A comprehensive experimental study on mechanical behavior, microstructure and transport properties
of 3D-printed rock analogs

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Abstract: 3D-printed (3DP) analogs of natural rocks have been used in laboratory tests concerning
geomechanical and transport properties. Rock analogs manufactured by 3D printing can be used to
manufacture batch of the samples with specified heterogeneity compared to natural rocks. Rock analogs
were manufactured with silica sand (SS) and gypsum powder (GP) using binder jetting (BJ) as well as
with coated silica beads (CSB) using selective laser curing (SLC). The uniaxial and triaxial compressive
tests were conducted to investigate the strength and deformation characteristics of 3DP rocks that were
quantitatively compared with natural rocks. CSB and SS specimens experienced tensile failure, while the
GP specimen has shown shear failure and shear-expansion behavior. The microstructural characteristics
(e.g. grain shape, pore type, and bonding form) of the SS specimen were similar to a natural sandstone
(Berea sandstone reported in the literature) with a relatively loose texture. In addition, 3DP rocks were
more permeable than Berea sandstone (permeability of SS, CSB, and Berea sandstone was 12580.5mD,
9840.5mD, and 3950mD, respectively). The effect of microscopic mechanical behavior on macroscopic
strength and failure characteristics was investigated using scanning electronic microscopy (SEM). CSB
and SS specimens could be suitable to simulate the transport behavior of the highly permeable
sedimentary rocks. The GP specimen could be used to study the large deformation characteristics and
creep failure mode of highly stressed soft rocks. Despite the early stage of 3DP rock analog studies, the
potential applications could be expanded by controlling the physical properties (e.g. wettability and
surface roughness).

Keywords: 3D printing; compressive strength; deformation and failure; microstructure; computed
tomography

1. Introduction

A rock is a natural porous medium related to many geological and engineering applications (e.g.
resource exploitation, civil construction, and tunnel excavation) (Tomac and Sauter 2018). Laboratory
experiments conducted on conventional rock samples are essential for validation of analytical and
numerical models, as well as for testing field applications (e.g. prediction of reservoir capacity and
optimization of exploitation scheme) (Li et al. 2016; Du and Wang 2019; Han and Yang 2019; Lv et al.

2019). However, the microstructural characteristics (e.g. pore structure and mineral composition) of natural rocks are often complex and controlled by diagenetic processes, which can lead to heterogeneity of macroscopic physical properties (Lai et al. 2017). The experimental tests cannot always be conducted repeatedly on the same rock sample, because the majority of these tests are destructive (e.g., compressive and tensile strength tests, core flooding, and mercury intrusion porosimetry (MIP)) (Bos and Spiers 2001; Song et al. 2017; Xiong et al. 2017; Song et al. 2019). Therefore, despite the consistency in testing environments and instrument configurations, the test results may vary due to sample heterogeneity. Various approaches (including sand packing and epoxy resin filling) have been attempted to manufacture rock analogs with reproducible macroscopic geomechanical and transport properties (Fatiguso et al. 2013; Kou et al. 2019). These methods remain time-consuming, and the results are uncertain in terms of consistency in structure and texture of samples.

