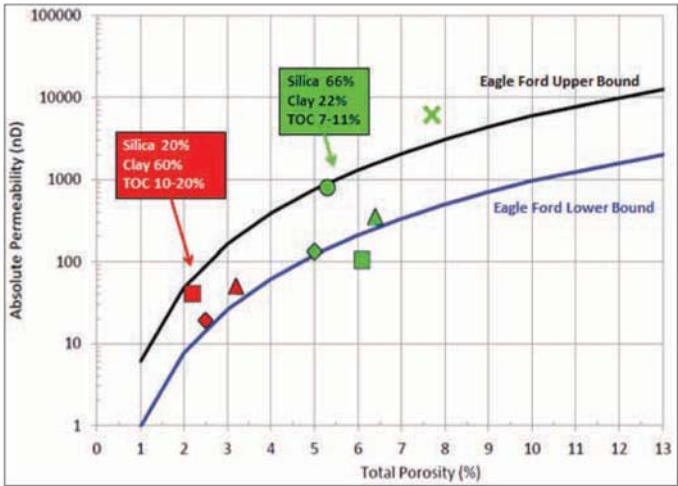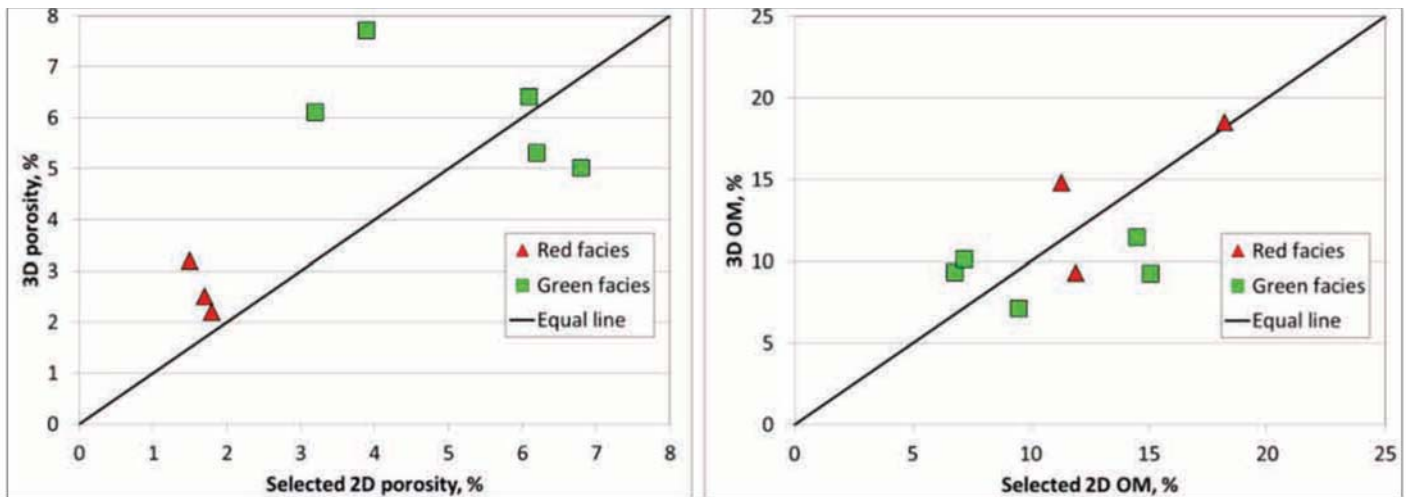XRD analyses for Sample #1 from the red facies and Sample #6 from the green facies. EDS is represented by the mineral distribution map and XRD by the pie chart.

Effects of Depositional Facies on Pore Types, Organic Matter, Rock Fabric and Reservoir Properties

Shale pore systems may generally be described and classified as inter-granular (between grains), intra-granular (within grains) or organic matter[13]. Porosity within organic matter would be formed by the shrinkage of kerogen during maturation. The inter-granular and intra-granular pores are inorganic and so would normally be located in the matrix. The organic matter itself may also be classified as nonporous, spongy or pendular[8].

In this study, each area of the 2D SEM produced two images: Secondary electron (SE2) micrographs that are used to quantify porosity and organic matter, and backscattered electron (BSE) micrographs that better display the contrast between the solid components of the rock.

Red Facies

Figure 10a presents examples of such images for the red facies – Sample #1. In this sample, in the SE2 image, the organic matter appears to be compacted between the delicate clay mineral layers and elongated in the horizontal direction. This compaction must have led to the compaction of the pores within the organic matter. The BSE image clearly shows the clay minerals oriented in the horizontal direction due to overborne pressure. The pores in this facies are almost all in the organic matter, and the porosity value of Sample #1 is around 2% with only 1% porosity connected in the 3D FIB-SEM volume. This has led to a very low matrix permeability of 40 nano-darcy (nD).

Green Facies

Figure 10b presents examples from the green facies – Sample #7. In this sample, in the SE2 image, the organic matter appears to be protected from severe pressure compaction between the strong quartz grains and the microcrystalline silica particles. The organic matter in this facies seems to have an irregular shape with a spongy type of porosity at 5%. Therefore, the pore space within the organic matter was preserved and gave good connectivity in 3D, which yielded a very high permeability value at 786 nD. The BSE image in Fig. 10b clearly shows the grainy structure of the quartz particles around the organic matter that is spread out in the whole image. The pores in this facies are almost all in the organic matter.

The poroperm characteristics of these shale facies are plotted from the Table 3 data and are shown in Fig. 11. The green facies samples are at the higher poroperm range. It is interesting to note that the red facies samples have higher concentrations of the organic matter – 10% to 20% – and yet gave lower poroperm values compared to the green facies samples with only 7% to 11% organic matter. These organic matter figures were derived from the 3D FIB-SEM volumes. In this perspective, flow properties of this shale formation are controlled more by the rock fabric, the mineralogy and the resultant porosity within the organic matter.
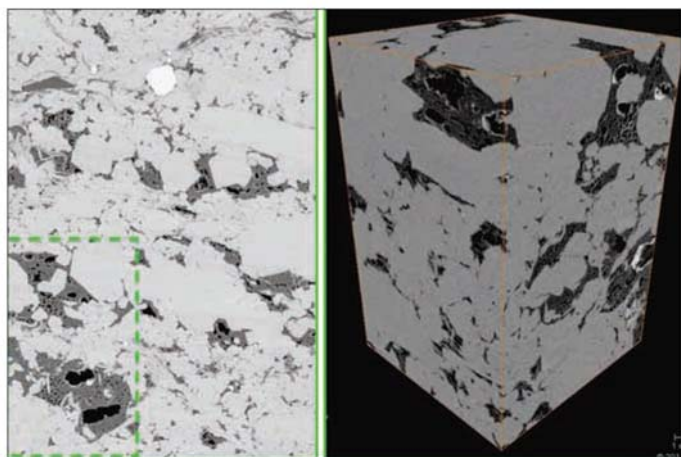
Fig. 14. Comparison between the data obtained from the selected 2D SEM image (left) from Sample #7 and the 3D FIB-SEM volume (right) — heterogeneity effect.
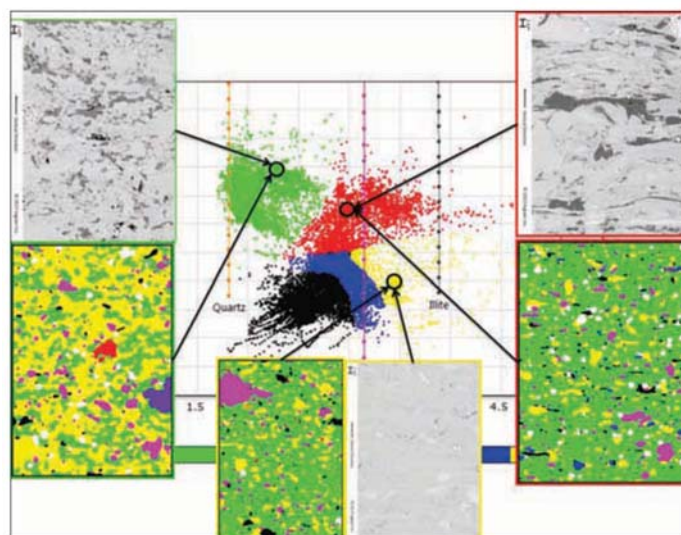

Fig. 15. Analysis of reservoir shale characteristics (porosity, mineralogy and organic matter) from core dual-energy CT scanning and SEM.

As an initial comparison of these poroperm results to those for North American shale plays, the data from this Middle East shale gas fell within the upper and lower bounds of Eagle Ford shale in the United States. Figure 12 plots organic matter vs. porosity, both derived from the 3D FIB-SEM volumes, and shows that the red facies have more organic matter with lower conversion ratios.

Effects of Heterogeneity

Shales are heterogeneous at millimeter to centimeter scales. Figure 13 compares the segmented porosity and organic matter data from the 2D SEM image with those from the 3D FIB-SEM volume. The figure shows that analyzed samples show porosity variations that could be larger than those of the organic matter

at different scales. Porosity estimations from the 3D volume could double the initial estimations from the 2D SEM images.

Figure 14 serves as a visual comparison of the 2D and 3D SEM images, where the porosity in this example (Sample #7) decreased from 6.2% in the 2D image to 5.3% in the 3D image, and the organic matter decreased from 15.1% in the 2D image to 9.2% in the 3D image. The average results from all 10 of the 2D SEM images from this sample were 4.8% porosity and 13.6% organic matter.

Close inspection of all 10 of the 2D SEM images acquired for every sample in this study revealed the porosity variation – within each set of the 10 2D SEM images – to be less than 3% porosity unit and the organic matter variation to be less than 5%.

Validation of Core Facies from Dual-Energy XCT

The initial plug sample selection in the core was based on accurate application of a dual-energy XCT imaging technique that produced continuous BD and PEF data along the core lengths. The sample selection targeted three different facies (green, red and yellow) in the core identified from the dual-energy CT data. The shale characteristics – porosity, organic matter and mineralogy – of the selected samples from the core facies were then confirmed through segmented porosity and organic matter values derived from high resolution SEM images and through EDS and XRD analyses. Figure 15 is a nice representation of the excellent match in porosity, mineralogy and organic matter between core facies described by dual-energy CT and by high resolution SEM images. This would assist in more efficient upscaling, improved reserves estimation and enhance well-to-well correlation.

Conclusions

Initial core facies characteristics – porosity, organic matter and mineralogy – of a shale formation in the Middle East were computed using the dual-energy CT scanning technique. This core facies analysis was used to locate potential sweet spots in the core for optimum sample selection. The selected plugs, following a well-defined DRP workflow, underwent multiresolution scanning to construct 3D FIB-SEM volumes for the determination of shale porosity, organic matter and mineralogy. The objectives of the study were to explore possible links between shale depositional facies and pore types as well as to quantify the relationship between porosity and matrix permeability for each identified facies in the core. The objectives of the study

were fulfilled and the following is a summary of the key findings in this shale play.

1. A robust dual-energy CT scanning technique was used to characterize a shale gas core and to identify potential facies intervals for DRP analysis.

2. Absolute shale matrix permeability was determined in horizontal and vertical directions in 3D FIB-SEM volumes.

3. Only two samples (out of eight) gave 3D connectivity in the vertical direction for permeability simulation in the silica-rich samples. This is consistent with a shale depositional environment and anisotropy considerations.

4. Almost all the porosity was found within the organic matter volume. Consequently, flow was only possible through organic matter within the 3D volumes.

5. The silica-rich facies gave higher poroperm characteristics compared to the clay-rich facies. This is due to the pressure compaction effect on the soft clay-rich samples, which caused the organic matter to be squeezed within a clay mineral framework, leading to closure of the pore space.

6. A very high permeability value (6,000+ nD) was simulated in one of the samples, which a visual examination determined was caused by an unrepresentative porous organic matter layer along the horizontal direction. Such an observation has led to the recognition of the importance of the visuals in explaining the petrophysical data in the samples.

7. A higher percentage of organic matter was not found to be a good indication for high porosity or permeability in the clay-rich shale samples in this study. The conversion ratios of organic matter should be taken into consideration when judging porosity or permeability.

8. A clear trend was observed between porosity and permeability in relation with the identified facies in the core.

9. The depositional facies was found to have a great effect on the pore types, rock fabric and reservoir properties. Of particular importance are the mineralogy and clay in the samples.

10. Shale heterogeneity in this formation showed larger effects on porosity variability than organic matter variations at different scales.

11. The results and interpretations in this study enhanced our understanding of the complexity of unconventional shale reservoir quality.

Nomenclature
K   permeability
Kh   horizontal permeability
Kv   vertical permeability
Z_{eff} effective atomic number
Ø   porosity

Acknowledgements

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Biographies

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Dr. Tariq M. Al-Ghamdi is a Reservoir Engineer working in Saudi Aramco's Reservoir Description and Simulation Department. His responsibilities include management and petrophysical evaluation of exploration and gas fields. Tariq is currently leading the unconventional Shale Gas team in Saudi Aramco. His main interests are optimizing petrophysical evaluation, permeability modeling and modeling saturation height function; recently Tariq has been involved in digital core analysis and numerical simulations of special core analysis and nuclear magnetic resonance. He has published and coauthored numerous papers and journals.

Tariq received his B.S. degree from the University of Tulsa, Tulsa, OK, his M.S. degree from Heriot-Watt University, Edinburgh, U.K., and his Ph.D. degree from the University of New South Wales, Kensington NSW, Australia, all in Petroleum Engineering.

Safouh Koronfol joined Ingrain Inc. in May 2012 and is the Operations Manager. He has 10 years of special core analysis experience. Safouh was the Head of the Special Core Analysis Department at Weatherford Laboratories Abu Dhabi and later became the SCAL coordinator between Weatherford Labs globally and Shell/Petroleum Development Oman in Muscat, Oman. Safouh received his B.S. degree in Industrial Chemistry from University of Aleppo, Aleppo, Syria. He is an active member of Society of Petrophysicists and Well Log Analysts (SPWLA), the Society of Petroleum Engineers (SPE) and the Society of Core Analysts (SCA). Safouh has authored and coauthored seven technical papers on both conventional SCAL and digital rock physics.

Dr. Moustafa R. Dernaika has been the Manager of Ingrain Inc. Abu Dhabi since 2010. Before he joined Ingrain, he worked for Emirates Link ResLab LLC (Weatherford Laboratories) as the Regional Special Core Analysis (SCAL) Manager in Abu Dhabi. He has 15 years of routine and SCAL experience with special interest in business development, project management and data interpretation.

Moustafa has written 26 technical papers. His current research areas include digital rock physics, dual energy coiled tubing applications and the variations of petrophysical and flow properties with rock types and wettability.

Dr. Joel D. Walls is a Geophysicist focused on research, development, and commercialization of advanced digital rock physics services for unconventional reservoirs. He joined Ingrain Inc. in 2010, and as the Director of Technology, Joel guides the development and commercialization of services focused on shale plays. Joel was a co-founder and the first president of the

Society of Core Analysts (SCA). He is a member of the Society of Economic Geologists (SEG), Society of Petroleum Engineers (SPE) and the Society of Petrophysicists and Well Log Analysts (SPWLA). Joel is the author of numerous publications in several geophysical and petrophysical journals, and holds four U.S. patents in the rock physics and reservoir characterization.

