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Historical Perspective

Pore-scale analysis of formation damage; A review of existing digital and analytical approaches

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ABSTRACT

Formation damage is one of the most challenging problems that occurs during the lifetime of a well. Despite numerous previous studies, an organized review of the literature that introduces and describes the digital and analytical approaches developed for formation damage analysis is lacking. This study aims to fill this gap through briefly describing the main mechanisms behind formation damage in porous media as well as investigating the main related experimental methods with an emphasis on novel imaging techniques. Specifically, there will be a focus on a number of modern and nondestructive analytical methods, such as dry/cryogenic Scanning Electron Microscopy (SEM), X-Ray Diffraction (XRD), CT-scanning (both using adapted medical scanners and the use of high-resolution micro-CT instruments) and Nuclear Magnetic Resonance (NMR), which obtain outstanding results for the identification of formation damage mechanisms. These approaches when used in combination provide a robust identification of damage processes, while they reduce the risk of operational mistakes for decision makers through visualization of the distribution, severity, and nature of the damage mechanisms.

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1. Introduction

Formation damage – an adverse change to the pore space of the reservoir rock during oilfield operations – is well-known as one of the most serious issues in the oil and gas industry. The damage is an end product of several mechanisms including physical, chemical, hydrodynamic, thermal, mechanical and biological damage which are triggered during drilling, production, workover, and hydraulic fracturing operations [1–3]. However, regardless of the reason, this phenomenon can cause changes in pressure, sand production, scale precipitation, pore blockage, and reduction in production rates below the expected economic limits [4,5], and lead to a reduction of production rate [6,7]. Furthermore, damage may also limit the ability to inject fluids of different composition into the formation.

In extreme conditions, formation damage might even force well abandonment, especially in the case of reservoirs with extremely low absolute permeability [8,9]. In any event, damage imposes extra costs related to well stimulation operations, and puts the economic validity of a well under question due to a significant reduction in the well flow rate [10,11].

During the last few decades, the above-mentioned consequences of formation damage have inspired the development of experimental and analytical methods for understanding, preventing and/or controlling formation damage in oil- and gas-bearing formations. In this regard, laboratory experiments are crucial to replicate downhole operating conditions and to help benchmark and validate numerical simulation models [3,12,13]. In addition, analytical techniques provide quantitative measurements of the severity of damage and to suggest procedures to prevent or reverse the reduction of reservoir deliverability avoiding further complicated expensive stimulation operations [14,15].

Traditionally, core flood measurements are used to quantify the reduction in permeability. These averaged measurements can be complemented by optical petrography, Scanning Electron Microscopy (SEM), X-ray Computed Tomography (CT) scanning, and Nuclear Magnetic Resonance (NMR) T2 relaxation measurements to identify the mechanisms of formation damage, from which methods to avoid or reduce the damage can be designed [16–19].

Each of these methods have their own advantages and disadvantages, both scientific and economic. In the end a combination of techniques are needed for a full understanding of formation damage, with recent advances in imaging technology offering considerable promise.

Despite the numerous studies carried out in this field, there exists a lack of an organized introduction to the existing formation damage analytical approaches. The present work aims to fill this gap through listing the major mechanisms contributing to formation damage phenomena as well as the most common applied analytical methods, especially recently-introduced imaging approaches.

2. Formation damage mechanisms

Drilling fluid invasion is considered as the first initiator of formation damage that plugs the pores around the wellbore and is usually difficult to be removed using the back flow method [1]. The magnitude of damage depends on a range of parameters including incompatibilities between drilling fluids and formation water, exposure time of drilling fluids, and the amount of mud filtrate leakage [20]. On the other hand, the accompanying solid particles may also cause significant damage depending on their size distribution, formation pore distribution, and pressure condition inside the wellbore [21].

In addition, the vast majority of natural damage leads to a severe reduction in reservoir productivity [22]. For instance, deposition of paraffins and asphaltenic parts of the crude oil might take place in the wellbore, near wellbore zones, production equipment, perforation channels, and producing slots [22,23].

Moreover, a permanent reduction in permeability is expected during secondary recovery processes mainly due to scale deposition resulting from incompatibility between formation water and injected fluids, as well as organic precipitations formed by variations in thermodynamic conditions [24,25]. Fines migration is another drastic damage resulting from the injection of high pressure fluids into the reservoir [26]. In addition, pore blockage in oil-bearing layers caused by the retention or adsorption of chemicals flooded within reservoirs is another significant cause of formation damage impeding the effective flow of chemical solutions, mainly including polymers, surfactants, and nanoparticles, inside the porous medium which leads to lower ultimate recoveries [5,27].

Table 1 lists the most common upstream operations, their possible associated damage and the potential prevention or remediation approaches.

3. Formation damage analysis approaches

This section describes the standard traditional and modern analytical methods used to analyze various kinds of formation damage.

3.1. Traditional analytical methods

Darcy's law and thin section petrography (TSP) are among the two main traditional approaches used for the evaluation of formation damage as discussed below.

3.1.1. Darcy's law

Darcy's law is used to measure the permeability of core samples before and after damage. Here, core permeability can be determined using the linear relationship between the injection rate and the pressure difference across the core sample.

One of the main disadvantages of this method is fulfilling the principle of steady-state conditions, such that there are no changes in the core during the injection test. Reaching steady state may be impossible to achieve, as damage continues, or takes a long time. Also, for tight samples, the relatively high displacing and confining pressures required might result in a low measured permeability caused by unavoidable changes in the stress state which compress the rock. Consequently, the use of Darcy's law is considered as impractical and inaccurate to evaluate damage in shales and tight sandstones [53]. Moreover, the difficulty of relating the results to well inflow performance is a frustrating aspect of this method [9].

3.1.2. Optical petrography (OP)

This technique includes microscopic analysis of a rock thin section, typically around 30 μm in thickness, which aims to describe various petrographic parameters including texture, fabric, sorting, primary and secondary porosity, fracture types, relative abundance, clay minerals, and cementation [3]. However, detection of fines invasion and alteration of the pore structure can be challenging and the information provided by this destructive technique is only applicable for samples for which thin sections can be acquired without altering the pore space [54].

Li and He [55] employed the OP method to analyze the interactions between filter cakes and rocks. They stated that thin section analysis can supply an appropriate means of assessing the formation damage mechanism, and can be considered as a pragmatic tool for the evaluation of drilling-fluid-based damage, particularly the effects of particle settling.

It is worth noting that, considering the recent developments in imaging techniques, OP is mainly used in preparation for other analytical methods, such as X-ray CT scanning, SEM and TEM, which are described next.

Table 1
List of the most common operations in upstream oil and gas industry, associated formation damage and prevention/remediation approaches.

Operation	Main damage mechanisms/effects	Prevention/Remediation
Drilling	• Solid/fluid invasion [1] • Absolute permeability reduction [1,28] • Pore plugging [1,29] • Emulsification [28,30] • Clay swelling [1,22] • Dispersion and precipitation of salts [1] • Increased interstitial water content [28] • Fines migration and sand production [25,31]	• Air and/or gas drilling [28] • Bridging material [32,33] • Filter-loss control agents (such as organic oil-soluble particles) [21] • Water-based muds (WBMs) with divalent ions such as Ca^{2+} and Mg^{2+} [21] • Perforation [2] • Dissolving the fines and enlarging the pore-throat sizes using acidizing and stimulation processes [2,34] • Using inorganic salts such as NaCl, KCl, NH_4Cl, and $CaCl_2$ in drilling fluids (known as stabilizers) [35] • Nanofluids [36–38]
Casing and Cementing Completion	• Hydration of cement [21] • Recreation of lime [21] • Fresh water invasion [22] • Clay swelling [22] • Clay migration [22] • Water blocking [39]	• Addition of KCl to spacer or cement slurry [20] • Fluid loss additives (such as HEC[4] & PVA[5]) [20] • Open hole completion with gravel pack [40] • Underbalance perforation [21] • Using clean perforating fluids [21] • Adequate shot density [21] • Negative pressure completion (especially in perforation) [41]
Stimulation	• Detrimental byproducts of stimulation [11] • Iron precipitation (due to acidizing by HCl) [42] • Permeability alterations [43]	• HCl/HF solutions (with HBF_4 [44], H_3PO_4 [45], $AlCl_3$ [46], H_3BO_3 [46] or CH_2O_2 [47]) • Using water- soluble polymers in fracturing fluids [48]
Primary production	• Blocking the pore throats [22,23] • Altering the formation wettability from water-wet to oil-wet [23] • Increasing hydrocarbon viscosity by nucleating water in oil emulsions [22,23] • Permeability reduction [23] • Plugging the production sluts and gravel pack [22] • Inorganic scale [22,25]	• Acidizing [49] • Injection of organic aromatic solvents and soaking [22,23] • Acid/solvent washes (for scale removal) [22] • Sand control (in case of fines migration) [50]
Water flooding	• Incompatible fluids [24] • Suspended solids [21] • Mineral scale formation [24] • Permeability reduction [24] • Bacterial problems [21] • Fines migration [51] • Microbial problems (like hydrogen sulphide gas by sulphate reducing bacteria (S.R.B.)) [52]	• Squeeze inhibitor treatment [24] • Keeping a constant temperature [24] • Filtration [21] • Using nanofluids (such as SiO_2) to prevent from fines migration [51]

3.2. Digital rock physics (DRP)

During the last decades, novel non-destructive imaging techniques and associated analysis and modeling, often called digital core analysis or digital rock physics, have transformed our understanding of processes in porous media [17,56].