Recent developments in additive manufacturing (AM), also known as 3D printing, offer a cost-effective and time-saving approach to prepare physical models with complex internal structures and geometries (Giannopoulos et al. 2016; Ngo et al. 2018; Francesca et al. 2019). 3D printing has been employed to generate several types of natural rock analogs both in geosciences and engineering. 3D-printed (3DP) rock analogs can support the quantitative validation of natural rock properties and physical processes independent from rock texture and testing environment (Ishutov et al. 2015; Hanaor et al. 2016; Ishutov and Hasiuk 2017; Song et al. 2018; Yin et al. 2020). The potential applications of these analogs have been investigated for petrophysical and mechanical behavior, as well as for transport, acoustic, and electrical properties (Jiang and Zhao 2015; Jiang et al. 2016; Zhu et al. 2018; Zhou et al. 2020; Ishibashi et al. 2020). 3DP rocks have shown remarkable advantages in various applications such as multiple-factors response (univariate tests can be performed on the sample with controlled structure and properties, i.e. the parallel experiment to distinguish the effect of multiple factors on the test results) and up-scaling issues (Martinez et al. 2015; Hanaor et al. 2016; Zhou et al. 2019). Transparent polymer resins combined with the photoelastic and stress-frozen techniques helped visualize the internal stress distribution and structure evolution of 3DP rocks (Ju et al. 2017; Ju et al. 2018; Ju et al. 2020). By using computed tomography (CT) imaging or digital image correlation (DIC), the dynamic fracture propagation and patterns of the 3DP rock-like material has been investigated (Liu et al. 2016; Sharafisafa and Shen 2020). Furthermore, a printed rock sample embedded with a customized fracture network allowed Suzuki et al. (2018) to verify fluid flow issues in a fractured rock mass. Idealized and natural pore network microfluidic chips have been printed to investigate the microscopic mechanism of multiphase flow, including viscous fingering and wettability alteration (Kofi et al. 2017; Li and Zhang 2019; Pisvesan et al. 2019). Other studies evaluated the similarity of mechanical and hydraulic properties between 3DP and natural rocks using powder materials, such as ceramic, silica sand, and gypsum (Ishutov et al. 2017; Kong et al. 2018a, b; Hodder et al. 2018a, b; Ardila 2018; Kong et al. 2019;).

3DP rocks can also be utilized to validate the analytical models in laboratory conditions using digital rock physics (Blunt et al. 2013; Blunt 2017; Liu et al. 2017; Wang et al., 2019). Previous studies have illustrated a comprehensive analysis of applications of 3DP rocks in concert with digital rock physics in geosciences (Ishutov et al. 2018; Suzuki et al. 2018; Kong et al. 2019). Powder-based 3DP rocks have a similar mechanical behavior as natural rocks, unlike polymer resin-based 3DP rock analogs. Nonetheless, polymer resin-based samples have transparency and plasticity that are used to reproduce the topology and surface morphology of porous media (Ishutov et al. 2015; Ishutov and Hasiuk 2017; Ju et al. 2017; Ju et al. 2018; Zhou and Zhu 2018; Hodder et al. 2018b; Gomez et al. 2019). The accuracy of 3DP rocks (e.g., pore geometry, mechanical strength) depends on multiple factors, including 3D printing techniques,

materials, post-processing, and even environmental variables during the printing process (such as temperature and moisture content) (Primkulov et al. 2017).

The main motivation of this study was to enhance the understanding of how microstructure and microscopic mechanical behavior impact the macroscopic response (e.g. mechanical behavior) of 3DP rocks. To this end, we performed a comprehensive experimental analysis of mechanical responses and microstructural characteristics of rock analogs 3D-printed in natural particle materials. The uniaxial-triaxial compressive tests were performed on 3DP rocks and compared with natural red sandstone (Yibin area, southwest Sichuan, China) which had a similar mechanical strength to 3DP rocks. In addition, the effect of thermal curing on the mechanical strength enhancement of 3DP rocks was investigated. Micro-computed tomography (micro-CT) and Scanning Electron Microscopy (SEM)-Energy Dispersive Spectrum (EDS) analysis helped acquire the grain, pore structure, and binder distribution of 3DP rocks. Pore network modelling (PNM) and flow simulation were performed on micro-CT images obtained for 3DP rocks and compared to Berea sandstone in terms of pore-throat structure, spatial connectivity (e.g. coordination number), pore shape, and transport behavior. The effect of microscopic mechanical behavior on macroscopic failure was discussed in an integrated way. The results obtained in this study might aid in revealing the mechanism governing the failure of 3DP rocks caused by various microscopic mechanical behaviors.

2. Materials and Methods

The experimental work conducted in this study can be divided into three parts: (1) sample preparation including 3D printing and thermal curing; (2) mechanical experiments including the uniaxial and triaxial compressive tests; (3) qualitative and quantitative characterization of microstructure including micro-CT imaging and SEM-EDS analysis.