He received his B.S. degree in Physics from Texas A&M University, College Station, TX. Joel received his M.S. and Ph.D. degrees in Geophysics from Stanford University, Stanford, CA.

Optimization and Post-Job Analysis of the First Successful Oil Field Multistage Acid Fracture Treatment in Saudi Arabia

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Abstract

Multistage acid fracture treatments are utilized in low permeability carbonate reservoirs (permeability <10 millidarcies (mD)) to stimulate the formation by creating highly conductive fractures in the formation and bypassing near wellbore damage. The fracture is generated at high pressures, which are required to break the rock open, while using a viscous pad. The fracture is then kept open by adding gelled or emulsified acid to create uneven etches on the surface of the fracture.

The pre-job analysis in the laboratory of the acid fracturing treatment fluids' reaction and compatibility is crucial as the operational success is highly dependent on the fluid's chemical reactions. The key problem with acid fracturing treatments is the difficulty in appraising the actual downhole reactions and performance of the treatment chemicals within the heterogeneous rock. This problem can be resolved when flow back fluids and the chemical ions are analyzed to understand the reactions that occurred downhole. Also, since acid fracture treatments require pumping large volumes of fluids, flowing back the entire amount of fluids becomes a challenge due to the low reservoir permeability and the associated reservoir rock capillary pressure effects.

This article will discuss an acid stimulated well's pre-fracture treatment evaluation based on laboratory experiments – coreflood, rock dissolving capacity and fluid compatibility – in addition to comparisons of the expected chemical ion returns with the actual ions observed in the flow back fluids.

The results of this flow back fluid analysis showed a recovery of 17% of the chemicals pumped during the treatment with a stabilized production rate of 3 thousand barrels of oil per day (MBOPD). Further water analysis indicated the presence of 25% to 30% formation water, while the critical ions analyzed showed the effectiveness of the corrosion inhibitor package, acid system dissolving capacity and crosslinker fluid recovery. It is expected that this article will provide a learning process for optimizing future multistage acid fracturing treatments in Saudi Arabia.

Introduction

The LF formation in Saudi Arabia is the deepest of the upper Jurassic limestone reservoirs in the K-field. Underlying two other carbonate reservoirs, the LF reservoir has been appraised as a relatively lower quality formation with RQI/FZI of ~0.13/~0.73 and a net to gross ratio of ~0.62. This reservoir is described as heterogeneous and clayey limestone/dolomite with a permeability range from 1 millidarcy (mD) to 15 mD and porosity range from 5% to 20%[1]. The LF reservoir is a 120 ft thick reservoir at a depth of 6,900 ft with a bottom-hole static temperature of 150 °F. Mineralogy of the core samples from this reservoir is mainly calcite with some dolomite and ankerite. Table 1 shows the minerals in the formation rock with their chemical composition.

No improvement in productivity was noticed with several attempts to stimulate wells in this reservoir. This is due to the tight formation and low reservoir quality. The decision to conduct and evaluate a multistage acid fracturing operation in this reservoir was considered highly important to assess the value of this completion design for developing this field. A multilateral well with

Calcite	Ankerite	Dolomite	Illite	Kaolinite	Quartz	Pyrite
$CaCO_3$	$Ca(Fe,Mg,Mn)(CO_3)_2$	$CaMg(CO_3)_2$	$(K,H_3O)(Al,Mg,Fe)_2(Si,Al)_4$ $O_{10}[(OH)_2,(H_2O)]$	$Al_2Si_2O_5(OH)_4$	SiO_4	FeS_2

Table 1. Chemical composition of minerals

three laterals was drilled with a plan to create seven fractures in the motherbore. The candidate well selected for the multistage acid fracturing was this trilateral oil producer. The main wellbore was completed with the multistage fracture completion, while the other two laterals remained open hole. These two laterals were isolated by a blanking pipe installed and connected between the upper production packer and the lower multistage fracture completion tie-back receptacle. The pipe, used to ensure isolation during the job, was re-moved after the operation was completed. In regards to the completion, both upper and lower completions were equipped with VM-95 tubular (VM = various material) to withstand the pressure of the operation. All equipment exposed to the fracturing operation in the wellbore was ensured to have a pressure rating of 10,000 psi. Also, it was ensured that sour service piping was used to avoid any corrosion from the hydrogen sulfide (H_2S) found in the crude oil from the initial analysis. The completion tubular mainly contained iron (Fe), manganese (Mn) and chrome (Cr), chosen to resist the expected sour environment of H_2S and keep it from corroding the tubular while producing the well. The Cr material in the tubular reacts with oxygen on the surface and forms an impermeable layer of chromium(III) oxide (Cr_2O_3). This layer protects the tubular from direct contact with H_2S, as shown in Eqn. 1:

$$4Cr + 3O_2 \longrightarrow 2Cr_2O_3 \qquad (1)$$
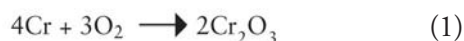
This layer is very sensitive to hydrochloric (HCl) acid, however, and will be removed if it comes into direct contact with it. So a good corrosion inhibitor package was used in this job to protect the chrome tubular from the multistage acid fracture treatment chemicals. The Fe, Mn and Cr ion analysis in the flow back water would help determine if there was any corrosion to the tubular while conducting the multistage acid fracture job.

The pumping schedule to fracture this well used cross-linked gel to initiate the fracture, followed with emulsified acid to achieve deeper penetration into the formation. This was followed with cross-linked gel to maintain the bottom-hole pressure, control the leakoff rate and create a viscous fingering effect, which alters the acid path in
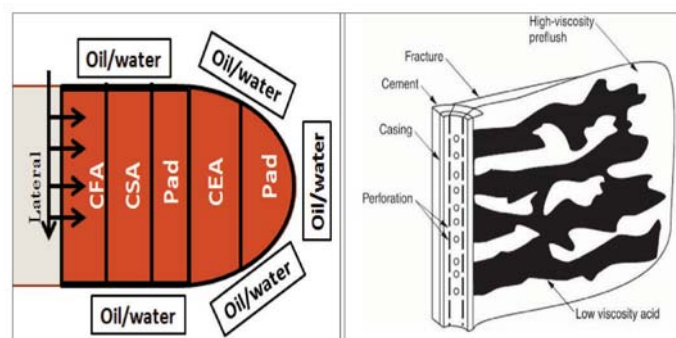
Fig. 1. Order in which chemicals were pumped in each stage and viscous fingering by the acid in the fracture.

the fracture and ensures irregular etched patterns along the fracture face. This was followed by gelled acid to break the cross-linked pad and create a different etching path on the fracture face, before concluding with a diversion pill. Figure 1 illustrates the sequence of the pumping schedule used to acid fracture this well. The same sequence of fluids was repeated two times before injecting the final stage with closed fracture acid (CFA). This final acid stage is pumped below fracturing pressure to allow the acid to deepen and widen existing channels, enhance fracture conductivity and minimize fracture closure effect[2].

Emulsified acid is a key stage in the sequence of fluids injected to create a fracture. Acid-in-diesel emulsions are used to create a retarded acid system, Fig. 2. Diesel acts as a diffusion barrier between the acid and the rock. De Rozieres et al. (1994)[3] studied the effective diffusion coefficients in such emulsified acid and found it to be three orders of magnitude lower than plain acid. Due to the lower reaction rate, the live acid can penetrate deeper into the formation, creating more effective wormholes. Broaddus et al. (1968)[4] showed that emulsified acid provides excellent etching and better fracture conductivity than regular acid. They also found that better fracture conductivity can be achieved by injecting different acid solutions with different de- grees of retardation. They proposed that the most retarded acid should be injected first.

Bartko et al. (2003)[2] studied the impact of acid type and formation lithology on fracture performance by
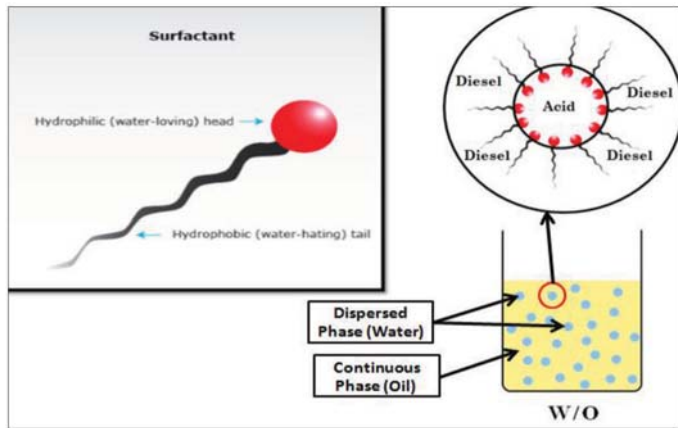
Fig. 2. Emulsified acid properties and components expected in the flow back.

evaluating field production data, flow back analysis and lab work. They found that emulsified acid shows superior performance in calcite and the more challenging dolomitic rocks. Fracture conductivity is achieved in acid fracturing by generating nonuniform rock dissolution at the fracture face. The degree of heterogeneity on the fracture face due to the physical and chemical properties of the formation rock influences the reaction rate and enhances differential etching[5]. Another factor that can affect the etching pattern is the diffe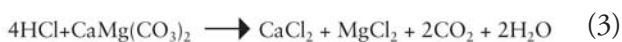rence in permeability and porosity, indicated by variation in acid leakoff rates[5]. Viscous fingering by different acids injected inside the fracture after a preflush with a cross-linked spacer helps to further enhance etching.

Researchers have reported their studies of chemical analysis of flow back samples after acid treatments in sandstone[6-8]. Nasr-El-Din et al. (1999)[9] and Mohamed et al. (1999)[10] examined acid return samples from carbonate reservoirs. Chemical analyses of the flow back samples collected during well production were used to assess the performance of the acid fracturing treatment. During acid placement and soaking in the wellbore, acid interacts with formation rock and downhole hardware to produce ions. Analyses of the concentration of key ions in the flow back samples, collected during wellbore cleanup, are carried out to identify the source of these ions (formation or hardware). In carbonate reservoirs, high concentrations of calcium (Ca) and magnesium (Mg) are expected in flow back samples as products of the acid's reaction with formation rock. Equations 2 and 3 show the reactions between HCl acid/calcite and HCl acid/dolomite, respectively:

$$2HCl + CaCO_3 \longrightarrow CaCl_2 + CO_2 + H_2O \qquad (2)$$

$$4HCl + CaMg(CO_3)_2 \longrightarrow CaCl_2 + MgCl_2 + 2CO_2 + 2H_2O \qquad (3)$$

From these equations, we can clearly see that Ca, Mg and chloride (Cl) ions are expected in our flow back water analysis. Other information that can be gained using flow back analysis is a check of the integrity of the wellbore hardware, determining if any severe corrosion to metal components has happened because of interaction with acid solutions. This can be assessed by monitoring the key ions (total Fe, Cr, nickel (Ni) and molybdenum (Mo)) found in the metal alloys used to build the downhole tubular. Additional important information that can be obtained from this analysis is the strength of the flow back acid. This important finding helps to protect downhole hardware from severe corrosion due to the presence of live acid in the flow back, and it also helps in optimizing the soaking time and overflush volumes, calculated to allow the acid to fully interact with the rock until the acid is totally spent. The presence of live acid can be detected using pH and acid concentrations.

The objectives of this study were to: (1) Evaluate the impact of breakers and acid stages on the viscosity of the cross-linked gel, (2) Assess the emulsified acid/rock interaction and its propagation in tight cores, (3) Evaluate acid stimulation treatments based on a chemical analysis of the return fluids, and (4) Investigate the amount of corrosion in the wellbore hardware.

Procedure and Experimental Work
Emulsified Acid Stability
One of the main chemical systems in multistage acid fracture operations is the emulsified acid. The emulsified acid has certain properties that help generate efficient reaction rates and the deep etchings that are very critical in this job[11]. The emulsified acid consists of acid, diesel and emulsifier. The most important criterion is to keep the emulsified acid stable for the amount of time needed to create etches and wormholes in the formation rock[12]. In addition, it is very important for the emulsified acid to be able to break down to its original components for an easy flow back operation. Tests were conducted by mixing the emulsified acid, then adding chunks of the reservoir's core sample to observe the reaction under reservoir conditions. The goal was to observe the time it took the emulsified acid to separate into diesel and acid. In addition, it was critical to check if any portion of the emulsified acid was stabilized after the reaction due to the fines in the reservoir, which would hinder the flow back operation.

Viscosity Measurements
The high-pressure/high temperature (HPHT) viscometer was used to measure the apparent viscosity
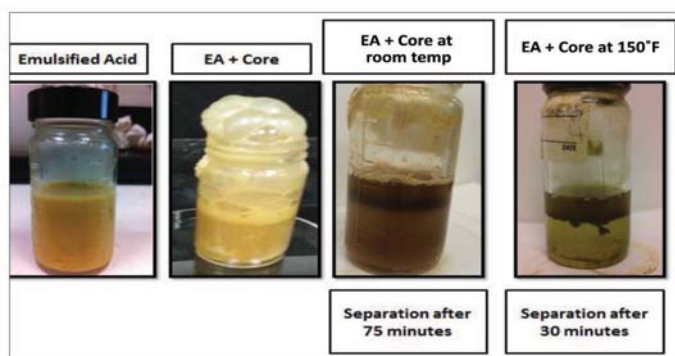
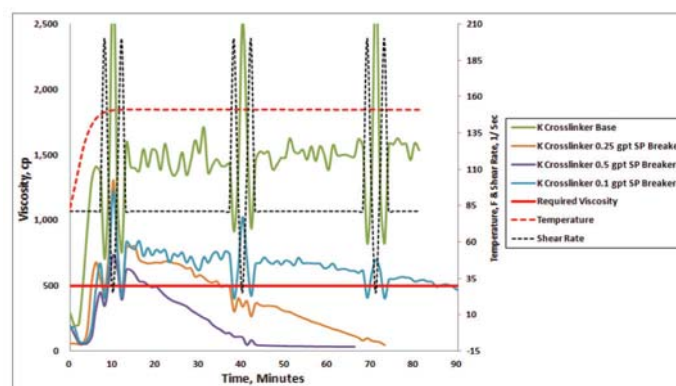Fig. 3. Results of emulsified acid reaction stability tests in the lab.

Fig. 4. Viscosity measurements of the low temperature instant crosslinker with different breaker concentrations.

Fig. 5. Crosslinker stability test when mixed with emulsified acid and no internal breaker.

of the fracturing fluid samples under different shear rates and constant pressure at reservoir temperature. The HPHT viscometer utilizes a standard R1/B5 bob and rotor, which requires a sample volume of 52 cm^3. The viscometer uses a sliding carbon block for dry heating, and a temperature sensor is mounted on the stator/bob to measure the sample's temperature. A pressure of 1,000 psi was applied to minimize evaporation of the sample, and to keep all generated gases in liquid state.

Viscosity measurements were performed under different shear rates to simulate the flow of the fracturing fluid through the production tubular, through the perforations and inside the created fracture. The goal of this test was to measure the time needed for the cross-linked fluid to break down so it could flow back easily during the flow back operation.