Three-dimensional (3D) imaging technology can be used to describe the nature and geometry of mineralogical components while, along with 2D optical identification (see the previous section), it can be used to identify organics in porous media [57]. However, 2D images alone represent a biased perspective of the pore structure and cannot assess the connectivity and hence permeability of the sample [56].

Inevitably there is a trade-off between the high resolution and ease of interpretation of 2D imaging methods – both optical and using electron microscopy – and the superior assessment of connectivity, but with generally lower resolution achieved by non-destructive 3D X-ray imaging methods [54]. We will now present the different methods and assess their strengths and weaknesses for the evaluation of formation damage.

3.2.1. Electron microscopy techniques

The wave characteristic of electrons was first theorized by Louis de Broglie in 1925. Subsequently, in 1932, Knoll and Ruska proposed the idea of an electron microscope to compensate for the limited image resolution in optical microscopes [58]. The resulting image could reach a 0.05 nm resolution and magnifications of up to about 10,000,000× mainly due to the fact that electrons have de Broglie wavelengths of about 100,000 times shorter than visible light [59,60].

3.2.1.1. Transmission Electron microscopy (TEM). The first commercial TEM was developed by Metropolitan-Vickers Company, UK in 1936 that was also a trigger in outlining the idea of a practical electron microscope. Further developments in the technology led to high-resolution transmission electron microscopy (HRTEM) [58].

TEM is acknowledged as a promising method providing accurate information on crystallography, microstructure, and composition of the regions of micrometer to sub-nanometer sizes in thin samples [61].

As an example of a recent application, Khishvand et al. [62] have demonstrated significant fines mobilization during low-salinity water flooding using TEM to image effluent samples at nanometer resolution (ranging from 0.28 to 9.9 nm). For instance, the TEM images in Fig. 1 evidence the presence of grain (possibly clay) particles in low-salinity water flooding and effluent samples with oil films adhered to their surfaces.

3.2.1.2. Scanning Electron microscopy (SEM). SEM provides magnified images that help to reveal microscopic-scale information on the size, shape, composition, crystallography, and other physical and chemical properties of a specimen [63]. It was developed by German physicists in the early 1900's in parallel to the development of conventional TEM [64]. The key advantage of SEM over TEM is that thick samples can be used, as the electron beam scans the surface – the beam does not have to pass through the sample to acquire an image.

Without any doubt, SEM plays a key role in various geological and petroleum engineering fields, including pore-scale and invasion analysis, sediment diagenesis, mineral alterations, texture and pore space properties, and the evaluation of fluids performance [65,66]. In particular, SEM analysis can produce high-quality images of porous media even

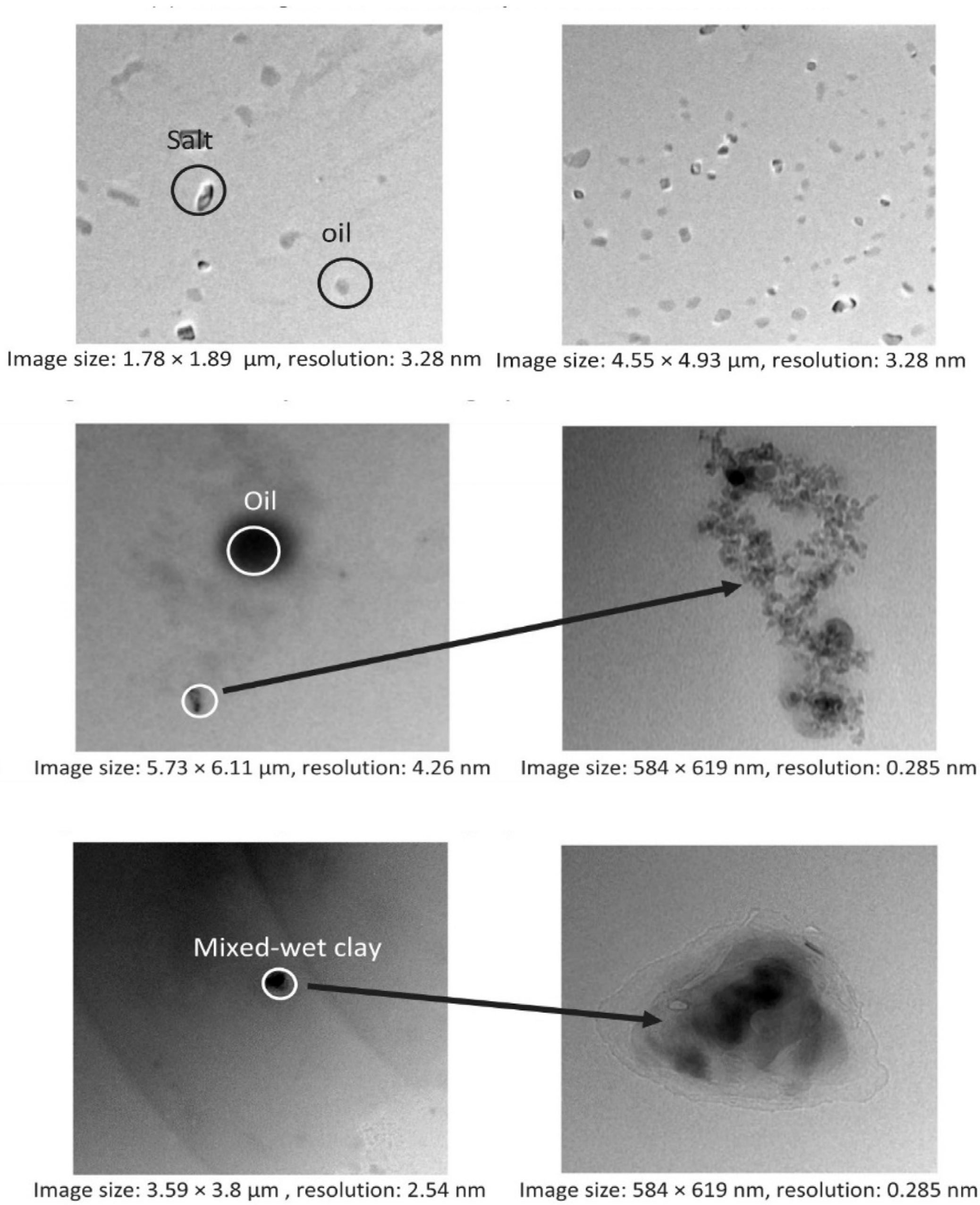

Fig. 1. (Top row) TEM images of the effluents of the HSWF (high salinity water flooding) experiments; (Second and third row) TEM images of the effluents of the LSWF (low salinity water flooding) experiments [62].

from low permeability reservoirs [17,57]. Moreover, combined with 3D image analysis techniques, it has been widely used for qualitative analysis of formation damage [9,67]. We now list some of the more important studies which have studied the different chemical, physical and biological processes causing formation damage.

Pittman and Thomas [68] discussed the applications of SEM to study reservoir rocks, including for clay minerals and the formation damage they can cause. Later, Yang and Sharma [69] employed this method to study the growth of large calcium carbonate or calcium sulfate crystals in the pore space, as well as the presence of other fines in a sandstone core invaded by cement filtrate.

Lappan and Fogler [70] studied the effect of bacterial polysaccharide production on formation damage and found evidence of bacterial plugging of the pores using the SEM imaging technique. In another study, Ghalambor et al. [71] investigated the nature of formation damage due to the growth of bacteria on formation minerals. They confirmed the growth of bacteria on the minerals and consequent reduction in permeability.

Merdhah and Yassin [72] investigated the permeability reduction due to scale formation and deposition during water injection where they employed SEM imaging to determine the particle size, morphology and formation of $CaSO_4$, $SrSO_4$, and $BaSO_4$ precipitates.

Leontaritis et al. [73] presented an approach for prevention and treatment of formation damage caused by asphaltene deposition using thin section and SEM analysis. Moreover, Arciniegas and Babadagli [74] used FIB-SEM to characterize the thickness of organic deposition (mainly asphaltene and resins) on the surface of glass beads.

Fraser et al. [75] used SEM and ESEM (Environmental SEM) to investigate the formation damage characteristics of Mixed Metal Hydroxide (MMH) drilling fluids and compared the results with the ones achieved from polymer-based fluids. They used the ESEM in early tests; however, due to the comparable results and the greater clarity of conventional SEM, ESEM was not used further for illustrative work. According to the results of SEM analysis, no external cake was visible in the case of the polymer/KCl fluid and the SEM showed solids invasion to around 200 μm depth following the formation of an internal cake.

Note that ESEM is an extension of the SEM method in which some of sampling preparations and handling requirements for SEM are eased to provide a broader range of observation conditions, especially for non-solid materials. SEM and ESEM are quite similar from a hardware perspective. The main difference is the introduction of gases into the specimen area of ESEM which aims to mitigate the charging effects in insulators, to provide an alternative mechanism for amplification of electron signals, to examine the surface of practically any specimen (wet or dry, insulating or conducing), and finally, to provide direct observation of hydrated/liquid specimens in their natural state [76,77].

Byrne et al. [78] presented the application of dry SEM and thin section analyses to determine solid damage mechanisms, such as, clay fines migration, scale precipitation and drilling mud solids invasion. Moreover, they investigated the other types of formation damage, such as wettability alteration, microemulsion-related damage, fluid retention, and oil or water blockage using cryogenic SEM analysis. Furthermore, the study introduced a new technique of cryogenic SEM using EDS (Energy Dispersive X-Ray Spectroscopy) analysis for X-ray mapping of the remnant mud bodies to determine solid and fluid distributions.