2.1 Preparation of 3DP Rock Samples

There are seven categories of AM process recognized (ISO/ASTM 52900 2015), including binder jetting (BJT), directed energy deposition (DED), material extrusion (MEX), material jetting (MJT), powder bed fusion (PBF), sheet lamination (SHL), and vat photopolymerization (VPP) (Fig.1(a)). The general 3D printing workflow consists of five steps including (I) model design, (II) model output (generally in the STL file format), (III) model slicing, (IV) printing, and (V) post-processing (Fig.1(b)). All materials used in AM can be divided into two categories according to their inherent properties: (1) organic (polymer resin and plastic, such as acrylonitrile butadiene styrene (ABS) and polylactic acid (PLA)) and (2) inorganic (ceramic, gypsum, cement, and silica sand) (Ma et al. 2017; Zhou and Zhu 2017; Ngo et al. 2018; Gell et al. 2019).

(a)

Fig. 1 Summary of (a) AM processes and corresponding materials (MEX: material is selectively dispensed through a nozzle or orifice; DED: a focused thermal energy source is used to melt or sinter materials being deposited; SST: the liquid bonding agent is selectively deposited to join powder material; MJT: droplets of feedstock material are selectively deposited; PBF: powder bed is selectively fused or sintered by a thermal energy source; SHL: sheets of material are bonded; VPP: the liquid photopolymer material is selectively cured by light-activated polymerization) and (b) illustration of the general workflow for SLA technique

Previous studies indicated that high-molecular polymer materials such as thermoplastic and photosensitive resin were prone to large plastic deformation and a unique surface property, which made simulating the mechanical behavior and surface property of natural rocks problematic (Jiang and Zhao 2015; Zhou and Zhu 2017; Ngo et al. 2018). However, natural powder materials, e.g. silica sand and gypsum owe the advantages on simulating surface roughness and wettability properties as in natural rocks. Hence, three types of natural powders, including silica sand (SS), coated silica beads (CSB), and gypsum powder (GP), were used as the matrix material in this study (the particle size distribution shown in Fig. 2). BJT and SLC techniques were adopted to manufacture the rock analogues using Furfuryl alcohol and Phenolic-based resins as binders (Table 1).

Fig. 2 Particle size distribution

Table 1. Materials and printing techniques used in this study

Category	Grain material	binder	Technique	Layer thickness*	Manufacturer
CSB	Coated sand-bead	Phenolic	SLC	0.1mm	EOS PA 2200
SS	Silica sand	Furfuryl	BJ	0.2mm	Voxeljet 1000
GP	Gypsum powder	Furfuryl	BJ	0.2mm	Voxeljet 1000

*represents the minimum layer thickness along printing direction (the Z-axis direction in this case)

Two types of printing techniques were used (SLC and BJ) to produce the 3DP rock analogs. For the SLC technique, which was a type of DED AM process, a HRPS-VI 3D printer (located at Suishen Technology Co. in Beijing, China) with a layer thickness of 0.1 mm was used to fabricate the CSB specimens. By using a computer-controlled laser to heat the binder coated on the surface of silica beads selectively, the initial printing layer was constructed. Then the job platform moved down to construct the next layer, and the same process of selective heating repeated until the entire model was complete. For the BJ technique, a VX1000 3D printer (located at Suishen Technology Co. in Beijing, China) with a layer thickness of 0.2 mm and 10% binder saturation was used to prepare the SS and GP specimens. This technique jets binder directly onto the powder layer according to the designed shape to bond the grains. Due to the CSB specimen was printed with pre-treatment sand-beads, the principle of choosing binder saturation for this kind of specimen is to ensure the surface of particles as smooth as possible, which is beneficial to laser curing during the printing process. Consequently, the different binder saturation was chosen for CSB and SS specimens. All the 3DP rock analogs are designed as intact cylindrical shape without controlling the internal structure (50 mm in length and 25 mm in diameter; Fig.3), which was in accordance with the standard compressive test requirements of a 2:1 height to diameter ratio (ASTM D4543-19 2019).. Primkulov et al. (2017) indicated that the thermal curing could improve the peak strength of the 3DP rocks. Hence, some of the printed samples have been cured at a temperature of 80°C for 24 hours to study the effect of thermal curing on compressive strength.