Gel Stability

The crosslinker system is a key component in multistage acid fracturing operations. The crosslinker builds high viscosity and breaks open the rock. The crosslinker fluid then keeps the fracture open while maintaining high viscosity to enable the acids and chemicals to flow inside and react with the rock. Furthermore, it enables the fluids to viscously finger through and thereby randomly etch the rock, which results in the high conductivities that enhances production. Moreover, the high viscosity

of the crosslinker helps with controlling acid leakoff, thereby generating deeper etching and fractures. The challenges, though, are the removal of the viscous fluid after the operation is completed and unplugging the crosslinker filter cake from the permeable reservoir areas. This is achieved by adding breakers to the cross-linked fluids to lower the crosslinker's viscosity so it can be flowed back easily. Because recovery always ranges between 30% to 50%, it is never clear how much fluid was broken[13]. As a backup analysis, tests were run to see the interactions between the viscous crosslinker and the different chemicals pumped in the fracture. The tests were conducted under reservoir conditions. The goal was to observe how long it would take the fracture fluid to break down after being exposed to all the chemicals in addition to the internal breaker.

X-Ray Diffraction (XRD)

X-ray diffraction (XRD) was used on core samples to gain knowledge about the mineralogy of the reservoir rock. The samples were crushed to fine powder using a mill. The clay-size material – <2 microns equivalent spherical diameter – was separated from the larger size particles by sedimentation techniques. The clay size fractions of the sample were then dried using air on a glass slide. The air-dried glass slide was glycolated in a desiccator containing ethylene glycol at 60 °C in the oven. The core samples were then analyzed by XRD, which included

Plug	Calcite	Ankerite	Illite + IS	Kaolinite	Quartz	Pyrite	Grain Density
#	wt%	wt%	wt%	wt%	wt%	wt%	g/cm³
Well-88							
2	68	18	7	3	3	1	2.75
49	82	11	3	2	1	1	2.75
Well-37							
813H*	89.7	9	0.2	0.8	0.3	-	2.75
815H	98.9	0.9	-	0.2	-	-	2.75
829H	97.3	1.2	0.2	1.2	0.1	-	2.75
803H	96	4	Trace	Trace	-	-	2.75
837H	98	2	Trace	Trace	-	-	2.75
*Microline - Trace							

Table 2. XRD results after analyzing the core plugs from the target reservoir

the identification of the crystalline phases. Subsequent semi-quantification of the XRD data was done using the Rietveld refinement method.

Environmental Scanning Electron Microscope (ESEM)
Microstructural characterizations, in terms of porosity, pore size, and the presence of clay and foreign materials in the pores, are important in understanding the behavior of reservoirs. Such characterizations also help in selecting the right acidizing treatment for the formation. We employed environmental scanning electron microscopy (ESEM) analytical techniques, used with an integrated, ultra-thin window, energy dispersive X-ray spectrometry (EDS) detector, to perform comprehensive microstructural characterizations of the core samples in this reservoir. The ESEM/EDS data identified the minerals in the core, any materials blocking the pore space, the type of cementing materials present and also the elemental compositions of the plug samples.

In addition, ESEM assesses any formation damage that may have been caused by the chemical treatments, leading to a reduction in productivity. The primary goal of this test in the study was to identify the main components of the reservoir core samples and correlate them with the XRD results to have a better understanding of the acid/rock reactions during the multistage acid fracturing job. Furthermore, a second set of cores that had been previously exposed to a damaging drilling mud filtrate were tested with ESEM to show its measurement of the magnitude of the clay swelling damage on the core pore throats.

Rock Solubility
Rock solubility is very important to know in a multistage acid fracture operation because the whole mechanism of this stimulation treatment relies on acid reactions. We ran several tests by dissolving core plugs in the different HCl acid recipes (emulsified acid, gelled acid and straight HCl acid) and concentrations being used on-site. The goal of this test was to measure the amount of rock that would be dissolved before flowing back the fluids. From this test we could decide whether the operation time would be sufficient to spend the acid, and therefore, protect the tubular from corrosion while flowing back. This test was conducted under reservoir conditions, and graphs were generated to illustrate the solubility capabilities of the acid system with the reservoir rock.

Coreflood
A coreflood apparatus was designed and built to simulate fluid flow in porous media in the reservoir. A positive displacement pump equipped with a programmable controller was used to deliver fluids at constant flow rates at variable speeds up to 200 cm³/min and pressure up to 10,000 psi. The pump is connected to two accumulators to deliver brine or chemical solutions. Accumulators with floating pistons rated up to 3,000 psi and 250 °F were used to store and deliver fluids. A set of valves was used to control the injection of the fluid into the core sample. The core holder can accommodate a core plug with a diameter of 1½" and a length up to 3". Pressure transducers were used to measure the pressure drop across the core. A back pressure regulator was used to control the flowing pressure downstream of the core.
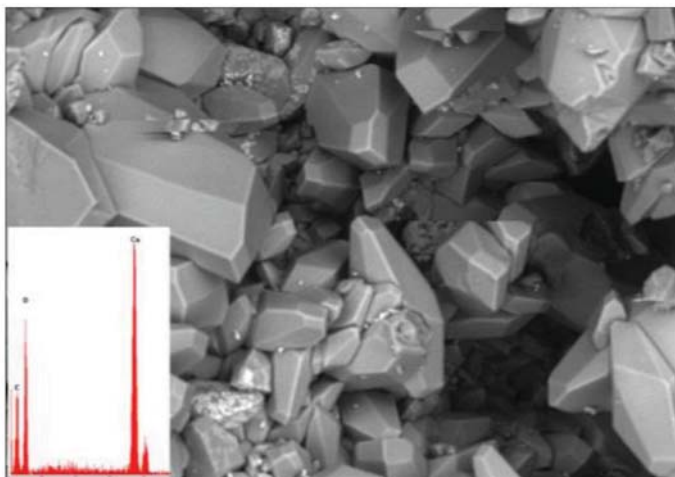
Fig. 6. ESEM for the core sample taken from the formation rock indicates abundant calcite.
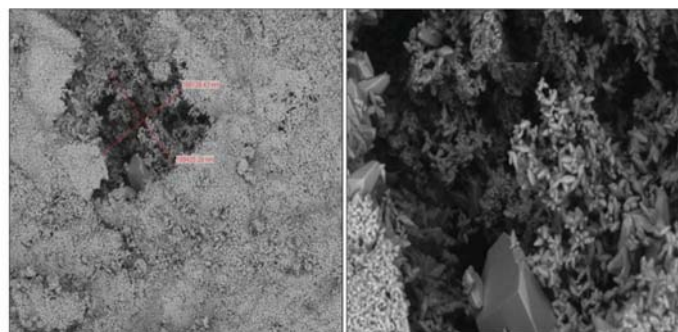

Fig. 7. ESEM shows possible damage caused by clay swelling indicating the importance of a clay control chemical (left): ESEM pore throat picture shows the possible clay damage due to not using a clay control agent, thereby hindering the flow back (right).
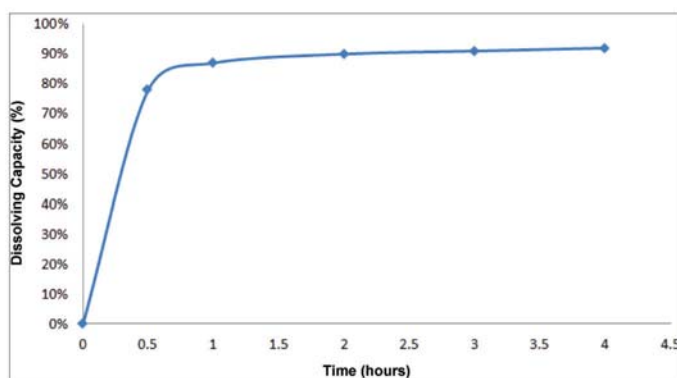
Fig. 8. Emulsified acid dissolving capacity and solubility test.

A second back pressure regulator was used to control the confining pressures on the core plug. A convection oven was used to provide a temperature controlled environment. A data acquisition system was used to collect data from the pressure transducers.

Below is the procedure used to prepare the core for the coreflooding experiments:

• The core samples were dried overnight in a 100°C oven.
• The core samples were loaded into the core holder and confining pressure was applied.
• A 6% potassium chloride (KCl) brine was injected in the core to establish ~100% fluid saturation.
• The base permeability to water was measured using different flow rates (0.5 cm³/min and 1 cm³/min).
• An emulsified acid was pumped to create wormholes in the core.

Computed Tomography (CT) Scan

An X-ray computed tomography (CT) scan was used on the cores after running the coreflood experiments. By scanning the cores, we were able to determine the density distribution and therefore track the wormhole propagation created after flooding the cores with the different HCl acid recipes. In addition, this type of test would help in understanding the distribution of minerals in the rock and how the acid would react with the rock. From this analysis we could also anticipate the type of ions to expect from the reactions in our flow back water analysis. The results were then processed to achieve a two-color scale to magnify the wormhole propagation in the core.

Flow Back Analysis

Fluid samples were collected and concentrations of key ions were analyzed. Ca, Mg, Fe, Cr, Ni and Mo concentrations were measured using inductively coupled argon plasma emission spectroscopy (ICP)[14]. Cl ions were measured by titration with a 0.1 N silver nitrate solution using an auto-titrator. To measure the pH, a pH meter was used.

Results and Discussion
Emulsified Acid Stability

Figure 3 shows the emulsified acid stability test results.

Fig. 9. The emulsified acid core flooding test shows that the emulsified acid's wormhole needed less than 1 PV to break through.

Fig. 10. Core sample CT scan, after flooding the core with emulsified acid, to characterize the wormhole.
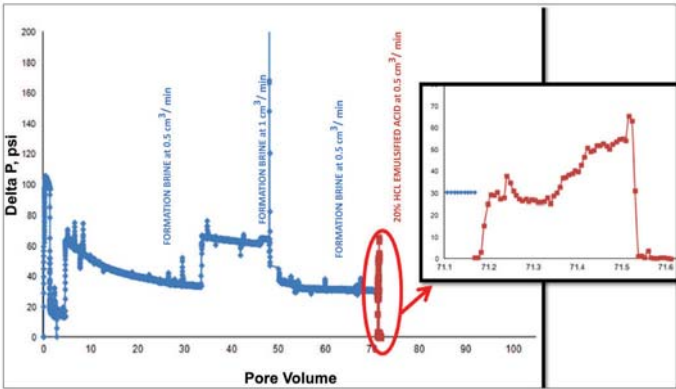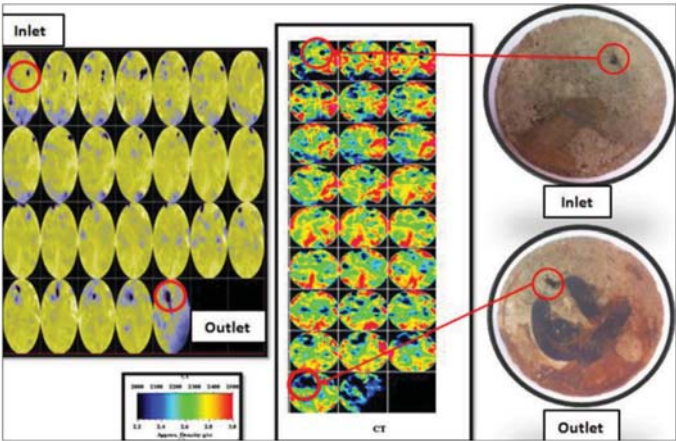
The results indicate that the emulsified acid is going to break into three layers after 75 minutes at room temperature following its reaction with the core. The main two layers are diesel and HCl acid, while the third layer is a portion of the emulsified acid that has not separated completely. This third layer could be a result of fines stabilizing the emulsion and could cause some reactions in unwanted areas of the completion/reservoir.

The same test was then conducted under reservoir conditions, and the results show a clear two layer separation of diesel and spent acid after an incubation time of 30 minutes. These two layers can easily flow back without issue during the flow back operation. The Ca and Mg ions are the major ions available in the acid portion of the emulsified acid after it separates. If the acid is not totally spent due to a too short soaking time, the separation of acid from the emulsion will increase the corrosion rate of the tubulars.

Viscosity of Cross-linked Pad

Figure 4 shows the effect of the internal breaker concentration on the viscosity of the cross-linked gel. The test was run for the following concentrations of internal breaker: 0.1 gpt, 0.25 gpt and 0.5 gpt. The results indicate that increasing the concentration of the internal breaker will reduce the gel breaking time from 90 to 20 minutes, which will also result in reducing the gel viscosity below 500 centipoise at reservoir temperature. The flow back operation was planned to start after 1 week, giving the breakers enough time to react and break the cross-linked gel for fracture cleanup.

Since the treatment plan specified that the acid stage be

Parameter	Ca	Mg	Cl	Fe	Mn	K	Na	Sr	SO$_4$
Mixing Water	217	55	271	5	0	15	171	3	622
Formation Water	37,000	6,700	122,500	30	0	1,000	34,000	1,400	480

Table 3. Comparison of ions found in mixing water vs. formation water

Parameter	Ca	Mg	Cl	Fe	Mn	K	Na	Sr	SO$_4$
Calcite/Ankerite	✓	✓		✓					
Chemicals/Mixing Water	✓	✓	✓			✓	✓		✓
Formation Water	✓	✓	✓	✓		✓	✓	✓	✓
Tubular				✓	✓				

Table 4. Main ions in flow back water
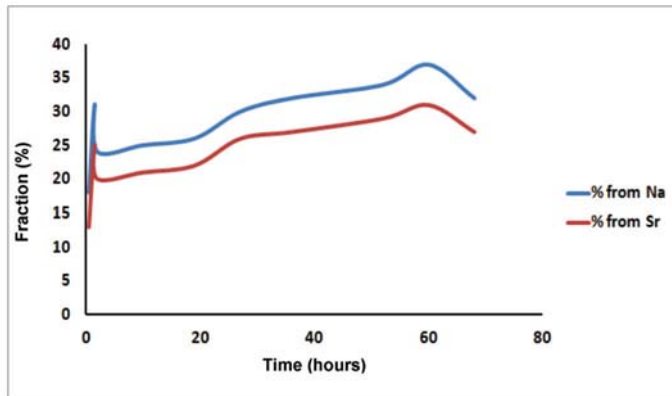
Fig. 11. Ratio of formation water in the flow back water using Na and Sr ions as base comparison.

Fig. 12. Portion of Ca ions generated from the formation rock reactions and from the formation water mixing with the flow back water.

Fig. 13. Portion of Mg ions generated from the formation rock reactions and from the formation water mixing with the flow back water.

Fig. 14. Ca and Mg trends are similar and decreasing toward the end time indicating that they are generating from the same source, which is the formation rock's reactions.

injected after each cross-linked pad, the effect of mixing the acid system with cross-linked gel was examined. Figure 5 shows the effect of mixing the cross-linked pad (without breaker) and emulsified acid at a 1:1 ratio. The results indicate that the emulsified acid was able to break down the viscosity and so would allow easier cleanup of the fracture. Many field operations have trouble flowing back the cross-linked fluid because it doesn't break down completely even though it has adequate internal breaker concentrations, so these results were comforting. Given the hard to remove filter cake generated on the fracture face, the results showing the acid breaking down the cross-linked fluid will definitely help in flowing back the fracture fluid more easily.