Later, Sánchez et al. [79] performed a study on the behavior of scleroglucan-based formulations generally proposed for high-temperature zones and/or high permeability reservoirs against the more common xanthan-containing polymer flooding. Here, the cryo-SEM dynamic filter-cake observations evidenced a lower filter-cake permeability obtained under dynamic conditions with homogeneous structure, no aggregates, and a structural polymer network.

Schembre and Kovscek [80] used SEM to analyze the composition of selected effluent samples to explain the mechanism of fines migration at elevated temperature that confirmed the production of fine clay material.

In another study, the evolution of sand pack pore and grain structure was determined using SEM imaging and compositional analyses where it was shown that hot alkaline water injected at rates close to field rates resulted in grain-cementing precipitation [81].

Habibi et al. [82] studied the use of different types of nanoparticles (NPs), including MgO, SiO_2 and Al_2O_3, to reduce fines migration in synthetic porous materials where SEM imaging was carried out for qualitative observation of fines attachment to pore surfaces. This work clearly showed the adsorbed fines on the treated solid surfaces (Fig. 2). The results suggest the MgO modified nanofluid as the most efficient one to treat synthetic porous media.

Laboratory experiments were conducted by Tang et al. [83] to evaluate formation damage caused by complete water vaporization and salt precipitation in sandstone reservoirs where SEM imaging was used to observe the presence of crystal salts in pore throats. In particular, SEM analysis suggested that the treated core inter-granular pores were filled mainly with NaCl crystals causing blockage of the small pore spaces.

Kar et al. [84] investigated the effect of various types of clay minerals on the performance of Steam-Assisted Gravity Drainage (SAGD) where the SEM-EDS technique was used to study the structural, elemental and petrophysical changes during the process.

Zhang et al. [85] took SEM images to study the texture of the fines generated from the shale fracture face where the results showed that clay content in shale rocks is the fundamental cause for the significant reduction of fracture conductivity after water flow.

Later, Xu et al. [86] reported the application of SEM imaging to investigate the effects of formation damage on microscopic features of surface morphology in low permeability reservoirs. The results not only showed changes in surface structures but also contributed to better understanding of the Specific Surface Area (SSA) and Pore Size Distributions (PSDs).

SEM-EDS imaging was also employed to study the effect of mud weight on filter cake properties by Fattah and Lashin [87] where a greater reduction in permeability of the filter cakes derived with the fluids treated with barite was observed compared with the ones treated with calcite.

Later, Yao et al. [88] employed SEM to distinguish and analyze micromorphological and geometrical features of the fines generated from tectonically deformed, undeformed and granulated coal horizons.

Rabbani and Salehi [89] presented a dynamic model for deposition of mud solid particles over and through the porous sandstones during permeability plugging experiments where SEM coupled with image processing were used to find the porosity and pore size distribution of mud cake.

Table 2 presents a list of previous work, not already discussed above, on the application of SEM in formation damage analysis in chronological order. The overall conclusion of this section is that SEM techniques are widely used to study the nature of formation damage, as well as the composition and structure of effluent from damaged rocks.

3.2.2. X-ray diffraction (XRD)

XRD is a powerful nondestructive method to analyze the structural parameters of crystalline materials based on phase identification through comparison with a reference database of X-ray diffraction patterns [108,109].

Application of this technique in the oil industry dates several decades, including for the analysis of rock composition, mineralogy and clay fraction identification [54,110], studies of the effect of temperature

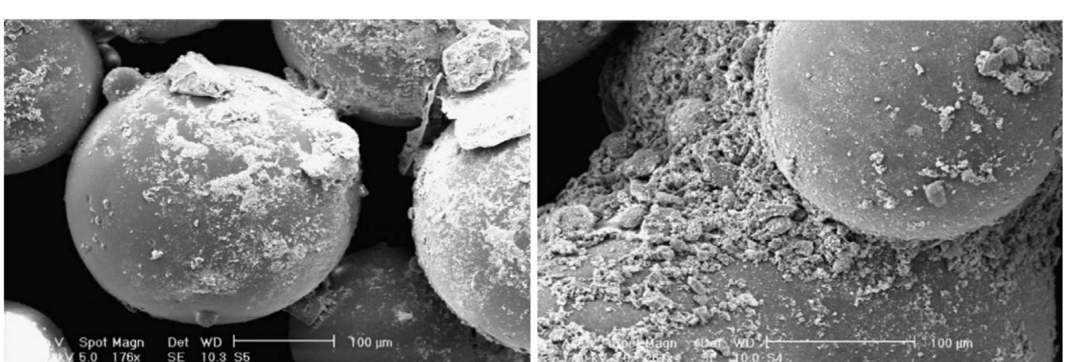

Fig. 2. (Left) Fines attached on glass-beads surface without any treatment; (Right) attached fines on treated glass beads with MgO [82].

Table 2
A list of previous work on the application of SEM in formation damage analysis (sorted in chronological order).

Authors	Method (s)	Aim(s)	Major achievement(s)
Hartmann et al. [90]	SEM	To analyze the original structure of drilling fluid filter cake through frozen-hydrated and freeze-dried stages combined with the study of fluid loss data.	Combination of SEM with fluid loss analyses resulted in a better understanding of formation damage and the principles behind the creation of filter cake.
Argillier et al. [91]	Cryo-SEM	Cryo-SEM-based characterization of mud cake structure achieved from water-based drilling fluids with new formulas.	Correlation of internal and external structural characteristics of filter cake.
Merdhah and Yassin [72]	SEM	To analyze scale formation and deposition caused by water injection.	Evidence of the formation of $CaSO_4$, $SrSO_4$, and $BaSO_4$ precipitants.
Polson et al. [92]	ESEM	To measure the wettability of quartz surface with and without biofilms (bacterial/ fungal colonizations).	Identification of wettability alterations from hydrophilic towards hydrophobic condition.
Blkoor and Fattah [93], Al-Yaseri et al. [94]	SEM-EDS	To investigate the influence of XC-Polymer on drilling fluid filter cake properties and formation damage.	SEM analysis provided a good method to study the structure, morphology and average quantitative chemical compositions of the filter cake.
You et al. [95]	SEM	SEM-based determination of occurrence state and morphology of salts within the pore spaces of tight sandstone sample.	SEM images showed that chlorite and illite/montmorillonite were the main clay minerals in the samples.
Zhang et al. [96]	SEM	To investigate rheological properties and formation dynamic filtration damage evaluation of a novel nanoparticle-enhanced viscoelastic surfactant fracturing system constructed with wormlike micelles.	The result of SEM indicates formation of nano-filter cake that can decrease the amount of viscoelastic surfactant fracturing fluid filtration.
Abhishek et al. [97]	SEM	To visualize the adsorption of silica nanoparticles on Berea sandstone and their effect on fluid/rock interaction during LSW flooding.	SEM imaging confirmed a preferential adsorption of NPs obtained by static adsorption tests and visually shows that more NPs adhere/adsorb on quartz surface compared to kaolinite while it did not lead to blockage of pore throats.
Khan et al. [98]	SEM	To investigate the morphology of gel injection's formation damage before and after treatment with ultrasonic waves and chemical agents.	SEM imaging is applicable to analyze the variation in viscosity of gel sample and efficiency of ultrasonic waves stimulation.
Yang et al. [99]	SEM-EDS	To investigate formation damage due to fines migration induced by the leak-off of low salinity potassium chloride (KCl) fracturing fluids.	Direct evidence of fines migration as a formation damage mechanism using SEM imaging of core sample, SEM-EDS analysis of produced solids and close matching of lab data with an analytical model.
Zhao et al. [100]	SEM	To evaluate formation damage mechanisms associated with drilling and completion fluids for deep-water reservoirs.	The main mechanisms of formation damage by drilling and completion fluids were analyzed based on pore throats blocking by solid invasion in which are ideal for formation of an external filter.
Azizi et al. [101]	SEM	To analyze the impacts of scale inhibitors on the morphology and deformation of precipitated scales.	SEM analysis revealed that scale inhibitors can affect the shape of scale crystals.
Ghamartale et al. [102]	SEM	To determine pore structure alterations after ultrasonic treatments.	SEM imaging suggests that, owing to the high heterogeneity of dolomite samples, ultrasonic wave treatments were not effective.
You et al. [103]	SEM-EDS	To investigate fines migration in geothermal reservoirs.	Illite/chlorite and kaolinite deposition caused permeability reduction in geothermal reservoirs.
Wang et al. [104]	SEM-EDS	To detect fines migration throughout the hydrate dissociation processes.	Formation damage caused by fines migration was identified using SEM imaging.
Abbasi et al. [105]	SEM-EDS	To determine the morphology and composition of mixed salt precipitation that may occur during smart water injection in carbonate formations.	The results of SEM-EDS analysis suggest SrSO4 as the dominant produced scale that affects mixing of the injected water with formation water.
Prempeh et al. [106]	SEM-EDS	To analyze the effect of a capillary-entrapped phase on fines migration in engineered sand-packs with different percentage of kaolinite and residual oil.	Presence of a residual phase significantly decreases the magnitude of formation damage and the amount of produced kaolinite.
Wang et al. [107]	SEM-EDS	To analyze reservoir damage during acidizing for high-temperature and ultra-deep tight sandstone.	SEM-EDS analysis evidenced the migration of fine particles and subsequent blockage of the pore throats with a consequent decrease of permeability.

and pressure on clay swelling [111], and measuring the density of grains [112]. It has been also used to study various types of formation damage, such as the reduction of permeability due to clay swelling [113] and KCl brine injection [114].