Fig. 3 3DP specimens (a) CSB sample (SLC technique); (b) SS sample (BJ technique); (c) GP sample (BJ technique)

2.2 Mechanical Testing

Unconfined Compressive strength (UCS) and triaxial tests were performed in the State Key Laboratory of Oil and Gas Reservoir Geology and Exploitation, at the Southwest Petroleum University in Chengdu, China. Two copies of each sample were used for mechanical testing (Table 2). The UCS of 3DP rocks was tested following ASTM D7012-14e1 on a SHT-4106 universal material testing system (Fig.4a) with a maximum operating load of 1000 KN. The force control mode was utilized to initiate the loading process with a rate of 0.05KN/s until failure. The measurement accuracy of force, displacement, and deformation was $\pm 0.5\%$. For comparison, two natural red sandstones (burial depth <200 m) collected from Yibin area, Sichuan province, were used to test the UCS, and the UCS ranged from 9.8 MPa to 12.3 MPa. In addition, the results of Sydney sandstone (burial depth <150 m) reported by Bertuzzi and Pells (2002) were also collected for comparison.

The triaxial tests were performed on a GCTS (Geotechnical Consulting and Testing System) RTR-1000 mechanical testing system (Fig.4b) following the ASTM D2664-04 standard to obtain the shear strength characteristics of 3DP rocks (e.g. inter-friction angle and cohesive force). This system had axial capacity of 1000 KN with a maximum confining and pore pressure of 140 MPa. The specific configurations of both the confining pressure and displacement recorder system are highlighted in Fig.3b,

and a detailed description of this system can be found in Meng et al. (2016). To avoid the hydraulic oil entering the specimen during the compression process, the 3DP specimen was sandwiched between two subplates and sealed into a latex-set, following placement in the pressure chamber. The conventional triaxial stress condition was used in this study (i.e. $\sigma_1 > \sigma_2 = \sigma_3$). Considering the low compressive strength of 3DP specimens, the confining pressures were set to 2 MPa, 4 MPa, and 6 MPa, respectively. The stress-control mode was used at the initial loading stage. A loading rate of 0.01 MPa/s was applied, according to the hydrostatic pressure condition, until the predetermined value (confining pressure) was reached, finally switching to displacement-control mode. A load was applied at a rate of 0.005 mm/s until failure.

Fig. 4 SHT-4106 universal material testing system (a) and RTR-1000 high-temperature and high pressure triaxial testing system (b)

Table 2. Samples prepared for the compressive test

Category	Length/mm	Diameter/mm	Weight/g	Density/g/cm ³
CSB-1*	50.2	24.9	43.63	1.79
CSB-2#	50.1	25.4	43.74	1.74
SS-1	48.82	24.22	32.05	1.42
SS-2	49.6	24.6	33.07	1.40
GP-1	50.17	25.17	21.67	0.87
GP-2	50.15	25.31	22.12	0.88

-1*represents original sample, and -2# represents cured sample.

2.3 Characterization of Microstructure

Micro-CT imaging was performed in the State Key Laboratory of Oil and Gas Reservoir Geology and Exploitation, at the Southwest Petroleum University in Chengdu, China, on the ZEISS XRADIA MICROXCT-400 X-ray scanner to acquire the microstructure of 3DP rocks (e.g. grains and pores). The scanner was equipped with three types of magnification lenses (4x, 10x, and 20x), with a maximum spatial resolution of 1 μ m. The maximum resolution of the CCD camera was 2048*2048/pixels. SEM-EDS analysis was performed in the School of Geoscience and Technology, at Southwest Petroleum University in Chengdu, China, on the FEI Quanta 650 field emission SEM scanner. The FESEM scanner was equipped with a high resolution and stability Schottky field emission electronic gun with a theoretical resolution of 1 nm and 2.5 nm for secondary electronic and backscatter, respectively. The SEM images