Mineralogical and Rock Analysis

The XRD results showed that the samples consisted of carbonate minerals (calcite and ankerite), with minor quantities of clay minerals (kaolinite, I-S and illite), iron sulfide (pyrite) and sand (quartz) in some of the samples. Table 2 gives the bulk mineralogical composition of seven core plugs from this reservoir. The data showed that calcite was the most dominant mineral in the samples, with wt% ranges between 68 and 99. The next two most dominant minerals were ankerite and dolomite, with 1 wt% to 18 wt%. Clay minerals were detected in some of the cores and reached up to 14 wt% in the extreme case, but were absent in other core plugs. Total clays of mixed layers illite-smectite, illite and kaolinite were also detected in all the samples. A minor quantity of potassium iron sulfide was also found in the samples.

Figure 6 shows the ESEM image confirming the presence of calcium carbonate ($CaCO_3$), mainly in the reservoir core sam- ple. From this analysis we could safely say that $CaCO_3$ would be the main component to react with the HCl acid, and we expected a large amount of calcite reaction product ions in the flow back water
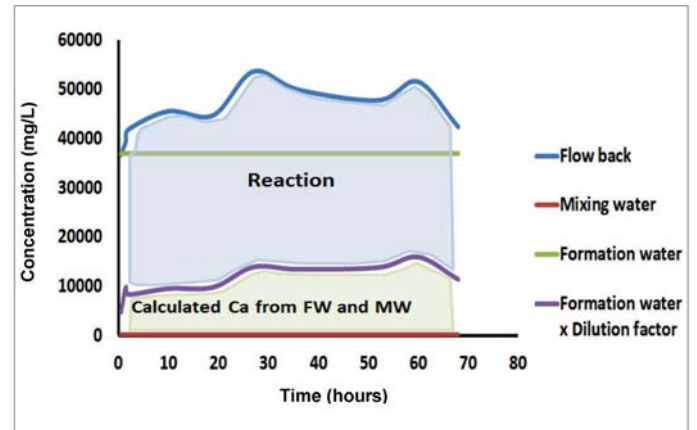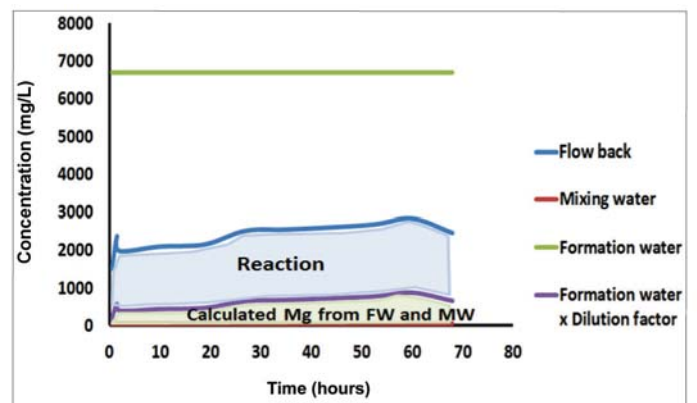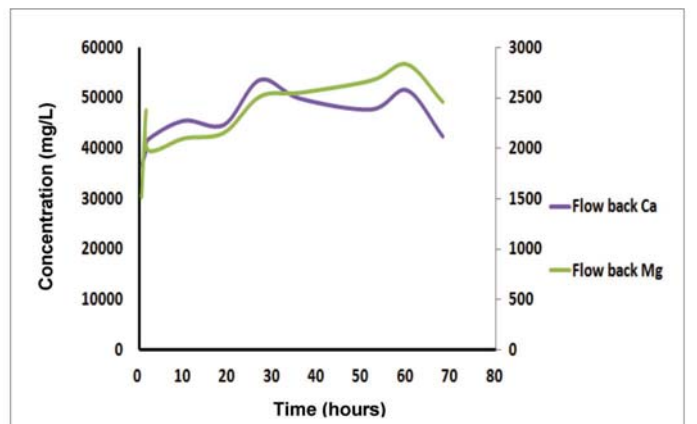
analysis. Figure 7 shows the distribution of clays on the pore throat in the reservoir core sample. This indicates the importance of using a clay control agent in this multistage acid fracture job to protect the pore throats from plugging, thereby blocking both flow back and production.

Rock/Acid Interaction

Figure 8 shows the results of rock solubility vs. time.

Fig. 15. Flow back water pH analysis indicates the presence of spent acid only.

Fig. 16. Portion of total Fe ions generated from the ankerite reactions/corrosion and from or the formation water mixing with the flow back water.
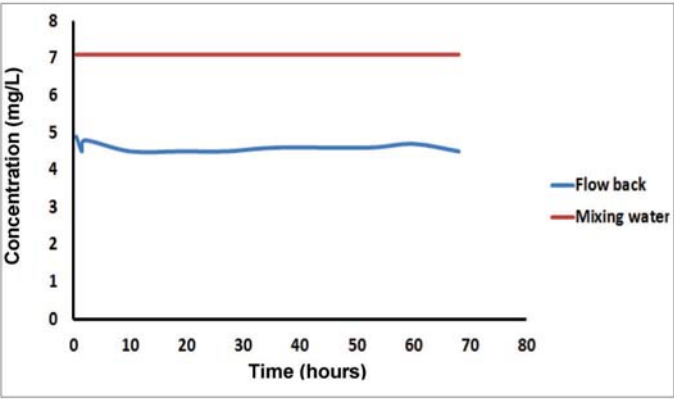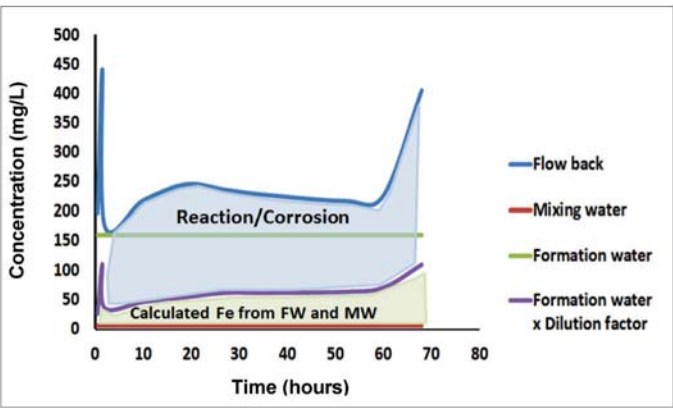
The results indicate that the emulsified acid was able to dissolve around 90% of the core in 1 hour with no precipitation. After 2 hours, the graph starts to plateau, which indicates that no further reaction was happening and the acid was spent. This result can be used to design the field operation and recommend the minimum required soaking time for acid to be spent, preventing the flow back of live acid. These results indicate that we should expect Ca and Mg from the calcite and dolomite reactions in the flow back analysis. Moreover, according to the XRD analysis, Mn and Fe were also expected, due to the presence of ankerite in the core sample.

Figure 9 shows a pressure drop as a function of the injected pore volumes (PVs) of brine and emulsified acid in the core- flooding experiment using core samples from the target reservoir. The figure shows the initial pressure drop resulting from pumping the brine. The figure also shows the pressure drop resulting from pumping the emulsified acid. The emulsified acid was more viscous than the brine, so it resulted in a higher pressure drop while the rate parameter remained constant. The results also indicate that the amount of emulsified acid required to generate a wormhole and break through the core was less than 0.5 PVs.

Figure 10 shows the CT scan results. The results indicate that the minerals are distributed all over the core with no specific pattern, making it a heterogeneous sample of the reservoir. The wormhole generated from the emulsified acid is shown favorably dissolving the calcite minerals compared to the dolomite and ankerite. This will promote a differential etching phenomenon due to mineralogical difference, which is a favorable attribute for effective fracture conductivity. In addition, there was no face dissolution observed on the core after pumping the emulsified acid.

Flow Back Analysis

According to the rock mineralogy determined by XRD and ESEM analysis of the core samples from the LF formation, we expected the acid-rock interactions to yield Ca and Mg mainly, with some Fe and Mn[14]. Table 3 shows a summary of the ions found in the mixing and formation brine, while Table 4 shows the expected ions in the flow back analysis.

Dilution Effect of Mixing Formation Brine with Stimulation Mixing Water

To calculate the mixing coefficient, A, which results from mixing formation brine and the water used in mixing the stimulation fluid, Eqn. 4 was used:

$$\text{ClFlo} = A \times \text{ClFor} + (1 - A) \times \text{ClMix} \qquad (4)$$

where:

CIFlo = Concentration of ions in the flow back water
CIFor = Concentration of ions in the formation brine
CIMix = Concentration of ions in the mixing water

The key ions to apply to this equation are ions that exist already in the formation brine or mixing water and are not a byproduct of treatment fluid reactions with the formation rock/wellbore materials. Since the formation brine was the only source for strontium (Sr), that element was used to calculate the dilution coefficient for the other ions. Figure 11 shows the dilution coefficient as a function of time that was calculated from the Sr concentrations.

Reaction Products in Flow Back Samples

Figure 12 shows the Ca concentration in the flow back water as a function of time. The amount of Ca in the flow back water was much higher than the Ca in both
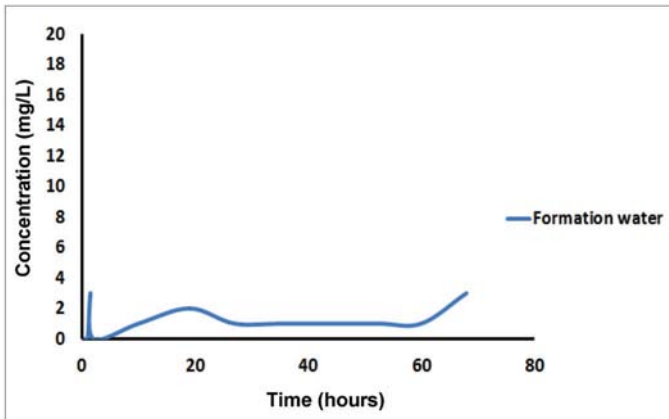
Fig. 17. Flow back water Mn ion analysis confirms minimal corrosion.

Fig. 18. Fe and Mn trends are similar at the end but different from the Ca and Mg trends, indicating that they are generated from a different source.

Fig. 19. Cr, Ni and Mo concentrations are very low, which indicates that corrosion of the tubular didn't occur during this multistage acid fracture job.

the mixing water and the formation water. This is an indication that the desired reaction between the HCl acid and the calcite in the formation rock occurred. Figure 12 also shows the ratio of Ca in the flow back water that came from the formation water vs. from the reaction of the HCl acid with the formation. The results indicate around 10,000 mg/L Ca in the flow back water coming from the formation water and 40,000 mg/L as a result of the HCl acid reacting with the reservoir rock. This large amount of Ca from the reaction was expected according to our dissolving test results in the lab section.

Figure 13 shows the Mg concentration in the flow back water analysis in comparison to Mg amounts in the mixing water and formation water. The amount of Mg in the flow back water is higher than the Mg in the mixing water but lower than the Mg in the formation water. This can be an indication that the reaction between the HCl acid and the dolomite in the formation rock has occurred. Applying the dilution factor to find the calculated Mg concentration can help differentiate the source of Mg ions. Figure 13 also shows the ratio of Mg in the flow back water that came from the formation water vs. from the reaction of HCl acid with the formation rock in the reservoir. The results indicate around 800 mg/L of Mg in the flow back water coming from the formation water and 2,200 mg/L as a result of the HCl acid reacting with the reservoir rock. This large amount of Mg from the reaction was expected according to our dissolving test results in the lab section.

Figure 14 shows the trends for both Ca and Mg that indicate they were generated from the same source, due to similar slopes at the larger time values. In this case our source was the formation rock. The downslope showing the trend decreasing toward the end of the flow back operation indicates less reaction with the formation rock.
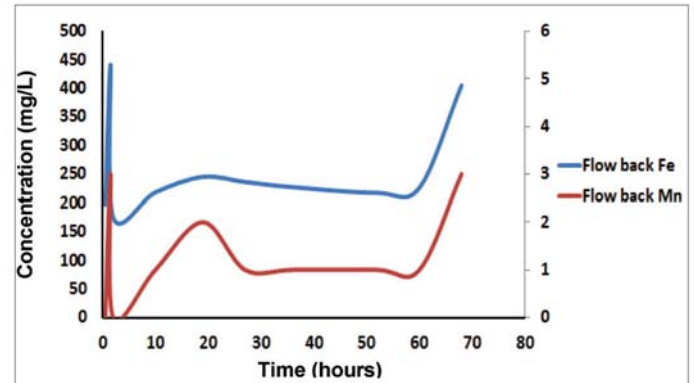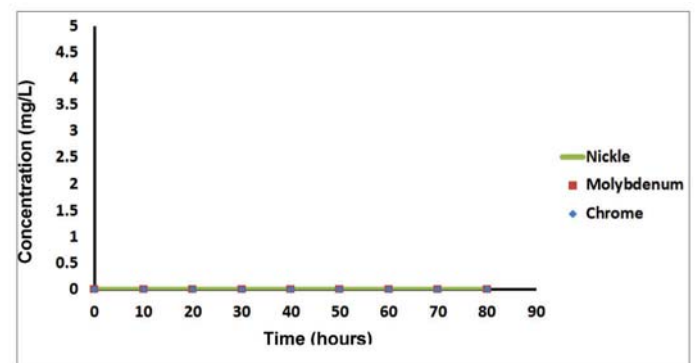
Figure 15 shows the pH value as a function of time in the flow back analysis in comparison to pH values in the mixing water. The pH value in the flow back water is 5 compared to a pH value of 7 in the mixing water. The reason behind that is the reaction of HCl acid with the formation rock:

$$H_2O + CO_2 \longrightarrow H_2CO_3 \qquad (5)$$

The reaction equation yields water (H_2O) and carbon dioxide (CO_2), and therefore forms carbonic acid, which lowers the pH values of spent HCl acid to around a pH of 4.5 to 5. This is critical to know because at this pH the acid is fully spent and ready for flow back. A lower pH value would indicate that the flow back water is going to be acidic and will require a good corrosion inhibitor package to protect the tubular during the flow back operation.

Corrosion Products in Flow Back Samples

Figure 16 shows the Fe concentration in the flow back analysis as a function of time. The amount of Fe in the flow back water is higher than both the Fe in the mixing

Fig. 20. Flow back water Sr ion analysis confirms possibility of formation water in the flow back.
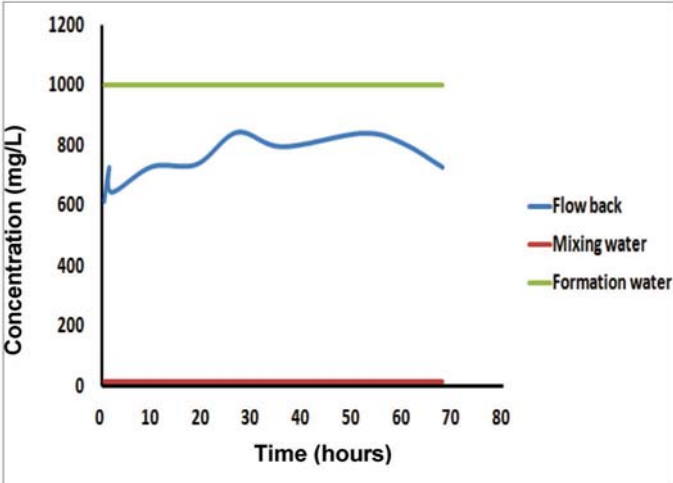
Fig. 21. Flow back water K ion analysis indicates the possibility of formation water in the flow back.

water and the Fe in the formation water. Fe ions in the flow back samples can be generated from two sources: as a reaction byproduct of ankerite with acid or as a result of corrosion to wellbore tubular. To confirm the presence of corrosion, an analysis of Mn ions and Cr ions was required. Figure 16 also shows the ratio of Fe in the flow back water that resulted from the formation water and from the reaction of HCl acid with the formation ankerite rocks or from the corrosion of the tubular due to the reaction with HCl acid. The results indicate around 100 mg/L of Fe in the flow back water coming from the formation water and 300 mg/L as a result of the HCl acid either reacting with the ankerite in the reservoir rock or causing minor corrosion to the tubular. This small amount of Fe is an indication of only minor corrosion in the tubular.