In pioneering research, Gkay and Rex [115] employed a combined XRD and SEM technique to analyze clay migration where they concluded that dispersion of kaolinite or mica needles was the root cause of formation damage in sandstone core samples.

Zhou [116] discussed the application of XRD analysis to determine the effect of fluid and overburden pressure, temperature, pH, salinity and brine composition on clay swelling in aqueous solutions in which was later developed by Zhou et al. [111]. They also concluded that osmotic swelling can cause more severe formation damage in compared with crystalline swelling.

Krishna et al. [117] employed XRD analysis of clay samples to evaluate the dependency of basal spacing of smectite on the type of cations and suggested crystalline swelling as the main cause for reduction in the recorded permeability values. Following from this work, Amorim et al. [118] revealed a stronger control over clay swelling for $CaCl_2$ brine samples compared to NaCl and KCl solutions based on the results of XRD analysis.

Al Moajil and Nasr-El-Din [119] analyzed the manganese citrate precipitated during acidizing operations using a combined XRD, XRF (X-ray fluorescence) and ESEM method where they found a direct relationship between the magnitude of precipitation and initial concentration of acid and temperature. It was also recommended that high-pH fluids should not be used for cleaning of wells drilled with Mn_3O_4-based fluids.

Combining SEM-EDS, X-Ray Diffraction (XRD) and Nuclear Magnetic Resonance (NMR) analysis methods (which is described later), Zhou and Nasr-El-Din [120] investigated interactions of phosphonic-based hydrofluoric acid with clay minerals in sandstone cores as a function of initial acid concentration, reaction time, and temperature. At the end, no trace of aluminum fluoride (AlF_3) in the reaction products was identified through elemental analysis of kaolinite, bentonite, and illite

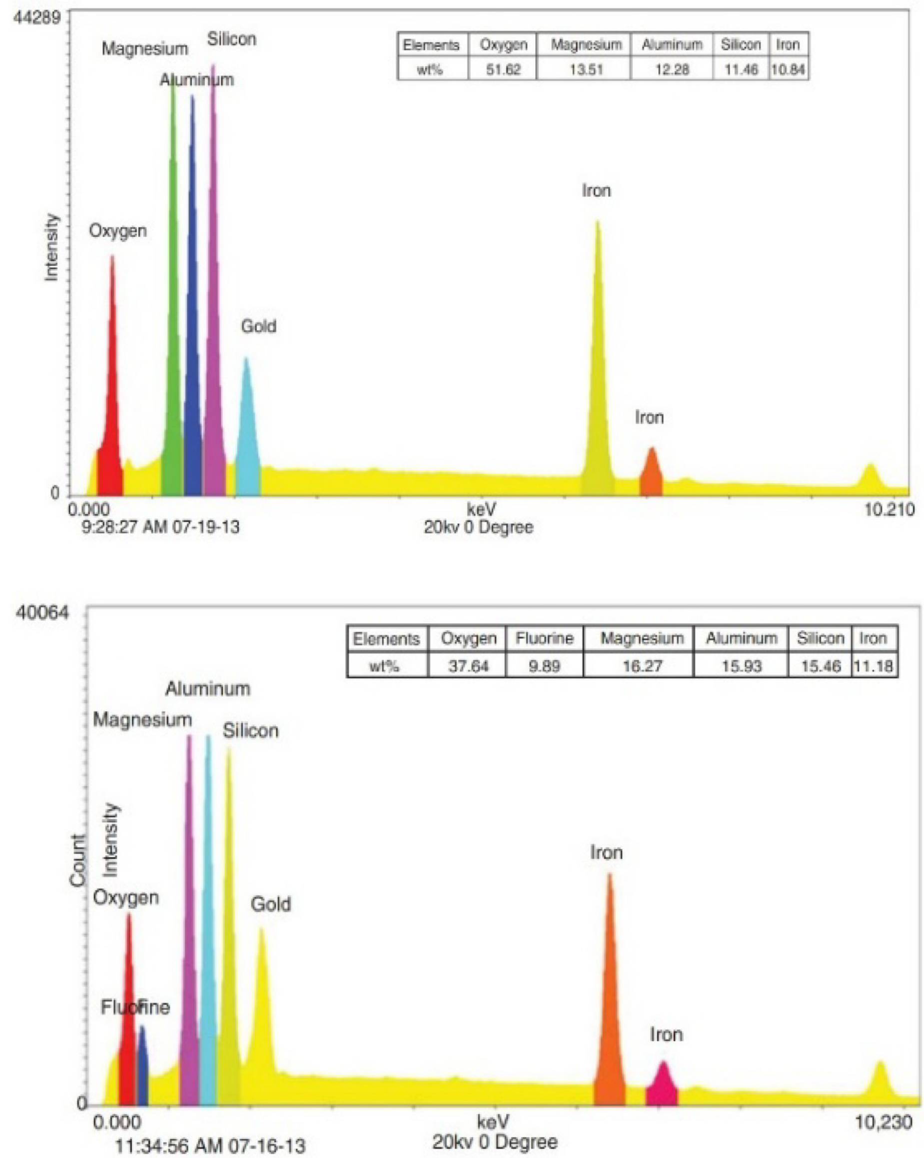

Fig. 3. (Top) Elemental analysis of chlorite before treatment with full-strength phosphonic-based HF acid; (Bottom) Elemental analysis of chlorite after treatment with full-strength phosphonic-based HF acid at 77 °F for 24 h [120].

samples while the story was different for the case of chlorite mineral. Fig. 3 shows a comparison of the chlorite spectrum before and after treatment that confirms the presence of F peak in the spectrum.

Recently, Ahmad [121] investigated the role of flow rate and fluid alkalinity on fine particle movement using XRD diffractometric plots of selected sandstone core samples (examples are shown in Fig. 4). They suggested that migration of illite, kaolinite, chlorite, and fine particles of other minerals is the main cause for reduction in permeability based on the results of XRD and SEM analysis.

X-ray diffraction offers a technique complementary to direct imaging, allowing the chemical composition of fines material and other causes of formation damage to be determined with great accuracy. Usually these studies are performed in combination with imaging to allow the structure and nature of the damage to be determined as well.

3.2.3. X-ray computed tomography (CT) scanning

X-ray imaging is an extremely powerful and nondestructive technique to probe the internal structure of rock samples [122,123]. The first CT scanners were introduced by Hounsfield in 1969 and became commercially available in 1972 when this technology was employed in the UK for widespread medical use [124,125].

In the petroleum industry, CT scanners were first used in the 1980's [126,127] to determine reservoir rock characteristics [128]. As listed in Table 3, they can be categorized into four classes based on object size and spatial resolution. The instruments can either be adapted medical scanners with limited power and resolution, industrial or special-purpose X-ray instruments designed for high resolution, as well as imaging using bright synchrotron radiation which allows images to be acquired in a few seconds [129].

3.2.3.1. Formation damage analysis using conventional CT-scanning. Gilliland and Coles [131] used conventional CT-scanning (a low-power adapted medical scanner) to investigate the severity and location of the damaged zones. In the same way, Ozgen Karacan et al. [132] suggested that the CT-scan technique is a reliable method to evaluate the homogeneity or heterogeneity of core samples.

Bartko et al. [133] revealed the efficiency of CT-scanning to evaluate acidizing systems and the associated reactions between acid with

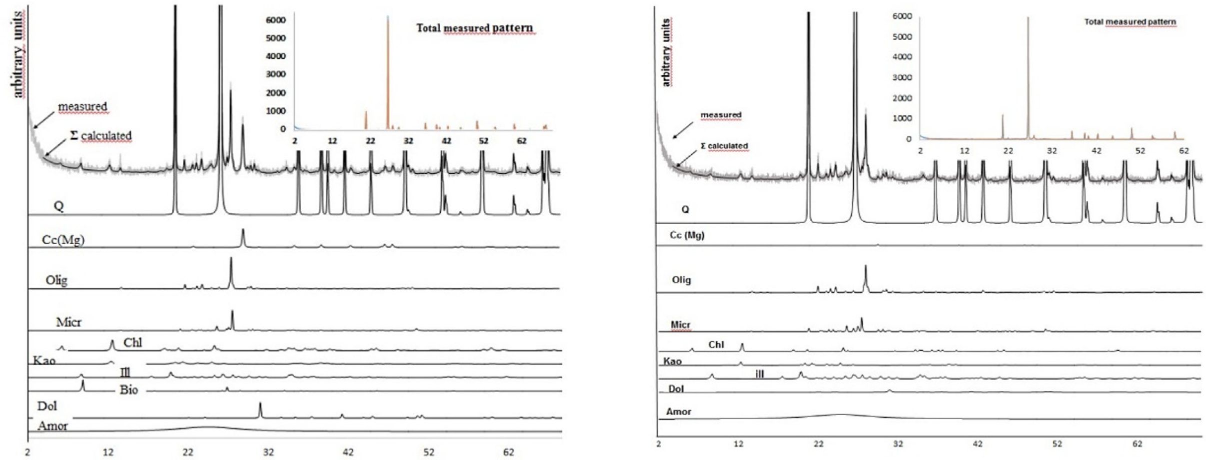

Fig. 4. Examples of XRD patterns of selected sandstone core samples [121].

minerals. Vickerd et al. [134] also employed this technique to investigate the influence of acid gas flooding on morphology, size, and distribution of the pore structure, as well as fine dislodgement and desiccation.