could provide detailed information on microstructural characteristics, including pore types and grain bonding, which was a powerful supplement to micro-CT imaging. The mineral composition of the specimen on the same area captured under the SEM scanning was determined with the EDS analysis. This technique allowed us to separate the binder from grains and pores and investigate the binder distribution and pore-filling phenomenon. The porosity of 3DP rocks was also tested using QYK-II type of a helium porosimeter at the Southwest Petroleum University in Chengdu, China, to distinguish the porous characteristics of 3DP rocks that were used to verify the results calculated from CT images.

Considering the imaging resolution, the whole cylindrical sample could not be used to obtain a good view of the microstructure with low resolution, thus several core plugs were drilled from the whole samples (4-5 mm in diameter and 7-10mm in length) for CT imaging (Fig.5a). A pore network model was applied to CT images to quantify the microstructural characteristics (e.g. pore-throat radius and coordination number). The coordination number is a parameter to quantitatively characterize the connectivity of porous media by counting the number of throats connecting a pore (Song et al. 2019). Before the PNM extraction, an image processing workflow, including denoising and segmenting, should be conducted on the selected region of interest (ROI) to enhance the image quality. The PNM was extracted from the binary images of grain and pore phases using a maximum ball algorithm (MBA) (Wang et al., 2019). The MBA incorporates the pore network as pores and throats represented by spheres and cylinders, respectively (Dong, 2007). A voxel-based flow simulation (Saxena et al. 2017; Song et al., 2019) was conducted on the binary CT images to compute the absolute permeability of 3DP rocks, and the results were compared to Berea sandstone to characterize the transport behavior.

Nine fragments (3 original specimens before the UCS test (Fig.5b), 6 specimens (3 cured and 3 uncured) after the UCS test (Fig.5c)) were collected for SEM scanning to investigate the microstructure and microscopic failure pattern. Additionally, EDS analysis was performed during SEM scanning to determine the composition, especially the distribution of binder between grains.

Fig. 5 Samples prepared for micro-CT imaging and SEM scanning (a) core plugs drilled for CT scanning, (b) fragments of uncured specimen before UCS test for SEM scanning, and (c) fragments of uncured and cured specimens after UCS test for SEM scanning

3. Results and Discussion

3.1 Mechanical Behavior of 3DP Rocks

3.1.1 Unconfined Compressive Strength (UCS) Testing

The axial stress-strain curves of 3DP rocks are plotted in Fig 6 (“-1” and “-2” signs represent uncured and cured specimens, respectively). Among the three uncured 3DP rocks, the CSB specimen has the highest peak strength of approximately 6.16 MPa and an axial strain of 1% at the failure point. While the uncured GP specimen has the lowest compressive strength at 3.74MPa, the sample exhibits a plastic expansion phenomenon.

Fig. 6 UCS stress-strain relationship of 3DP rock and natural rock (a) CSB, (b) SS, (c) GP, and (d) red sandstone

For the CSB and SS specimens, in the initial process of the elastic deformation, the specimen experienced porosity reduction. After the porosity compaction, it exhibited a linear stress-strain relationship. With the increase of axial load, there was a plastic deformation approaching the peak strength (Fig.6a and 6b). After the peak point, the axial stress decreased while the strain continuously increased showing strain-softening characteristics, especially for the SS specimen (Fig.6b). For the GP specimen, the mechanical behavior was quite different from CSB and SS specimens, which exhibited a strong plastic expansion phenomenon and a lower yield strength of 3.74 MPa (Fig.6c).