Figure 17 shows the Mn concentration in the flow back water analysis. The flow back water analysis showed some returns of Mn ions, but the concentrations were very low. This could be an indication of minimal corrosion. In addition, Mn ions could be coming from the ankerite's reaction with the HCl acid. Figure 18 shows that Fe and Mn ions followed the same trend, increasing at the very end of the flow back operation. This is an indication that both Fe and Mn ions originated from the same source; however, this source was not the same as the Ca and Mg ions' source.

Analysis of the Cr, Ni and Mo in the flow back samples was conducted to confirm the presence of corrosion. The ions targeted by this analysis are part of the construction material of the tubular. Figure 19 shows concentrations of Cr, Ni and Mo as a function of flowing time. There was no indication of these ions in the flow back samples: they were all below the detection limit of 1 mg/L. This
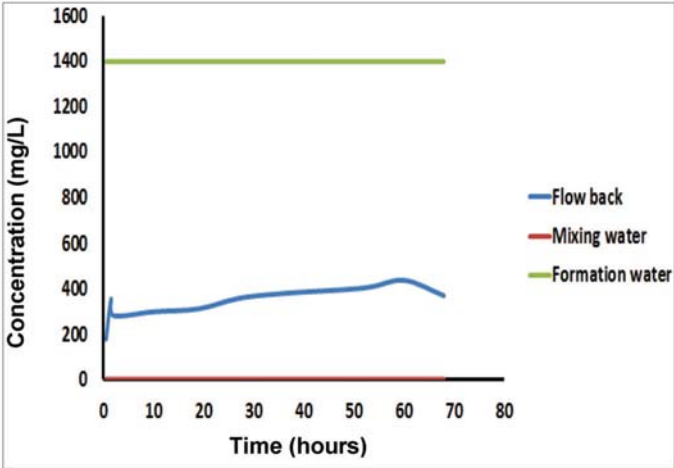

Fig. 22. Flow back water Cl ion analysis indicates possibility of formation water in the flow back.

Fig. 23. Flow back water Na ion analysis indicates possibility of formation water in the flow back.
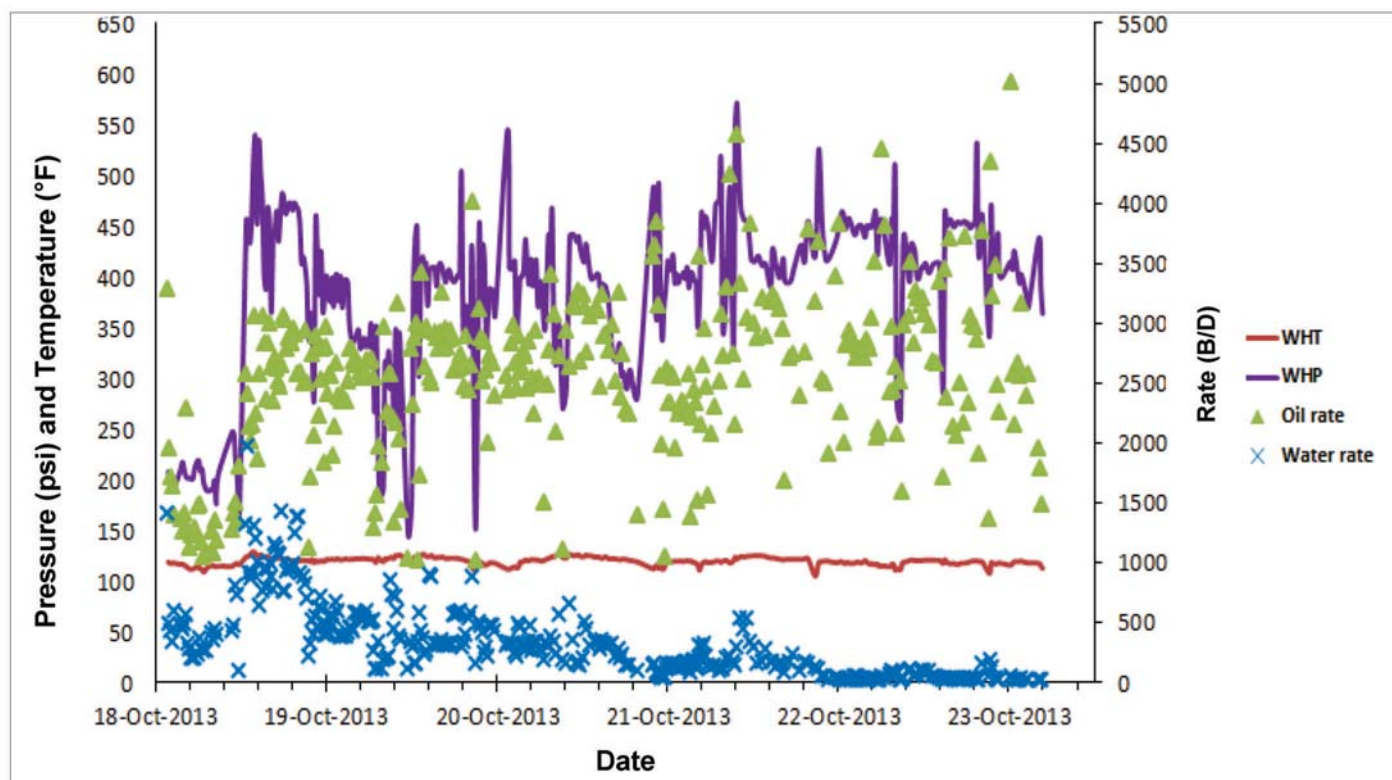
Fig. 24. Oil and water flow back rates during cleanout.

Parameter	Stage 1	Stage 2	Stage 3	Stage 4	Stage 5	Stage 6	Stage 7
Treated Water	13,896	0	11,138	10,206	10,212	10,221	9,341
Pad	19,192	0	18,566	19,161	18,981	19,116	19,204
CEA	17,247	0	17,807	17,985	17,985	17,239	17,851
CSA	14,590	0	17,145	17,516	26,981	18,175	18,050
CFA	6,199	0	4,249	4,932	5,002	4,875	4,506
Diverter	4,070	0	3,964	4,035	4,044	4,113	3,830
Pumped Volume (gal)	75,194	0	72,869	73,835	83,205	73,739	72,782
Total (gal)	451,624						
Recovered Water (gal)	77,584						
Recovered (%)	17%						

Table 5. Pumped chemicals

finding eliminates the possibility of corrosion in the completion hardware.

Formation Water Ions

Figure 20 shows the Sr concentration as a function of time. The amount of Sr in the flow back water is higher than in the mixing water but lower than in the formation brine. This indicates that the formation brine (KCl) is part of the flow back fluid.

Figures 21 and 22 show K and Cl concentrations as a function of flow back time. The presence of K and Cl ions is usually helpful in confirming the percent of formation water in the flow back water; however, fluids containing KCl and HCl acid were pumped during this job and that will mislead the calculation of the formation water percentage.

Figure 23 shows the sodium (Na) concentration in the flow back water analysis in comparison to Na amounts in the mixing water and formation water. Because Na was one of the ions that was not included in our pumped chemicals, the amount of Na in the flow back water is a clear indication of formation water in our flow back water.

The dilution factor was then calculated by dividing the flow back water's Na amount by the formation water's Na amount at different times of the flow back. This yielded 25% to 30% of formation water in the flow back water at a late time, which is consistent with the Sr analysis previously mentioned.

Flow Back Rates

The main objective of flow back is to ensure maximum treatment fluid returns and to signal a successful operation. The capability of a well to flow is a major indicator of success; however, in this particular job, field designs for flow require artificial lift for pressure support. The well was designed to flow using an electric submersible pump and expected to flow at a rate of 2 thousand barrels of oil per day (MBOPD). The flow rate results from the flow back observed following treatment were very promising. The well was able to flow for 8 hours before the rate was deemed too low for treatment fluid recovery. After that, the well was artificially lifted using nitrogen pumped through coiled tubing at the bottom. The flow back showed that the well was producing at an average rate of around 3.5 MBOPD, Fig. 24. In addition, we were able to recover 17% of the treated chemicals pumped, as shown in Table 5. Further water analysis indicated the presence of 25% to 30% formation water while the critical ion analyses showed

the effectiveness of the corrosion inhibitor package, acid system dissolving capacity, and crosslinker fluid recovery. Also, it is expected that for future jobs, optimization will be conducted in regards to increasing the percentage of treatment fluid recovered.

Conclusions

Based on the results from experimental work and flow back analyses, the following conclusions can be drawn:

• The viscosity of the cross-linked gel was reduced by the internal breaker and the gel's interaction with the injected acid stages.

• Emulsified acid was efficient in generating wormholes with less than 1 PV of injection in the coreflooding experiment conducted on cores from the LF formation.

• Propagation of wormholes in the core samples is sensitive to rock mineralogy, as shown in the CT scan images, due to the different reaction rate of acid with various carbonate minerals.

• The flowed back acid was fully spent due to the extended soaking time and proper displacement.

• There was no indication of corrosion to the downhole completion in the flow back analysis results.

Acknowledgements

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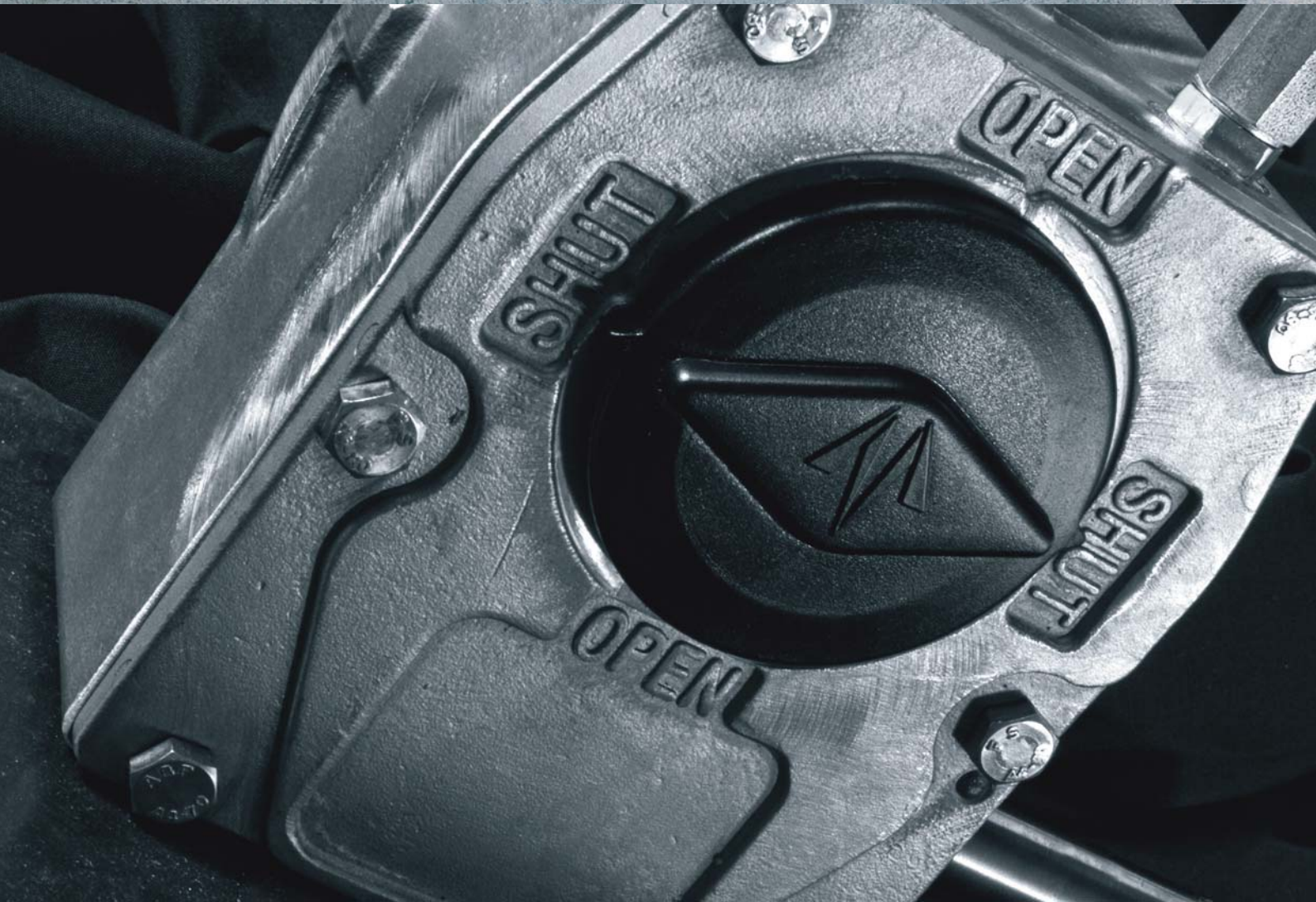
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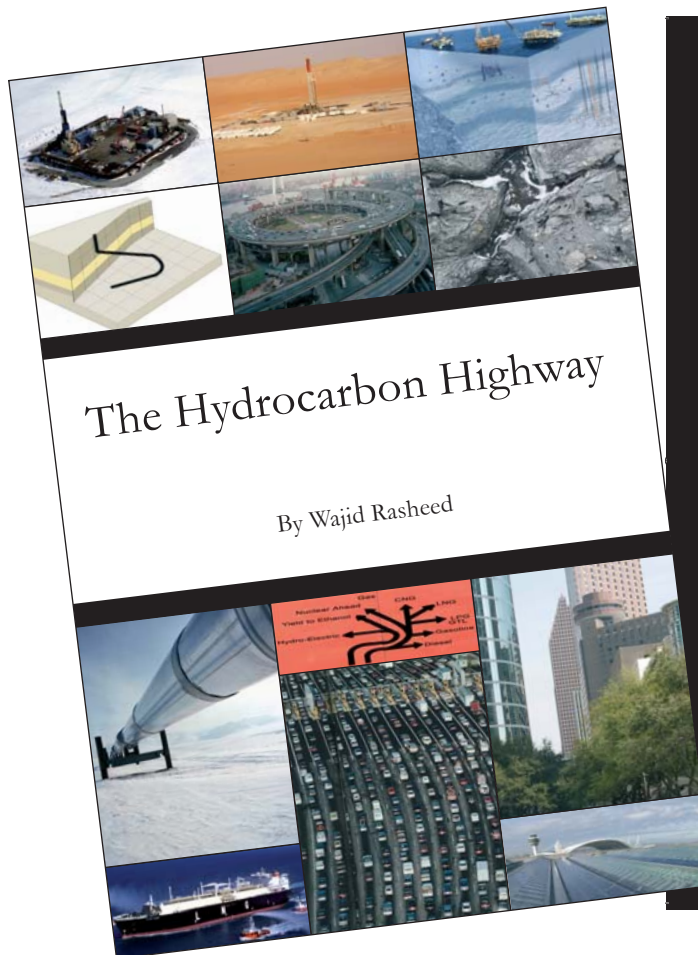
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Dr AbdulAziz Al Majed, the Director of the Centre for Petroleum and Minerals at the Research Institute at King Fahd University of Petroleum and Minerals

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This chapter reveals how oil and gas asset production can be maximised through an understanding of petroleum and reservoir types.