Mahmoud [135] assessed the scale damage from precipitation of calcium sulfates during low and high salinity water injections and, using CT imaging, observed a greater magnitude of damage inside the core samples flooded with high salinity water.

Tran et al. [136] presented a good example from a combined CT scanning and Nuclear Magnetic Resonance (NMR) to analyze the formation damage mechanisms for a single phase flow core-flooding. NMR is described later in this paper.

Khan et al. [137] conducted a couple of experiments to analyze the particle flow infiltration while flooding into soda-lime glass bead core samples. Using modified medical CT-scanners, they measured the variations in porosity caused by fines migration.

In a recent study, Shamsi et al. [138] used CT-scanning to study the damage occurred by injection in both heterogeneous and homogenous porous media where they observed a non-uniform damage pattern even in homogenous core samples. They also realized a greater magnitude of damage in the inlet of core samples.

The problem with the use of conventional X-ray imaging using adapted medical scanners is that they provide images of rather poor resolution and cannot accurately capture the texture of the pore space and certainly cannot elucidate pore-scale mechanisms. Industrial scanners have more power, but are more expensive, and are not routinely used in oilfield applications, despite having far superior imaging characteristics [139]. Furthermore, imaging has to be combined with other techniques, such as XRD or XRF to provide chemical information [140]. What is needed is a non-destructive imaging technique with micron resolution, described next.

3.2.3.2. Formation damage analysis using micro-CT scanning. The recent advent of micro-CT imaging has provided new understanding of the pore-scale structure of the rocks and the fines and fluids within them [54]. Moreover, a deeper insight into the interactions between drilling/completion fluids and host rock can be achieved through a combination of micro-CT scanning and other available imaging methods at different scales [9,67].

Table 3
A general classification of existing X-ray computed tomography based on Carlson et al. [130].

Type	Scale of observation	Scale of resolution
Conventional (medical)	m	mm
High-resolution industrial	dm	100 μm
Ultra-high resolution industrial	cm	10 μm
High-resolution micro-tomography	mm	μm

Applications of this method for the characterization of drilling-mud filter cake and damage have been reported in several studies, such as, Elkatatny et al. [141] and Green et al. [54]. The latter presented a novel approach named "difference mapping" that is based on a combination of common geological methods and micro-CT scanning scanning to quantify the change in rock structure due to damage.

In a case study on an oil filed in East Malaysia, large field 3D micro-CT scanning accompanied by Field Emission Scanning Electron Microscopy (FESEM), Pore Network Modeling (PNM) methods and a set of petrophysical analyses were used to identify homogeneous regions of core plugs and to exclude the damaged zones during coring operations [142]. The authors suggested a combination of micro-CT and PNM as an innovative option to quantify the potential influences of core-flooding experiments on multiphase flow and to identify the sensitivities of core handling.

Later, Ma et al. [143] investigated the evolution and internal damage of hydrated shales based on micro-CT scanning and found a gradual increase in damage variables against immersion time with the highest rate of damage during the initial steps.

Using a combination of micro-CT and SEM-EDS, Yu et al. [144] found an 80% of reduction in permeability caused by clay migration during fresh water injection while a smaller 50% decrease was estimated through image-based computations. Moreover, the observed increase in intensity of CT number near the core outlet was attributed to fines straining and clay relocation.

Yang et al. [145] employed 3D micro-CT imaging to analyze the potential damage mechanism of Dongying sandstone during workover fluid flooding and formation liquid flooding. Meanwhile, the changes of pore structure before and after displacement were quantified based on micro-CT intensity profile, pore radius and coordination number that suggested the pore spaces were partially blocked by clay minerals and moving particles. Fig. 5 presents examples from segmented 2D slices and 3D visualizations of the sample.

Table 4 summarizes the main formation damage studies based on CT and micro-CT scanning techniques, not including the work already discussed.

3.2.4. Nuclear magnetic resonance (NMR)

The NMR technique is another method used for the analysis of rock samples: the spins contained principally in protons are aligned in a

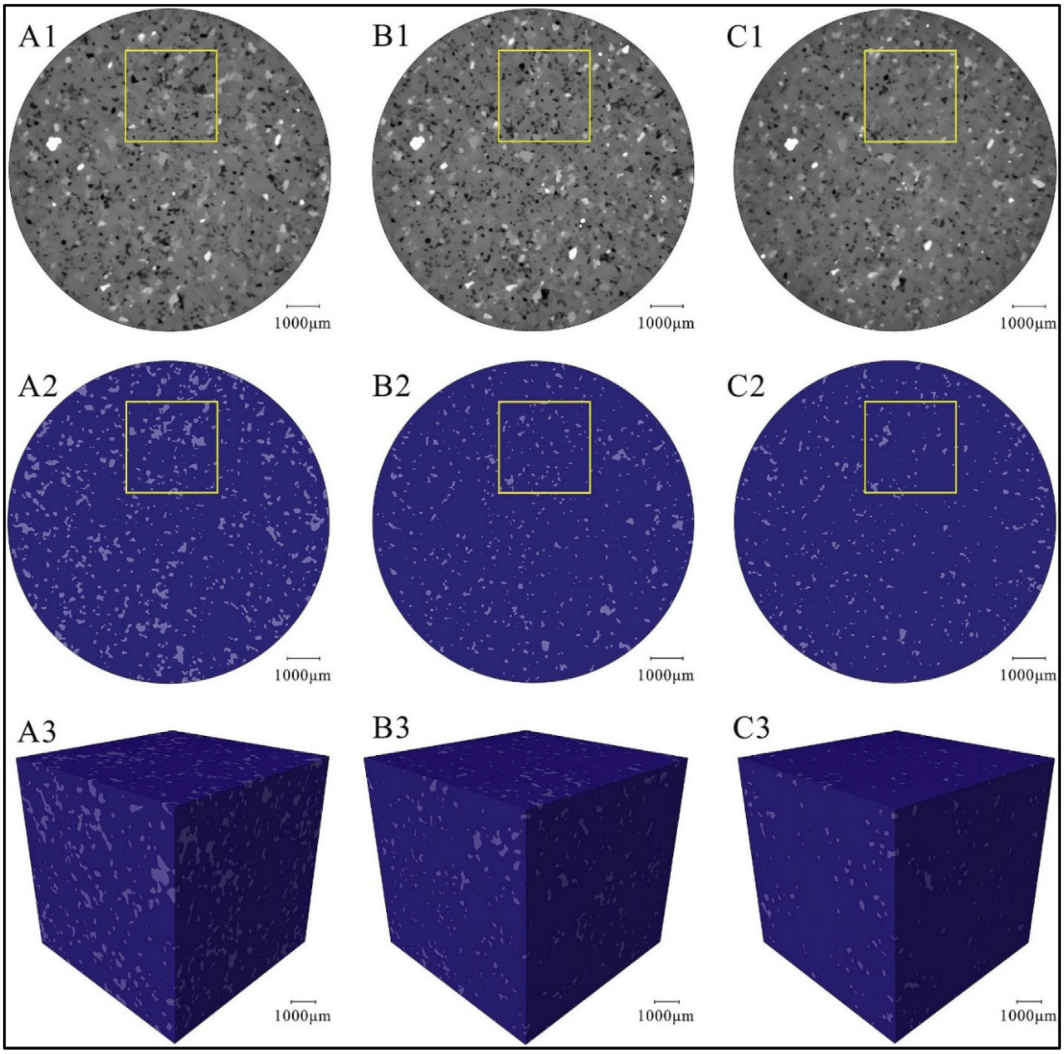

Fig. 5. Raw (top) and segmented (middle) 2D slices and 3D segmented pore space (bottom) through sample 2 (24 μm resolution, pore space is purple and rock matrix is dark blue); dry sample: (A1), (A2), (A3); sample after workover fluid flooding: (B1), (B2), (B3); sample after formation liquid flooding: (C1), (C2), (C3) [145].

magnetic field; when the spins relax, the resultant electromagnetic signal is detected [154]. NMR provides a fast, nondestructive, and convenient tool for the characterization of complex porous media [155–157].

The measurement of how the NMR signal decays, and the total signal, can be used to infer the pore size distribution, porosity and fluid distribution [53,158]. Liu et al. [159] also used NMR to quantify the motion of water within a core sample.

Tandon et al. [160] assessed the application of NMR to evaluate proppant packs and associated mechanical damage. It was concluded that NMR analysis is quite sensitive to permeability and pore structure, presence of fines, mechanical damage, and proppant coating and type.

Guo et al. [156] examined water sensitivity, solid phase adsorption, and damage caused by CarboxyMethyl Guar (CMG) and HydroxyPropyl Guar (HPG) gum fluids using a combination of NMR-based experiments, mercury injection analysis, SEM, and core-flooding tests. They could provide a micro-perspective basis for analysis of damage mechanism, type and origin.

Recently, Wu et al. [161] developed a new experimental method to evaluate the formation damage induced during drilling of sandstone reservoir based on a combination of NMR analysis and large-sized model tests. It was found that reservoir permeability and overbalance pressure can affect both degree and depth of the damage while rate of permeability alteration was found to be higher in low permeability sandstones.

In addition, Lufeng et al. [162] employed a combination of NMR, SEM and Pressure Transmission Tests (PTTs) to evaluate damage in sandstone formations. The results of NMR analysis revealed that the internal reasons for water-blocking and water-sensitivity damage are related to the amounts of mobile water and bound water, respectively.