When crude oil first came into large-scale commercial use in the 19th century, it was stored and shipped in wooden barrels with one barrel equal to 42 US gallons or 159 litres. The term 'wet' barrel denotes a physical barrel of oil that is actually delivered or consumed as opposed to a futures or other paper barrel that is traded.

Asphalt, bitumen and crude are common terms describing different forms of petroleum that can be found in a typical 'barrel' of oil[1].

The term comes from the Latin petra – "rock" and oleum – "oil". For lay people, petroleum itself is

**PRODUCT GALLONS
PER BARREL**

- Gasoline — 19.4

- Distillate Fuel Oil — 10.5

- Kerosene-Type Jet Fuel — 4.1

- Coke — 2.2

- Residual Fuel Oil — 1.7

- Liquefied Refinery Gases — 1.5

- Still Gas — 1.8

- Asphalt and Road Oil — 1.4

- Raw Material for Petrochemicals — 1.1

- Lubricants — 0.4

- Kerosene — 0.2

- Other — 0.4
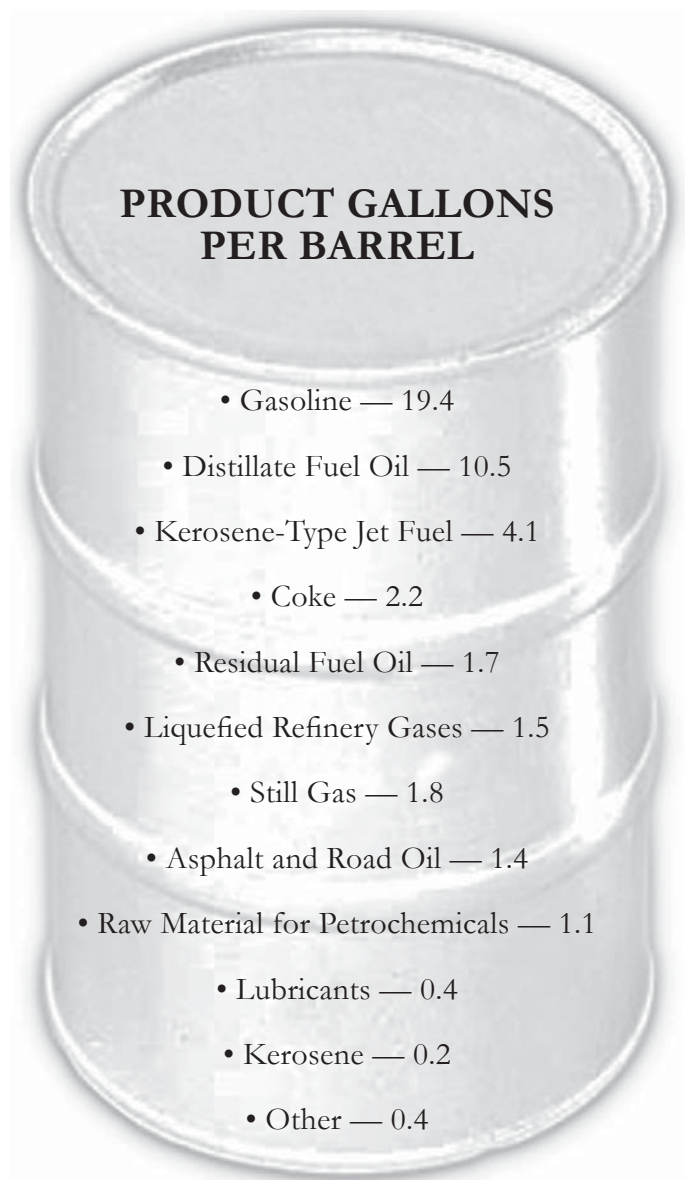
Table 1 - Products Per Barrel of Oil (in Gallons).

Note: Distillates includes both home heating oil and diesel fuel. Residual fuel oil refers to heavy oils used as fuels in industry, marine transportation, and for electric power generation. Figures are based on average yields for U.S. refineries in 2005. One barrel contains 42 gallons of crude oil. The total volume of products made is 2.7 gallons greater than the original 42 gallons of crude oil. This represents 'processing gain.' (After API)

a generic term that covers all naturally occurring hydrocarbons as well as refined products or derivatives.

For purists, however, petroleum refers to chemical compounds made up of hydrogen and carbon atoms; consequently, the classification hydrocarbon is more appropriate. Definitions aside, hydrocarbons in their 'un-produced' state are found in underground accumulations or reservoirs of oils, gases, water and impurities located at depths ranging from 2,000 ft (610 m) to 25,000 ft (7620 m). Petroleum naturally seeps to the earth's surface along faults and cracks in rocks gathering in tar, asphalt, pitch or bitumen lakes. Shortly, we will consider the make-up of reservoirs but first of all, what's in a barrel of oil?

Figure 1 - Nature's Best Is Sweet and Light (EPRasheed)

Figure 2 - Heavy Oil Is Unable to Flow at Atmospheric Conditions (EPRasheed)

Nature's best orange juice is sweet and light, as is its crude; however, not all of the 200 or so naturally occurring varietals of crude oil are so blessed and this affects their commercialisation. Sweet crude has less than 0.5% sulphur content – increase this figure and it turns 'sour'. Light crude has a density of 20° or more using the American Petroleum Institute's (API) specific gravity scale and has light hydrocarbon fractions. Heavy crude has more complex fractions with higher densities and lower API gravities[2].

The Colour of Oil

Generally speaking, the colour of crude oil intensifies with its density and viscosity. While black oil is hard-to-pour and has high density and viscosity, green to yellow oils are runny and have low density and viscosity. The term 'crude' refers to petroleum straight from the wellhead in its 'unrefined' state that can generally flow in atmospheric conditions. Where petroleum is unable to flow in atmospheric conditions, it is often referred to as heavy oil, tar or bitumen[3].

Technologists quibble on when crude gets heavy; some say this happens at 25°API or less and others say 20°API or less. This is important because heavy oil trades below its lighter counterpart. For our purposes, the definition of heavy oil is 20° API or lower and further detail is found in *Chapter 8: Extreme E & P*. Finding heavy or light crude oil depends entirely on the presence of cap rock and permeability, as these will prevent or permit oil and gas to leak to the surface and be dispersed. In Venezuela's Orinoco Belt, for example, heavy oil deposits are found close to the surface with the lighter fractions of oil having migrated or dispersed over the years, leaving only the heavier residue.

Sour as a Skunk

Sour crude with its high sulphur content sells below its sweet counterpart – the gap can be US $5 or more and is likely to increase in the future. The gap exists because sour crude requires specialised refining treatment before it can be sold; however, there are more sweet than sour refineries worldwide. Consequently, a refining preference for sweeter crude exists[4].

The naturally occurring sulphur compounds or 'mercaptans' present in sour crude are powerfully smelly and are also found in garlic oils and skunk secretions. The malodorous mercaptans are by-products of decaying organic matter and they must be treated which adds to refining costs; however, mercaptans have a market value. They are used to imbue an odour to commercial natural gas so the general public can easily be alerted to a gas leak. Untreated natural gas is odourless, and without the tell-tale smell of the mercaptan additive, the public could be unaware of a gas leak until it was too late and someone was asphyxiated or an explosion occurred. Getting rid of sulphur, water, chlorides and other such impurities improves quality, increases value and stretches the world's oil reserves but it also adds to cost.

Table 2 shows a series of oil and gas compounds and their respective molecular weights and common names ranging from methane gas (CH_4), petrol (C_5H_{12} to C_7H_{16}) to asphaltene ($C_{80}H_{160}$). Many characteristics such as density, viscosity and flammability are determined by molecular weights and greater detail is available in *Chapter 11: Refining*.

The range of oil varietals is illustrated by extremely light oil, which has a relative density and viscosity below that of water, to extremely heavy oil which has a relative density close to that of water and high viscosity that can be 100 to 100,000 times that of water.

Molecular Weight

Typically, oil has a carbon content of 84 to 87% weight and a hydrogen content of 11 to 14% by weight[6].

Component	Boiling Point °C	Black Oil	Volatile Oil	Gas Condensate	Wet Gas	Dry Gas
Methane, CH_4	-161	49.0	64.0	86.0	87	96.0
Ethane, C_2H_6	-88	2.8	8.0	4.4	5	2.8
Propane, C_3H_8	-42	1.8	4.5	2.4	5	0.3
n-Butane, C_4H_{10}	-1	0.8	2.0	0.8	0.6	0.2
i-Butane, C_4H_{10}	-11	0.8	2.1	1.0	0.6	0.2
n-Pentane, C_5H_{12}	36	0.7	1.5	0.3	0.5	0.1
i-Pentane, C_5H_{12}	27	0.5	1.5	0.5	0.5	0.1
n-Hexane, C_6H_{14}	69	1.6	1.4	0.6	0.3	0.1
Colour of liquid at surface		black	brown	straw	white	-
Liquid Specific Gravity		0.853	0.779	0.736	0.758	none
°API		20-35	38-50	50-70	50-70	none
GOR scf/bbl		50-1500	2000-40,000	3000-18,000	>100,000	none

Table 2 - Crude Oil and Natural Gas Varietals, After Professor Richard A. Dawe

Table 2 shows that hydrocarbons exist with varying densities and viscosities. Viscosity is a prime determinant of the ability to produce and refine oil. It is worth noting that the number of hydrocarbon compounds increases dramatically due to isomers, differing arrangements of the same number of atoms. In the case of hexane (C_6) there are five isomers, for decane (C_{10}) there are 75 and for C_{30} there are more than four billion. Although laboratory analyses of reservoir hydrocarbons can profile all compounds containing as many as 20 carbon atoms, it is usually sufficient to profile compounds containing up to six or seven atoms, with a general number being used to represent the total proportion of heavier molecules that are present[7].

The general trends depend on the ratios of methane (CH_4) and the heavier components. The intermediates, C_2-C_6, control the GOR and API grade. The percentages shown are representative only and each category can be considered as flexible. Additionally, once production starts from a reservoir, the state of equilibrium that has been established over geological time is destroyed. Pressure gradients are created and the chemical composition and the physical properties of the fluids in the reservoir change. This happens as the pressure exerted on the fluids changes from the reservoir to the wellbore to the surface and, over time, as the fluids constituting the reservoir change.

Analysis is usually presented in terms of C_1, C_2, C_{n+} with n often being 7, 12 or 20. Compounds that are not expressed in this way are usually treated as a composite fraction characterised by a molecular weight, density and/or a boiling point.

For E & P purposes, physical properties such as colour, API grade, viscosity, bubble point pressure, Gas-Oil Ratio (GOR), pour-point, and kerosene content are characterised. For downstream purposes, actual hydrocarbon compositions and fraction descriptions are required. Traditionally, the analysis of produced fluids was performed in the laboratory and could take weeks or more to obtain. Nowadays, real-time formation testing tools can provide analysis of produced fluids in near real-time at the wellsite.

Saturated Oil

Produced oil will always contain a certain amount of dissolved gas. The exact amount depends on reservoir conditions such as temperature and pressure as well as the composition of the oil. If the oil cannot dissolve any more gas under the prevailing conditions, it is termed saturated; the excess gas has moved to the top of the reservoir and formed a gas cap. If the oil can dissolve more gas, it is termed undersaturated, and no gas cap will be initially present on production. The GOR is the ratio of the volume of gas produced to the volume of liquid and may be expressed as cubic feet per barrel depending on the units used for measuring gas and liquid. For gas wells, the inverse ratio is sometimes used and the liquid-gas ratio is expressed in barrels per million m^3 (or million cubic feet)[8].

Impurities

Reservoir characteristics depend on the interplay between the molecular arrangements of the hydrocarbons, the extent of liquid and gas phases as well as the existence of impurities. Aqueous impurities are caused by differing levels of salinity and mineral salts within water that were present within rock pores before hydrocarbons migrated into the reservoir rock, displacing a certain volume of this water. The volume
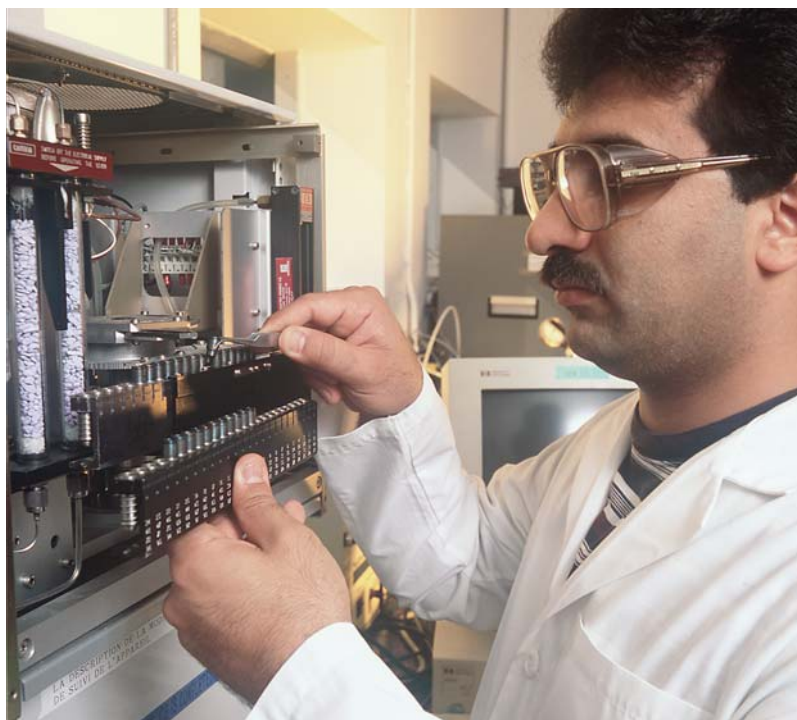
Figure 3 - Checking Crude Samples (Saudi Aramco)

of water that remains after migration is known as 'connate water' and it is common for large volumes of water to be produced in conjunction with oil and gas.

Water

Water is present at all stages of oil production. Connate water found in the reservoir at discovery can occupy 5 to 50 % of the pore volume and it is common for large volumes of water to be produced in conjunction with oil and gas (it is not always the case that a reservoir has reached maturity simply because it is producing water. See *Chapter 9: Mature Fields* – Water Management). It is also usually very salty, often more concentrated than seawater. Levels of water saturation can be accurately measured by well-logging, surface monitoring as well as permanent downhole monitors. Water breakthrough causes production problems including corrosion and scale, particularly as reservoir water often contains salts up to 250,000 mg/l, in comparison to sea water which contains 35,000 mg/l of salts.