Bageri et al. [163] investigated secondary formation damage during the filter cake removal process using combined NMR, micro-CT and core flooding techniques. Fig. 6 shows the NMR analysis of the pore structure before and after injection of a barium-saturated chelating agent that releases barite precipitates into the pore space and absorbs cations from either minerals or other pores. Note that, in this study, micro-CT analysis rejected the idea that wormholes were generated due to invasion of filter cake solvent and its subsequent interaction with the rock matrix.

Kamal et al. [164] used a combination of NMR, SEM and core-flood experiments to study the effect of clay mineral concentration on formation damage during water/HCl injection in sandstone formations and concluded that chlorite and illite clay minerals control pore throat plugging and permeability reduction.

In a recent study, Gomaa and Mahmoud [165] studied formation damage resulting from injection of traditional mud acids into illitic sandstones and introduced a novel method of stimulation based on in situ generated Hydro-Fluoric (HF) acid. They used the results of NMR

Table 4
A list of previous work on CT & micro-CT applications in formation damage analysis (sorted in chronological order).

Author(s)	Method	Aim(s)	Main achievement(s)
Karacan and Halleck [146]	CT	To analyze the porosity damage in crushed zone around the perforation tunnels caused by shaped-charge perforating operations.	A compacted zone with variable thicknesses was identified along the perforation tunnels during perforation of gas-saturated rock samples, though they could not observe any compacted zone in the case of liquid-saturated samples.
Okwen [147]	CT	To investigate asphaltene-based damage during tertiary CO_2 flooding.	Imaging showed that reducing the concentration of formation water acting as a CO_2 buffer in crude oil can minimize the flocculation of asphaltenic components.
Guo et al. [148]	CT	To study the filtrate invasion in sandstone core samples during core-filtration analyses.	More realistic in-situ monitoring of filtrate invasion of oil-based drilling fluids was permitted using CT images accompanied with core-flooding experiments compared with HPHT filter press tests.
Yerramilli et al. [149]	CT Optical petrography	To investigate injectivity decline during produced-water reinjection.	Face plugging at injection face and surface deposition caused injectivity declines in porous media.
Ma and Chen [150]	micro-CT	To characterize meso-damage in shale hydration.	Rapid growth of micro-cracks was found in early soaking time, which is considered as the main meso-damage period.
Hu et al. [151]	micro-CT	To study the mechanisms of proppant-fluid-formation interactions in shale reservoirs.	Treatment fluids can decrease the proppant embedment effect and maintain the effective fracture width. Moreover, proper selection of treatment fluid is crucial in reducing the formation-damage-related costs.
Grove et al. [127]	micro-CT	To investigate perforation damage.	High quality quantitative data were achieved on porosity and permeability in both native rock and around the crushed zone.
Ibrahim and Nasr-El-Din [152]	CT	To analyze the fracturing fluid saturation after leak-off and flow-back steps as well as the impacts of soaking time on regained characteristics.	Shut-in wells drilled in shale formations and tight sandstones caused significant decreases in regained flow rates and permeability.
Godinho et al. [153]	CT	To analyze the particle invasion, migration and deposition in porous media based on time-lapse radiography.	Formation damage mechanisms can be efficiently analyzed using quantitative time-lapse radiography which can ease the optimization of drilling and EOR fluids.

analysis to demonstrate the generation of micro fractures and increase in porosity of the core samples due to mineral dissolution.

The key feature of much of this work is the use of a combination of methods to study formation damage. Table 5 lists other work where several different approaches have been used to elucidate the damage mechanism and quantify its effect on pore structure and productivity.

3.3. Other analytical approaches to study formation damage

There are other – more advanced and experimental methods – to study formation damage. These techniques may become more widely adopted as the technology develops.

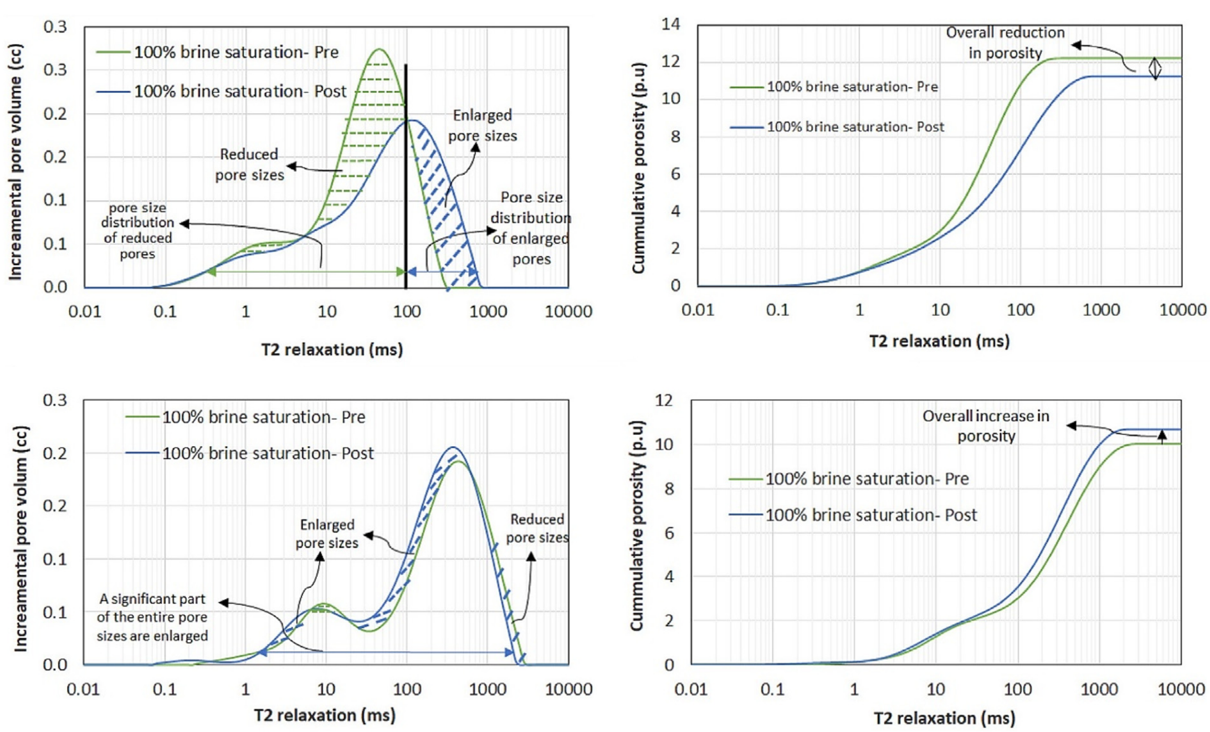

Fig. 6. (Top row) Incremental pore volume and cumulative porosity for sandstone sample obtained from NMR measurements. (Bottom row) Incremental pore volume and cumulative porosity for a carbonate sample [163].

Table 5
A brief summary on the combined application of formation damage analytical approaches.

Authors	Methods	Aim(s)	Major achievement(s)
Longeron et al. [166]	Experimental approach including: static and dynamic invasion tests, and characterization of both external and internal mud cakes using Cryo-SEM	To explain the relationship between filtration rate and permeability impairments. To plot the porosity and oil & water saturation profiles.	• Local saturations and pressure measurements are crucial for the identification of the extent and amount of damage. • Polymeric non-salted mud induced a small damage on moderate clayey sandstones containing viscous crude oil.
Leone and Scott [25]	XRD, SEM and TEM	To analyze the primary damage mechanism during water-flooding in clay-rich reservoirs. To evaluate the characteristics of permeability damage during water injection. To distinguish between likely damage mechanisms, such as, fines migration, and clay swelling and dispersion.	• The significance of damage during brine injection was observed to be a strong function of mineralogy. • The occurrence of a critical velocity, along with other observations, indicate that the primary damage mechanism is fines migration.
Aliaga et al. [167]	CT-scanning, SEM	To investigate the permeability reduction in sand packs caused by solids generation and migration.	• Effluent concentrations, including the produced solids, can be simulated based on a fractional flow model for solids migration.
Rahman et al. [168]	Thin-section, XRF, XRD, SEM, capillary pressure and porosity tests	To study the effect of ion exchange and fluid flow on formation damage of reservoirs containing illitic clay minerals. Morphology of illite in pore spaces of this sandstone and its influence on petrophysical properties.	• XRD study confirmed that illite present in the sandstone samples is susceptible to swelling and migration. • Above a critical salt concentration, however, the illite remains attached to the pore walls.
Weaver et al. [169]	SEM-EDS, XRD, and XRF	To study the damage caused by geochemical reactions between proppant and formation.	• The study revealed loss of permeability and porosity due to proppant dissolution and subsequent remineralization in the pack. • Damage depends on the composition of both the proppant and the formation materials that should be considered when selecting a proppant for high--temperature or high-stress wells.
Mohsenzadeh et al. [170]	SEM, XRD, X-ray CT along with core flooding tests.	To investigate the formation damage due to deep eutectic solvent used as an EOR agent.	• Precipitation and deposition inside pore spaces were identified as the main sources of formation damage based on the results of SEM and quantitative XRD analysis. • CT-scanning was used to observe the damage.
Al-Yaseri et al. [67]	SEM, NMR, micro-CT and core-flooding	Pore-scale analysis of formation damage in Bentheimer sandstone.	• It was revealed that barite particles were first trapped in thin pore throats prior to filling the adjacent pore spaces. • The volume of trapped fines decayed rapidly against the depth of core sample.
Al-Yaseri et al. [94]	SEM, NMR, micro-CT and core-flooding	To investigate the impact of fines and rock wettability on reservoir formation damage.	• A greater magnitude of damage was observed in case the rock surface and the fine (Barite) particles had the same wettability. • Wettability controls the 3D distribution of fines in the pore space.
Li et al. [171]	SEM, TSA, XRD, adsorption and ion analyses.	To analyze the formation damage during alkaline-surfactant-polymer (ASP) flooding.	• The flow paths of chemical solutions and the location of well affect the distribution of formation damage. • Absorption and scaling mainly lead to a decrease of permeability-porosity at injection and production wells respectively. • Corrosion and grain migration mainly result in increase of permeability and porosity at intermediate distances.
Wang et al. [172]	XRD, SEM, X-ray CT and NMR	To study the effect of microwave heating on petrophysical properties of sandstone samples and to eliminate the formation damage caused by water blocking and clay swelling.	• The intensity of heat is determinant in changing the structure, texture and mineralogy of sandstone. • The growth of micro-fractures into larger fractures or pores, can be reflected by the change of T_2 distribution of fully saturated samples. • Intergranular fractures were identified based on SEM imaging of treated samples.
Ji et al. [173]	NMR, SEM-EDS and ^{19}F NMR	To better understand the mechanism of $AlCl_3$ working as a retarding agent in mud acid.	• ^{19}F NMR showed that AlF_3 and AlF_4^- in the spent acid were the only two aluminum fluoride complexes. • More F^- can complex with Al in the Al-based retarded mud-acid system than in the mud acid system, which could lead to a deeper penetration of the acid into the formation.
Fang et al. [174]	NMR T_2 spectrum, MRI image and quantitative evaluation of minerals by scanning electron microscopy (QEMSCAN)	To investigate the alkali sensitivity damage mechanisms in tight oil reservoirs.	• Identification of the extent of potential permeability impairment induced by alkali sensitivity. • Chlorite and illite dispersion and migration were determined as the main triggers for damage. • Generation of silicide precipitation due to reactivity of rock framework with hydroxide is another important alkali sensitivity damage mechanism. • Variations in pore distribution resulted from alkali sensitivity damage can be observed through MRI

(continued on next page)

Table 5 (*continued*)

Authors	Methods	Aim(s)	Major achievement(s)
			analysis of the cores exposed to the fluids with high pH values.
Yang et al. [175]	AFM (atomic force microscopy) and SEM	To investigate the application of silica nanoparticles and environmentally-friendly materials (high-temperature resistance mud) for shale gas horizontal drilling.	• Shale pores were blocked by silica nanoparticles and the average pore size was reduced by 50%. This phenomenon can mitigate pore pressure transmission, reduce the permeability by 98%, and thus enhance the wellbore stability.
Assem et al. [176]	NMR, SEM-EDS, XRF and XRD.	To investigate the factors affecting the interactions of regular mud acid with sand and clay-based proppants under downhole conditions.	• Cristobalite dissolved in the mud acid more than mullite, which can cause fines that might reduce proppant-pack conductivity.
Dai et al. [177]	SEM, microscope system, NMR	To study the effect of supercritical CO_2 fracturing fluid on permeability alteration in tight oil reservoir cores.	• With the increasing thickener contents and filter loss of fracturing fluid, core permeability regains turned to be declined. • Tight cores were more likely to receive more damage in permeability. • The retention of thickener in microchannels (micro pores) were confirmed to be the main mechanism for permeability alteration.
Kamal et al. [178]	SEM, NMR, and core-flooding	To identify the effect of rheological behavior of viscoelastic surfactants on formation damage in carbonate rocks.	• Low-viscosity fluid was found to have more formation damage potential compared with the high--viscosity one. • NMR and core-flooding results revealed that the damage took place both in pore bodies and pore throats. However, most of the surfactant was retained in pore throats.
Radwan et al. [179]	TSA, SEM-EDS, XRD	To introduce a diagnosis work flow to find location and potential source of damage, and recommend a suitable treatment method.	• The proposed formation damage diagnosis work flow was effective and can be applied at any oil or gas well. • Geological studies, petrographic description and mineral identification are very important tools in evaluation of formation damage.
Wang et al. [180]	Low-temperature ash (LTA) + XRD combined with (SEM)	To study water and salt sensitivities in lignite reservoir under different pH.	• Lignite reservoirs with different pH have different degrees of water-sensitive damage. Partial alkalinity and acidity conditions are strongly water sensitive, while strong alkalinity conditions are weakly water sensitive.
Mahmoud and Al-Hashim [181]	XRD, SEM, CT scan, and NMR	To study the effect of flooding sandstone core samples with high pH EDTA chelating agent solutions and subsequent oil recovery process.	• Optimal EDTA solution concentration was determined so that there was no fines migration, clay swelling, and non-clay minerals dissolution. • Gray Bandera core samples were found to exhibit incompatibility with a solution of more than 5 wt% of EDTA and the high permeability enhancement because of the interaction of EDTA with ankerite mineral.
Li et al. [182]	SEM, AFM, NMR and CT-scanning	To investigate the adsorption and retention of hydroxypropyl guar gum (HPG) fracturing fluid in porous medium as an important factor affecting the production in tight gas reservoirs.	• The adsorption and retention of guar gum in tight gas reservoirs resulted in a decrease of the pore to throat size ratio, and the most adsorption occurs at grain edges and rock beddings.
Hajiabadi et al. [183]	CT-scanning and SEM	To investigate rheological and formation damage behaviors of invert oil emulsions (W/O) with the addition of the carbon-based nanomaterials.	• Results proved the dazzling performance of smart nanomaterial in diminishing the severity of pore plugging. • Nanomaterial additives are highly efficient.
Othman et al. [184]	Micro-CT, Itrax-XRF scanning, SEM-EDS and nitrogen permeability	To investigate the mechanism of fines migration during CO_2 injection.	• Mineral dissolution (dolomite and other high--density minerals) dislodged quartz and clay mineral fines. • The permeability damage can be explained by fines migration and subsequent blockage.
Zhang et al. [185]	SEM, XRD, mercury intrusion tests and etc.	To understand the mechanism of formation damage during drilling process in the ultra-deep fractured tight gas reservoirs.	• Results showed that severe fluid sensitivity damage occurred with a decrease in fluid salinity, and an increase in pH values. • Abnormal low water saturation, mixed wettability, abundant clay minerals, and complex pore structures are contributing to the severe phase trapping damage. • As a result of a smaller particle size distribution compared with the larger natural fractures, the existing drilling fluids show a high dynamic damage rate and poor loading capacity.
Zhang et al. [186]	SEM, NMR and pressure-transmission tests	To investigate mechanisms of formation damage from water and alkali sensitivity for a reservoir in China.	• The pore sizes affected the damage mechanisms and suffered from obvious reduction as a result of water-sensitivity damage. In fact, small pores were damaged by swelling of mixed-layer illite/smectite, and the large pores suffered from the combined damage of swelling of smectite and migration of illite.

Table 5 (*continued*)

Authors	Methods	Aim(s)	Major achievement(s)
Zhang et al. [187]	SEM-EDS, XRD and X-ray Photoelectron Spectroscopy (XPS)	To investigate water/rock interactions during hydraulic fracturing in Marcellus shale and to evaluate the formation damage caused by reinjection of flow back water.	• It was suggested that the reuse of non-treated flow back water in field hydraulic fracturing operation can cause blockage of larger pores and flow pathways.
Hajiabadi et al. [188]	CT-scanning, SEM and core-flooding	To study the effect of surface modified nano-silica on drilling fluid and formation damage.	• CT-scanning and SEM revealed the significant effect of NPs in drilling fluids on reducing the depth of penetration. • There was less signs of drilling fluids component in the outlet face of the cores injected by drilling fluids containing surface modified with NPs.
Gamal et al. [189]	NMR and SEM-EDS	To evaluate the effect of exposure time on unconfined compressive strength and the integrity of pore system of Buff Berea sandstone rock samples due to the interaction with barite-weighted mud.	• Extended interaction of mud filtrate with Buff Berea sandstone rock can cause severe formation damage as mud particles, clay swelling, and fines migrations plug pores and throats of the rock. • NMR results confirmed that there was a corresponding increase in formation damage as shown by a progressive decrease in the rock porosity and permeability with time. • The exposure time was found to be a controlling factor for both reducing the rock unconfined compressive strength and porosity.
Kudrashou and Nasr-El-Din [190]	CT-scanning, XRD and SEM-EDS	To investigate the formation damage associated with mineral alteration and formation of swelling clays caused by steam injection in sand packs.	• The dominant formation damage mechanism for sandstone rock with calcite cementing material found to be associated with silica dissolution/-precipitation effects. • For sand packs with quartz, kaolinite, and carbonate minerals this mechanism was shown to be associated with kaolinite-fines migration. • In mixtures of quartz with montmorillonite formation damage was mainly caused by clay swelling, which led to filling of pores with montmorillonite.
Yang and Chen [191]	SEM, micro-CT	To investigate lost circulation material (LCM) soaking process on fracture plugging for fluid loss remediation and formation damage control.	• SEM and micro-CT images indicated that a longer soaking time could lead to a less porous and strengthened plug structure.
Ismail et al. [192]	SEM and core flooding tests	To visualize the invasion depth of drilling mud particles and to evaluate the effect of bridging agent (calcium carbonate) particle size and distribution on permeability and porosity near wellbore.	• The effectiveness of bridging agents depends both the size of particles and the concentration of the bridging agent. • The size calcium carbonate should be almost identical to the pore throat median size of the formation face.

3.3.1. Magnetic resonance imaging (MRI)

In this technique, imaging is based on NMR response as a function of spatial position of the protons [174] that is accepted as a non-destructive and robust technique for visualization of formation damage [138].