Water and oil also create emulsions which are difficult to break and disposing of produced water can generate an environmental burden as it must be disposed of adequately. Further, any incompatibility between injected water and connate water can create chemical scale[9].

Water is nearly always present in gas reservoirs and reservoir gas is often substantially saturated with water vapour at the temperature at which it enters the wellbore. With the change in temperature and pressure from the subsurface to surface, the gas will not be able to hold as much water and it will condense both within the well during the upward travel of the gas and in surface equipment. Much of this condensed water is carried in the flow lines into the separator as entrained droplets. Water can form hydrates with natural gas, which can create production difficulties, rendering metres and valves inoperative and, on occasions, causing disasters. Low temperature separators are needed to remove the entrained water close to the wellhead before the gas arrives at trouble points. In many cases, appreciable amounts of water will settle to the bottom of the well and can, in time, saturate the zone surrounding the wellbore so that the permeability to the flow of gas may be materially reduced. This reduction can result either from water blocking or clay swelling and can be responsible for a gradual decrease in deliverability and periodic remedial work-overs[10].

Other impurities can be metallic such as vanadium or non-metallic such as hydrogen sulphide (H_2S). If there is any measurable sulphur content (more than one part per million), then the sulphur components, H_2S, can cause considerable damage to the production facilities unless they are designed to handle sulphur. The sulphur components are also poisonous to humans

hence lowering the commercial value of the oil or gas. They therefore have to be extracted, but can be converted to sulphur and sold on as a useful product. The production equipment has to use special quality steels to prevent rapid corrosion. Getting rid of sulphur, water, chlorides and other such impurities improves quality, increases value and stretches the world's oil reserves but it also adds to cost[11].

Releasing Hydrocarbons

The production of underground hydrocarbons is based on the release of trapped and pressurised fluids. Production involves a reduction in pressure and temperature from downhole reservoir conditions to atmospheric or surface conditions. As a result, hydrocarbons originally present as only liquid underground will separate into liquid and gas on their way to the surface, as soon as well pressure declines below the 'bubble point'.

In a mixture of liquids, the bubble point occurs when the first bubble of vapour is formed. For single component mixtures, the bubble point and dew point are the same and are referred to as the boiling point.

Hydrocarbons originally present as gas underground will generally produce some liquid at the surface due to condensation, which occurs when the pressure and temperature are reduced. The point at which natural gas components start to condense out of the gaseous system is known as the hydrocarbon dew-point and refers to the temperature (at a stated pressure) at which this occurs. Both bubble point and dewpoint are useful data when designing distillation refinery systems.

Surface facilities will mechanically separate gas from liquid using gravity separators or de-gassing facilities after which the volumes of liquid and gas are measured separately.

Gas

Natural gas volumes are reported in standard cubic metres [(s)m^3] or standard cubic feet (scf). Quantities of natural gas are usually expressed in cubic feet; a cubic foot is equivalent to approximately 0.028 m^3 at standard conditions[12]. For reserves valuation, gas is usually expressed in thousands (10^3) of cubic feet (Mcf), millions (10^6) of cubic feet (MMcf), billions (10^9) of cubic feet (BCF) or trillions (10^{12}) of cubic feet (TCF).

Methane is the most abundant component of natural gas and has numerous fuel applications. These range from liquefaction, compression, and Gas to Liquids (GTL). For further details, see *Chapter 13: Renewable Energy*. The second largest component is ethane which can be liquefied and sold as fuel, but is mostly used as a petrochemical feedstock. Propane and butane are also found in natural gas, albeit in smaller amounts, and are commonly separated and sold as Natural Gas Liquids (NGLs). This commercial value stems from their comparatively high-energy content. On a cubic foot basis, methane renders just over 1,000 Btu, while propane renders 2,500 Btu and butane 3,250 Btu[13].

Gas Condensate

Gas condensate or 'wet-gas' reservoirs are an important class of hydrocarbon accumulation and describe hydrocarbons which are gaseous in the underground reservoir. When the temperature and pressure of gas condensate are reduced to dew point, however, they partially condense to yield liquid condensate. Condensates are often characterised by low-density and high-API gravity (45° and above) and co-exist with natural gas.

Natural gas condensate is typically composed of pentane, hexane, heptane and octane. Liquids that condense are almost transparent or light yellow and can be refined in a way similar to very light crude oil[14].

Condensate-bearing reservoirs pose further production challenges due to the effect changes in reservoir pressure have on the hydrocarbons. Gas may be converted to liquid if its pressure drops below the dew point during production. If gas is preferable to liquid production, reservoir pressure can be maintained by fluid injection. Reservoir fluid composition determines:

• Fluid type—dry gas, condensate gas, volatile oil, black oil
• Method of fluid sampling, laboratory tests
• Surface equipment (type and size)
• Calculation procedures for determining oil and gas in place
• Techniques for predicting oil and gas reserves
• Prediction methods for future production rates, and
• Depletion plan and secondary or enhanced oil recovery methods.

Common Types of Petroleum

There are several common types of petroleum:

Associated Gas: Is the natural gas and NGLs, which under reservoir conditions, are dissolved in the crude

oil or are present as a gas cap above the oil in the reservoir.

Condensate or Distillate: Is the pale straw-coloured liquid with an API of 45° to 75° produced at surface from hydrocarbons which were originally gas or liquid in the reservoir. The term is often loosely applied to any liquid produced at the separator from light volatile oil or gas fluids.[15]

Conventional Black Oils: Are the most common reservoir liquids. They have: a viscosity low enough to flow naturally into a well; gravities that are usually between 20° API to 45° API; GORs ranging from 100-2000 scf/stb (20-360 m^3/m^3); specific gravity from 0.6 to 1.0; viscosities ranging from below 1cp; and, liquids that are about as thin as water to those that are >100 cp. They are black to green-black in colour.

Crude Oil (Oil): Is the common liquid form of petroleum produced from an oil reservoir when the gaseous constituents have been removed or have escaped and ranges from heavy tarry substances to conventional oil. Most petroleum liquid products and crude oils are lighter than water and their weight is often expressed in degrees (°) API.

The higher the number of API degrees, the lighter the oil. An API rating of 46° for a crude would mean that it is super light. Heavy oil would have an API of 18° to 20° degrees. The commercial value of oil varies according to its specific gravity; heavy oil trades at a lower value, i.e. less than 20° API trades at a lower value (US $5-10) to lighter oils, i.e. from 20° API to 45° API. Above 45° API, oil is considered superlight and has a progressively higher value (US $15 or more).

Gas Condensates: Condensates that are straw coloured and usually have a specific gravity above 45° API. The distinction between gas condensate, volatile oil fields and gas fields is important in practice as the reservoir may require different production and commercialisation strategies as discussed in *Chapter 11: Refining.*

Heavy Oil: Is so viscous that it does not flow easily into a well and has a gravity below 20° API and a viscosity above 20 cp as well as extremely low (negligible) production rates which often include large quantities of loose sand.

Natural Gas: Is a mixture of hydrocarbons consisting mainly of methane but also including ethane and minor quantities of NGLs.

Natural Gas Liquids (NGLs): Light hydrocarbons consisting mainly of propane and butane, which are liquid under pressure at normal temperature.

Oil Sands: Refers to heavy black tar (similar to bitumen) which is frequently mixed with high volumes of sand. They are found principally in Canada and Venezuela. Oil Sands require mineral extraction production akin to mining which is completely different to oil and gas well production.

Volatile Oils: Oils that have low specific gravities and viscosities, 45°-70° API and GORs in excess of 360/m^3 (2000 scf/stb). They are pale red to brown in colour[16].

Although the above nomenclature for hydrocarbon accumulations is useful, it should be appreciated that reservoirs do not follow strict definitions and have been found to produce hydrocarbons in almost every conceivable ratio. Additionally, variations in pressure and temperature mean that there are no clear divisions between the classes of reservoirs.

For our purposes, production mainly depends on the physical properties and behaviour of the reservoir fluids which change once production has commenced. Those changes will depend on what is in the reservoir.

What's In a Reservoir?

Reservoirs have been found to produce almost every conceivable ratio of hydrocarbons. It is this diversity, along with variations in pressure, temperature, depth, thickness, sealing faults and potential links to adjacent reservoirs, that leads to oil and gas accumulations being characterised as uniquely different or heterogeneous structures. Carbonate reservoirs are considered highly heterogeneous. Calcium carbonate is much more chemically active than the silica that constitutes sandstones. It is easily dissolved in water, even more so in acidic water. Mechanical properties are another significant difference. Carbonate rocks tend to be more prone to fractures than sandstones. For all these reasons, carbonates form different rock types with a heterogeneous distribution throughout the reservoir. Moreover, the poor correlation between porosity and permeability, and the presence of caverns and fractures, create very complex paths for fluids, making it difficult to accurately model the distribution of permeability in carbonate reservoirs[17].

Consequently, the challenge for the oil company is how

best to produce a particular oil and gas accumulation considering all these factors and simulating their interaction over time.

Reservoir Fluid States

Reservoirs are found at depths varying from 2,000ft+ (610 m) to deeper than 25,000ft+ (7,620 m). As noted in *Chapter 1: The Origin of Oil—Migration*, it is known that heavy oil is usually found in shallow reservoirs while lighter oil is found in deeper reservoirs, with gas alone found in the deepest reservoirs. Pressure and temperature conditions vary between reservoirs. Shallow reservoirs often have near standard conditions (15ºC [59ºF] and 15 psi [1 bar]) while deep reservoirs may have temperatures above 250°C (482°F) and pressures that may exceed 20,000 psi (1378 bar). Reservoir fluid states are held in a complex rock-gas-liquid system and can exist as aqueous and non-aqueous states or multi-phase immiscible fluids[18].

We have seen that hydrocarbons occur in unique ratios and diverse states. The same can be said of reservoirs. Reservoir engineers must have a thorough understanding of this heterogeneity, as this plays an important part in understanding how production should best be engineered. Physical properties are needed to accurately describe fluid pressures up to 1,500 bar (22,000 psi), the possibility of high temperatures (up to 250°C) and corrosive fluids (waters that contain more salt than seawater i.e. approximately 35,000 mg/l). Empirical data and laboratory modelling is often applied to field reservoir applications.

Depending on the oil and gas accumulation, and its reservoir pressure and temperature, hydrocarbons underground may be present initially as:

• Liquid only – oil reservoir
• Gas only – gas or gas/condensate reservoir, or
• Gas overlying liquid – oil reservoir with gas cap, or

gas reservoir with oil ring. The comprehension of such complex natural fluids comes from an understanding of simple and ideal systems, which are modelled in the laboratory. The data required includes: density; compressibility; formation volume factors and gas-oil ratios for determination of recovery factors; viscosity and gas-oil ratios for production rates; and interfacial tension for recovery efficiency, as it has a major influence on oil trapping. See *Chapter 1: Origin of Oil -Trapping Mechanisms*.

The Phase Behaviour of Hydrocarbons

As reservoir pressure drops, the resultant behaviour of the hydrocarbons depends upon the temperature and differential pressure as well as the composition of the hydrocarbons.

As pressure drops, gas expands and liquids tends to vaporise to gas. This is because molecules can move apart through their own kinetic energy breaking the weak bonds that hold them. (See *Chapter 11: Refining – Van der Waals Forces*). Conversely, if pressure is increased, molecules are forced closer together so that gas is compressed and forms a liquid. These changes from gas to liquid and vice versa are known as phase changes and are termed normal behaviour. Understanding this Pressure-Volume-Temperature (PVT) behaviour is essential because it controls the entire oil production process, while the physical parameters are needed to determine the process efficiency and sizing of facilities.

Multi-Component Mixtures

The behaviour of multi-component hydrocarbons presents greater complexity due to the different volatilities of the components involved. Consequently, vapour and liquid have different compositions when in equilibrium. As the pressure drops, the compositions of both the liquid and gas phases change continuously: the first gas appears at the bubble point and only liquid remains at the dew point. One consequence of

API Gravity (°API)	Classification	Specific Gravity (g/cc)
10° to 20°	Heavy Oil	1.0 to 0.93
20° to 30°	Medium Oil	0.93 to 0.87
>30°	Light Oil	less than 0.87

°API = (141.5°/SG - 131.5 [SG = specific gravity at 60°F = 1.0])

Table 4 - A Rough Classification of Crude Oil Is Sometimes Used Based on API Gravity

this behaviour is that the pressure-temperature plot is no longer a simple curve as for the single component; instead it is an 'envelope' – see Figure 4.

The maximum pressure defined by this envelope is known as the cricondenbar; above it, the liquid and gas phases cannot co-exist. The maximum temperature defined by the envelope (the cricondentherm) is, likewise, one above which the two phases cannot co-exist. The critical point is the point in the envelope at which the properties of the gaseous and liquid phases become identical – it is not related in any simple way to the cricondenbar or the cricondentherm.

The behaviour of the fluid as it leaves the reservoir (essentially an isothermal environment) and travels through the production tubing and wellbore to the separation facilities requires more complex considerations of the thermodynamic behaviour; however, simple laboratory measurements are sufficient for design calculations[19].

If the reservoir pressure is at the bubble point, the oil is said to be saturated. If the reservoir pressure is above the bubble point, the oil is said to be undersaturated. An oil reservoir which is discovered with a gas cap is at its bubble point and is, therefore, saturated. An oil reservoir that is unsaturated describes hydrocarbons above their bubble point, where the reservoir temperature is substantially below the critical point and surface GORs are low to moderate. On production, as the reservoir pressure drops, gas comes out of the solution (solution gas drive). The first gas liberated is composed principally of the lightest components (methane, ethane and propane) as they possess the highest molecular energy and the lowest molecular attraction for other molecules.

Vaporisation of the lighter components is usually followed by quantities of heavier components until at low pressures only a fraction of the original material remains liquid. Gas has formed due to vaporisation of the light components and, as a result, the remaining liquid is described as having shrunk in volume. For a black oil, the shrinkage is only a small amount (often less than 30%). It increases rapidly, however, through the low pressure range (separator pressures) and through volumetric loss of intermediate and heavy material from the remaining liquid. Shrinkage characteristics in this range of pressures are extremely significant because surface separation of oil from gas occurs under these conditions.

Condensate Fields

A condensate field is where the reservoir temperature lies between the cricondentherm and the critical temperature. In this case, if the overall reservoir pressure is allowed to drop, liquids condense out in the formation and may be lost because their saturation is so low that no liquid flow toward the well bore occurs (zero permeability to liquid). In order to prevent this valuable loss by retrograde condensation and to extract the liquids, reservoir pressure is often kept above the dew point by recycling the gas that remains after surface processing. A gas (wet or dry) field is one in where the reservoir temperature is above the cricondentherm. Once the gas starts to expand up the tubing to the surface, the temperature as well as the pressure falls, and this continues to the final surface conditions. Liquid hydrocarbons may condense out in the tubing and surface lines and are often recoverable. Low-temperature separation increases the yield of these valuable light-end liquids. A dry gas field is one in which the final point (normally the separator) lies to the right of the envelope and no liquids are formed.