Baldwin et al. [193] reported the efficiency of MRI to detect and quantify the formation of hydrate and dissociation in sandstone samples. In another research, Ersland et al. [194] revealed the efficiency of a combined MRI and Nuclear Tracer Imaging (NTI) for the evaluation of selected EOR methods in fractured oil reservoirs. They also demonstrated that MRI is useful to study multiphase flow behavior in fractured porous media.

Krebs et al. [139] presented the application of a combined MRI and micro-CT scanning method for visualizing of the wormholes induced during acid stimulation of selected carbonate samples.

Afrough et al. [195] used MRI to analyze fines migration inside core samples undergoing water-shock fines-migration experiment, and to map the T_2 distributions. They observed opposite trends in T_2 spectra of the core inlet and outlet where their pore-size distribution were shifted to larger and smaller values, respectively. This suggests the average pore size increased at the inlet of the core and reduced at the outlet, consistent with sweeping fines through the core where some fines were retained near the outlet. The authors stated that permeability impairment can be demonstrated using MRI and be applied to remedial or preventive processes during fines migration.

Further, Brattekås et al. [196] used MRI method to find the relationship between petrophysical parameters of oil-saturated fractured carbonate samples and leak off rate of hydrolyzed polyacrylamide (HPAM) gels. The results verified the presence of a stabilized displacement front as well as presence of wormholes in the gel.

Li et al. [197] employed a combined MRI, QEMSCAN and numerical simulation method to analyze the extent of porosity and permeability damage, and evolution of mineral content in tight reservoirs during water-flooding process. The study introduced clay migration and swelling as the main causes of water sensitivity in ultra-low permeable reservoirs.

3.3.2. Focused ion beam and scanning electron microscopy (FIB-SEM)

This technique produces high-resolution 3D image using a combination of high resolution SEM and the precise milling capability of FIB which permits destructive access to the inner structure of a sample [198]. While the resolution of the images is unparalleled, the method requires a long time of operation, only studies a small eroded area with a small observation range, and has a high cost in terms of equipment and expert operation [199].

Note that the development of FIB technique dates back to the mid-1970s at the University of Chicago and it improves the imaging resolution through prevention of hardness-related surface topographic variations [200]. The main principle of the apparatus is similar to SEM instruments, except that in FIB-SEM systems an ion beam is rasterized

Table 6
A brief comparison of the main damage analysis approaches with their pros and cons.

Method	Main applications	Benefits	Drawbacks
Darcy's law	• Estimation of relative and absolute permeability and fluid saturation	• Non-destructive • Convenient and simple method	• Time consuming • Cannot be used in a range of conditions including unsteady-state unsaturated and saturated flow, flow in fractured rocks and granular media.
Optical petrography	• Description of texture, fabric and sorting of rock samples • Identification of primary and secondary porosity • Analysis of fracture, relative abundance of minerals and cement/matrix	• Convenient and simple method	• Destructive • Time-consuming
Transmission Electron Microscopy (TEM)	• Analysis of the inner structure, morphology, composition and crystallography of a sample	• It provides high-quality and detailed images.	• Expensive • Difficult sample preparation • Sensitive to vibration and external magnetic fields • Samples are limited to those that are electron transparent, able to tolerate the vacuum chamber and small enough to fit in the chamber. • Images are in black and white
Scanning Electron Microscopy (SEM)	• Analysis of the size, shape, composition, crystallography, and other physical and chemical properties of a specimen	• High resolution topographical imaging • Elemental analysis • Rapid • Allows the examination of samples such as metals, alloys and ceramics, as well as polymers and biological materials	• Expensive instrumentation • Potential artifacts from sample preparation • Sample preparation may distort material • Sensitive to vibration and external magnetic fields • Images are in black and white • SEMs are limited to solid, inorganic samples small enough to fit inside the vacuum chamber that can handle moderate vacuum pressure. • Non-conductive samples must be coated with an additional thin layer (sputter coating)
X-Ray Diffraction (XRD)	• Identification of the content and type of minerals • Can provide information on the structural parameters of crystalline materials, such as phases, texture (preferred crystal orientations), average grain size, strain, crystal defects, and crystallinity.	• Non-destructive • Rapid • Inexpensive • Convenient and simple method • Interpretation of the resulting data is relatively straightforward • It requires preparation of a minimal sample for analysis	• Only identifies crystalline solids • It is not able to locate each mineral in space • X-Rays do not interact very strongly with light elements • Relatively low sensitivity • Preparation of samples may require grinding them down to a powder
CT-scanning and Micro-CT	• Imaging, quantifying properties and determining the distribution of fluids in porous rocks	• High image quality and spatial resolution • Non-destructive • Rapid • Provide 2D and 3D images • Provide accurate measurements	• Expensive • Limited information on chemical element distribution • Monochromatic until color coded by operator • Rendering of the images is time-consuming
Nuclear Magnetic Resonance (NMR)	• Transport processes, petrophysical phenomena, and chemical reactions in porous media	• Non-destructive • Rapid • Easy sample preparation • Can be used as a portable instrument	• Low sensitivity • Expensive

over the sample sequentially as the FIB cuts through the material. Secondary electrons generated via the interaction between sample surface and ion beam can then provide high-spatial-resolution images. Moreover, the Ga ions used in the majority of commercial FIB-SEM systems enable a precise machining of the sample [201].

It has been shown that FIB-SEM can provide microstructure analysis obtaining unprecedented 3D resolution of a few nm. This method has been applied to image both unconventional shale samples as well as sandstones [202,203]. Further work is needed to use this method to look at formation damage specifically. Here the challenge is to image a representative region of the pore space where sample preparation and imaging has not itself damaged the structure.

Lubelli et al. [204] used cryo-FIB–SEM technique to determine the effect of drying on clay minerals and concluded that drying can significantly change the total porosity and pore size distribution in bentonite while the impacts are milder in the case of kaolinite. In a similar study, Bai et al. [205] visualized different type and abundance of nanopores between 5 and 100 nm, reconstructed the 3D pore structure, and calculated tortuosity, permeability and porosity.

Arciniegas and Babadagli [74] analyzed precipitation of asphaltenes in heavy oils where the morphology of organic deposition on the surface of glass beads was characterized using FIB-SEM. The results revealed a systematic increase in the thickness of asphaltene and showed the significance for calculation of organic deposition surface roughness using FIB-SEM in the characterization of formation damage at different operational conditions.

3.3.3. Acoustic tomography (AT) technique

Acoustic attributes of rock samples can be determined using Acoustic Tomography (AT) which can be used to construct acoustic velocity tomograms to quantify damage caused by deformation phenomena, such as pore collapse, dilatant and elastic deformations, and normal consolidation phenomena [3].

Yamamoto [206] reconstructed high-resolution images of compressional wave velocity from various bottom sediments based on high-frequency cross-well acoustic tomography. A 3D wave-number spectra of the velocity variabilities along with the transformation procedures from velocity image to the images of porosity, density, shear strength, and permeability were determined for each measured velocity image.

Scott et al. [207] conducted two distinct type of experiments including recording the acoustic-wave velocity of samples during rock mechanical tests, and acoustic imaging of deformation using compressional-wave velocities to construct tomographic images from the localized pore collapse damage formed in a confined-indentation test. It was concluded that the extension of such technology is beneficial to monitor the production and mechanical reservoir damage.

Li et al. [208] employed acoustic tomography of unconsolidated porous rocks with residual paraffin saturation and found that acoustic attenuation is sensitive to the microscale- and grain-scale distribution of residual saturation (i.e. residual saturation strengthens the grain contacts).

Based on a couple of rock mechanics tests including velocity measurements of S and P waves, Soares and Ferreira [209] proposed a new method to analyze the mechanical formation damage caused by drilling and production operation. They showed that this approach allows an accurate evaluation of near-wellbore analysis.

Barri et al. [210] used a combined acoustic and CT-scanning approach to analyze the impact of wormholes on elastic properties in carbonate rocks treated by ethylenediaminetetraacetic acid (EDTA) and diethylenetriaminepentaacetic acid (DTPA) chelating agents. The results were indicative of the fact that stimulating with chelating agents had no special effect on mechanical attributes of harder samples, such as Indiana limestone cores, while significant changes were observed in the case of the weaker ones, such as Austin chalk.

3.4. Benefits and drawbacks

Table 6 presents a brief comparison of the existing methods based on their main advantages and disadvantages. In summary, imaging methods provide valuable information that cannot be obtained by conventional methods. In combination, modern methods of imaging and analysis can elucidate formation damage mechanisms and quantify their effects.

4. Conclusions

This study presented an overview of formation damage, which is one of the most challenging problems that occur during the production of oil and gas reservoirs. The different types of analytical techniques used to describe and understand formation damage have been described briefly and then assessed with illustrations of example applications in the literature. The emphasis of this review has been on image-based methods, summarizing the main applications, advantages and drawbacks of traditional methods, electron microscopy, X-ray Diffraction (XRD), Computed Tomography scanning (CT-scanning) and Micro-CT imaging, and Nuclear Magnetic Resonance (NMR).

While numerous efforts have been made on the development of novel approaches for formation damage analysis, more research is still needed to allow a routine comprehensive analysis. Specifically we recommend that the techniques described in this paper are used in combination – no single method provides a full assessment of the nature of the formation damage while quantifying its effects on pore structure and productivity. Instead an integrated approach is required which combines traditional methods, such as permeability measurement and optical microscopy, with more recent developments in high-resolution non-destructive 3D imaging.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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