Crude Oil Properties

The PVT characteristics of oilfield liquids are more complicated than for gases and it is usual to distinguish between saturated and unsaturated conditions. In the former, gas starts to separate from the liquid as soon as pressure begins to drop with production. In the latter, the pressure at which gas begins to separate from the liquid is some distance below the initial reservoir pressure at the bubble point, (Pb). The rate of pressure drop in an unsaturated depletion type field can be quite dramatic with a pressure drop of perhaps 1,000 psi for a production of only one or two percent of the oil initially in place. The reservoir fluids have pressure-dependent properties. It is necessary to know how the crude will behave as the reservoir pressure drops, or other reservoir conditions are altered to be able to determine how best to: produce a particular crude-oil accumulation; to forecast attainable production rates and the ultimate cumulative production; and, to develop EOR plans for a reservoir. These properties are measured in the laboratory using samples of crude taken from the field[20].

PVT Data for Oil

Oil and gas behaviour can be described by using functions of pressure and temperature. Various parameters such as oil and gas interaction, composition and the phase envelope need to be determined for each reservoir. This is often done by laboratory testing of bottom-hole samples or, by using Repeat Formation Testing (RFT) or Modular Formation Dynamics Tester

(MDT) tools. Additionally, oil and gas collected at surface may be recombined to represent the reservoir fluid as precisely as possible. This is, however, a difficult task. In many reservoirs, there are variations across the field and also between different reservoirs. Fluid sampling should be carried out as early as possible to ensure reserve calculations, well flow calculations and facilities design are based on representative samples. Great care is needed in conditioning the well to ensure that the fluid sample is representative. Generalised correlations have been developed which give information about the PVT properties for oil and dissolved gas using the available data obtained from a producing well test, e.g. oil gravity, gas gravity, producing GOR and reservoir temperature[21].

Difficulties arise from obtaining representative samples and deciding the correct thermodynamic path the fluids should follow in the laboratory to mimic the path followed by the hydrocarbons as they move through the reservoir to the well, to the surface and finally to the gauges and the stock tanks.

The processes affecting the fluids as they flow from the reservoir to the stock tank vary, but can normally be approximated to the flash or differential process. For instance, flash liberation can simulate the process in the tubing linking the formation to the surface and in the gathering lines from wellhead to separator because the agitation of the flow keeps the two phases in contact with each other. In the surface-gas separator, the pressure on the produced fluids is suddenly dropped and the gas evolved remains, for a time, in contact with the crude, i.e. a flash liberation.

In general, less gas is evolved in differential than in flash liberation, thus a greater proportion of the lighter hydrocarbons remain in liquid form when the pressure reduction follows the differential-liberation path. For black oils, the difference is usually small, but for volatile oils it can be substantial so that two or three stage separation is needed to drop the surface pressure from that at the wellhead to atmospheric (stock-tank pressure) to get maximum liquids (perhaps 8-11% more). Determination of the number of intermediate separators (GOSP) and the pressures at which they should operate depends on oil and gas properties as well as economic considerations[22] (see Figure 5 for Gas Oil Separator Plant).

Reservoir Pressure and Temperature
In normal conditions, reservoir pressure is about equal to the hydrostatic pressure (pressure due to a column of water) measured from the surface. The hydrostatic gradient is about 0.45 psi per foot (9.6 kPa/m). Temperatures increase with depth by 10°F to 20°F per 1,000 feet (1.8-3.6°C/100m). The table shows reservoir pressures according to depth.

In overpressured reservoirs, the initial pressure may be considerably higher. If different datum corrected pressures are found in different parts of the field, particularly after some production, it is likely that the field is not totally in communication and that there are sealing faults or isolated sands[23].

Reservoir Temperature
Primary recovery methods rely on the assumption that reservoir temperature stays constant. As fluids are produced any change in downhole temperatures due to production is compensated by heat from the cap or base rocks, which are considered to be heat sources of infinite capacity[24].

Average reservoir temperatures are therefore needed for laboratory analyses reflecting reservoir conditions. Reservoir temperatures are used to determine fluid properties such as viscosity, density, formation volume factor and gas in solution. Downhole gauges (during drilling or permanent) are used to measure reservoir temperature.

If a variation in temperature is detected across a reservoir after correcting for depth, an average value can be calculated and used as a constant reservoir temperature. For EOR, involving chemical and miscible processes, changes in temperature affect both the phase behaviour of injected and produced fluids, and therefore will affect recovery. The modelling of such processes must be accompanied by laboratory tests carried out using reservoir temperatures. In EOR processes that employ heat injection, such as steam or in-situ combustion, reservoir temperatures do not remain constant. In these cases, the reservoir temperature needs to be monitored all the time so as to detect the movement of the heat front[25].

Development of an Oil or Gas Field
Once a discovery has been made, appraisal wells are drilled to determine the extent of the accumulation. The important reservoir calculations from the discovery data are the minimum size of the accumulation and the minimum size needed for commercial production. The appraisal wells are then sited to attempt to answer the question, 'Is this economic?' rather than 'How large is it?' With each appraisal well comes a refinement of the
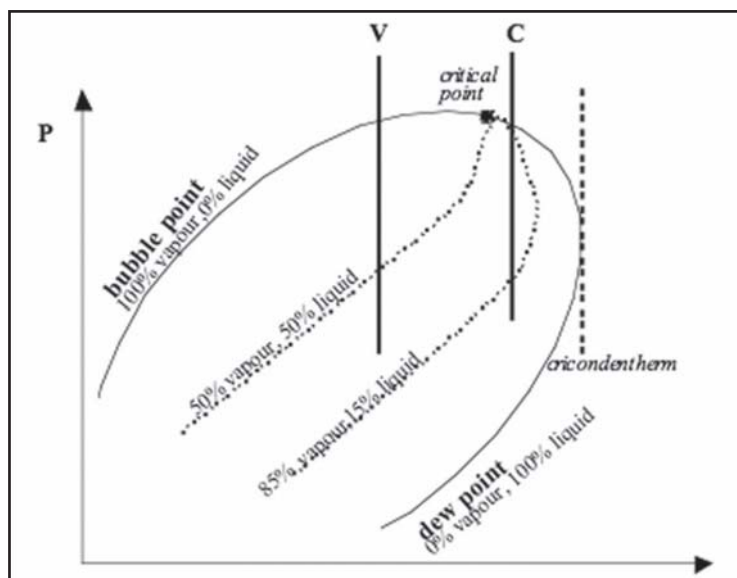
Figure 4 - Cricondentherm, After Professor Richard A. Dawe

geological model of the accumulation, as represented by maps and cross-sections, and a new economic assessment. If it becomes obvious that the accumulation contains sufficient oil or gas to be considered commercial, development plans will be formulated. The siting of development wells is different from that of the appraisal wells, as now the purpose is to produce the petroleum as efficiently as possible at the lowest unit cost. If the field is complex, with multiple reservoirs and faulting, the most efficient well-spacing may be initially difficult to decide as each fault block may have to be regarded as separate accumulations. Over time production of fluids from the reservoir will change fluid pressure and flow rates. Production engineers will critically examine these factors to ensure that production can maximised over the life of the field.

This was a tough chapter but we now know what is in a reservoir and what actually constitutes a barrel of oil. What we have yet to learn is where these barrels are. Who are the 'oil haves' and 'have-nots'?

Readers note; reservoir and reservoir fluid characteristics are well covered in industry texts. Physical and chemistry texts provide the background to PVT behaviour, single and multi phase fluid flow.

References

1. Crude oil volumes are still reported in barrels and in some cases in tonnes. However, the number of barrels contained in a tonne varies according to the type and specific gravity of the crude involved. An average number would be around 7.33 barrels per ton. Surface oil is reported at stock-tank (st) conditions, with volumes in cubic metres (m^3) or barrels [stb, or (st)bbl].

2. API What a barrel of crude oil makes. API Factsheet.

3. See Petrobras Technology Harts E & P, June 2003 p 45 for heavy oil definition below 19ºAPI.

4. TTNRG Nature's Best Wajid Rasheed.

5. Pricing differential is due to higher proportion of heavier and sourer (high sulphur) crudes that relative to light sweet production. More than half the world's produced oil is heavy and sour in quality and this proportion is expected to increase. This depends on the crude oil's molecular structure and sulphur content. The oil will be classified accordingly and priced using reference crudes. Some of the common reference crudes are: West Texas Intermediate (WTI), Brent blend from the East Shetland Basin of the North Sea. Dubai-Oman, used as benchmark for Middle East sour crude oil flowing to the Asia-Pacific region, Tapis (from Malaysia, used as a reference for light Far East oil), Minas (from Indonesia, used as a reference for heavy Far East oil), The OPEC Reference Basket, a weighted average of oil blends from member countries.

6. The compositions of different crudes are measured and published in assays. Refining engineers use assays to decide which crudes will be required to formulate products.

7. API 5 RP 44 Sampling Petroleum Reservoir Fluids Proper management of production from a natural gas

Figure 5 - GOSP In The Shaybah Field Saudi Arabia (Saudi Aramco)

or petroleum reservoir can maximize the recovery of the hydrocarbon fluids (gas and oil) originally in the reservoir. Developing proper management strategies requires accurate knowledge of the characteristics of the reservoir fluid. Practices are recommended herein for obtaining samples of the reservoir fluid, from which the pertinent properties can be determined by subsequent laboratory tests.

8. For gas wells, the inverse ratio is sometimes used and the liquid-gas ratio is expressed in barrels per million m^3 (or million cubic feet).

9. Formation, Removal, and Inhibition of Inorganic Scale in the Oilfield Environment Author: Wayne W. Frenier and Murtaza Ziauddin ISBN: 978-1-55563-140-6. See also Scale formation RP 45 Analysis of Oilfield Waters 3rd Edition/August 1998.

10. Refining costs Sulphur Corrosion Control Author: Charles Kirkley See also RP 49 Recommended Practice for Drilling and Well Servicing Operations Involving Hydrogen Sulphide Recommendations include well drilling, completion, servicing, workover, downhole maintenance, and plug and abandonment procedures conducted with hydrogen sulphide present in the fluids being handled. 2nd Edition / May 2001. Further cost is added at the refining stage.

11. The Color of Oil Economides et al. Publisher: Round Oak Publishing Company; (March 1, 2000) 220 pages ISBN: 0967724805.

12. EIA BTU fuel content.

13. See also API Manual of Petroleum Measurement Standards. This manual is an ongoing project, as new chapters and revisions of old chapters are released periodically.

14. Condensates Energy supplies are often quoted in barrel of oil equivalent (boe). The energy contained in 6000 scf (170 s m^3) of gas is about equivalent to that in one barrel of oil (0.16 s m^3), so for an oil with a gas-oil ratio of 1500 scf/bbl (266 m^3/m^3), 25% of the energy from the reservoir is contained in the produced gas. Thus for black oils about 10 % of the produced energy is in the gas, whereas for the gas condensate field about 75% of the energy is produced as gas. For this reason condensate reservoirs are not produced for the sake of the liquids only. A gas field of size 0.6 trillion scf is equivalent to an oil field of around 100 mmbbls.

15. See Advanced Reservoir Engineering Author: Tarek Ahmed and Paul McKinney ISBN: 0-7506-7733-3.

16. Saudi Arabia Oil and Gas Issue 4 The Carbonate Challenge (www.saudiarabioilandgas.com).

17. The behaviour of reservoir fluids is based on the laws of physical chemistry for perfect gases and the phase changes in gas-liquid systems.

18. The Flow of Complex Mixtures in Pipes, 2nd Edition, G.W. Govier and K. Aziz. Thirty-five years after its first publication, remains a fundamental resource, providing a unified approach to all types of complex flow.

Depth of Reservoir	Initial Pressure	Temperature
608m (2000')	61 bar (900 psia)	21-32°C /70-90°F
1520 m (5000')	153 bar (2250 psia)	38-65°C /100-150°F
3952 m (13000')	408 bar (6000 psia)	82-149°C /180-300°F

Table 5 - Reservoir Pressure by Depth

19. Lab Crude Samples McCabe, Warren L.; Smith, Julian C.; Harriot, Peter (2005), Unit Operations of Chemical Engineering (seventh ed.), New York: McGraw-Hill, pp. 737-738, ISBN 0-07-284823-5.

20. API 5 RP 44 Sampling Petroleum Reservoir Fluids Proper.

21. SPE 102854 Performance Appraisals of Gas/Oil Separation Plants by S. Kokal, SPE, and A. Al-Ghamdi, SPE, Saudi Aramco.

22. If the field is communication similar datum corrected pressures will be found as average reservoir pressure drops.

23. Fluid Flow & Heat Transfer In Wellbores A.R. Hasan and C.S. Kabir.

24. The properties of crude oil and hydrocarbon gases have been extensively studied over the past several decades and many useful tables and correlations can be found in prior work e.g. charts (Dawe and Bradley 1987, McCain 1990).

25. The compressibility of oil is not entirely pressure dependent. The reported density of the oil is almost always that of the stock-tank oil not the reservoir oil, although reservoir oil density varies with pressure due to the associated effect of the gas in solution, which varies with pressure.

An accurate knowledge of Pb (bubble point pressure) is important when producing a reservoir. It is the reservoir pressure below which gas comes out of solution so that production can become more complicated. Gas affects oil recovery and production rates, well performance, and vertical pressure-loss calculations.

26. The oil-formation volume factor, Bo. When the pressure is released to below Pb, gas comes out of solution. As the oil is produced the drop in pressure and temperature in the wellbore causes the oil to shrink.

27. The oil density, ro.

28. The API gravity (the equivalent oil density at stock tank conditions.

29. When the reservoir pressure drops below Pb, some gas is released from the oil in the formation. The gas-solubility factor, R, is the volume of free gas at standard conditions that is released from that volume of reservoir oil that results in a unit volume of stock-tank oil.

30. There is a great variation in oil viscosity mo, from formation oils that are thinner than water to heavy oils, having the consistency of a thick tar. The main effect of pressure (<Pb) on viscosity is that gas comes out of solution, and as the gas contains the lighter hydrocarbon molecules, the viscosity tends to increase as pressure drops.

The current version of IUPAC's standard is a temperature of 0°C (273.15°K, 32°F) and an absolute pressure of 100 kPa (14.504 psi).

Standard Conditions are generally 15°C and 1 atmosphere (or sometimes 1 bar) or 60°F and atmospheric pressure (14.7 psi).

Natural gas volumes are reported in standard cubic metres [(s)m^3] or standard cubic feet (scf). Quantities of natural gas are usually expressed in cubic feet; a cubic foot is equivalent to approximately 0.028 m^3 at standard conditions. However, for reserves valuation, gas is usually expressed in thousands (103) of cubic feet (Mcf), millions (106) of cubic feet (MMcf), billions (109) of cubic feet (BCF) or trillions (1012) of cubic feet (TCF). ●

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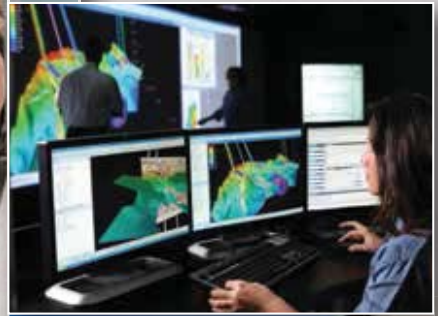
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Jan/Feb	Mar/Apr	May/Jun	Jul/Aug	Sep/Oct	Nov/Dec
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