Thermal curing as a post-treatment provides a significant improvement to the compressive strength of 3DP rocks. The increase in strength is observed to be 32.4%, 43.4%, and 145% for CSB, SS, and GP specimens, respectively (Table 3). Thermal curing accelerates the reaction of binder bonding between

grains and decreases the moisture content of the specimen, which supports the higher strength observed. For the GP specimen, the strength enhancement from post-processing reaches 145%. Gypsum powder is a porous material with a higher permeability when compared to silica sand and silica beads. Therefore, it is easier for binder to enter the matrix during 3D printing which leads to an abnormally high moisture content of the GP specimen. The mass loss of the GP specimen approaches 3 g after thermal curing, which suggests that the moisture content reaches 15%, while the moisture content of CSB and SS specimens is less than 5%.

The UCS of the cured 3DP rocks and those natural weak-cemented sandstones are distributed in a similar interval (Bertuzzi and Pells 2002). In addition, UCS data of 3DP rocks reported in previous studies (Tian et al. 2017; Kong et al. 2018a; Perras and Vogler 2019) are similar to the results obtained in this study. However, despite the improvement in strength due to the post-processing of rock analogs, they still can't yield the strength of most high-stress rocks (e.g. granite).

Table 3. Summary of UCS test results on 3DP rocks

Category	Ultimate load /KN	Compressive strength /MPa	Young's modulus /GPa	Poisson's ratio
CSB-1	3.005	6.16	1.087	0.154
CSB-2	4.058	8.16	1.255	0.136
SS-1	1.963	4.26	0.769	0.163
SS-2	2.426	6.11	0.792	0.161
GP-1	1.863	3.74	0.302	0.194
GP-2	4.477	9.18	0.432	0.175
Bertuzzi and Pells (2002)		5~20*	0.35~1.2	0.14-0.25
Tian et al. (2017); Kong et al. (2018a); Perras and Vogler (2019)		2.26~8.55 [#]	1.24~3.81	0.16-0.3
Gomez et al. (2019); Primkulov et al. (2017)		12~19	1.7~2.99	0.1~0.19 ^{\$}
* represents the distribution interval of natural sandstone UCS values reported in literatures.				
[#] represents the distribution interval of 3DP rocks UCS values reported in literatures.				
\$ represents the results obtained with a confining pressure and unconfining pressure conditions.				

3.1.2 Failure mode analysis

According to previous studies (You and Hua 1998), four types of failure modes exist for rocks under uniaxial compression: (1) multiple failure planes with a main plane throughout the specimen; (2) two shearing-planes throughout the specimen, i.e. "X" type of shear failure; (3) a failure zone with cone shape at ends of the specimen, i.e. "end cap" cone (Hodder et al. 2018b); (4) a local stress-concentration, i.e. "buckling failure". The failure mode of 3DP rocks under uniaxial load is presented in Fig.6. For convenience, we have re-drawn the crack pattern of 3DP rock to illustrate the failure mode.

The failure pattern after UCS test shows that CSB and SS specimens mainly experience tensile

failure with a shear crack at end of the core, while the GP specimen fails through shear. For CSB and SS specimens, there are multiple failure planes, but the main crack exists throughout the specimen (Fig.7a and 7b). For the GP specimen, there are two intersecting shearing-planes and a shear-expansion phenomenon (Fig.7c). In the UCS test of a natural red sandstone (Fig.7d), the compression-shear damage is the main failure mode. A shear crack plane exists at a certain inclination angle with the loading direction. During the compression of the natural rock, there are many rock particles falling from the failure plane, and this phenomenon is also observed in the CSB and SS specimens' failure. CSB and SS specimens both have shown tensile and shear damage phenomenon during compression. Although the GP specimen exhibited shear-damage behavior, there is large compression deformation prior to failure which is not consistent with the deformation characteristics of natural sandstone, but more like consolidated soil material. Thus, CSB and SS specimens can properly simulate the failure of the natural red sandstone tested in this study and some weak-cemented rocks reported in other literature (Bertuzzi and Pells 2002; Tian et al 2017; Perras and Vogler 2019) in terms of strength and failure characteristics.

Fig. 7 Photographs and re-drawn schematic of failure pattern of (a) CSB, (b) SS, (c) GP 3DP specimen and (d) natural rock after uniaxial compressive test

3.1.3 Triaxial compressive strength (TCS)

Triaxial testing is completed to investigate the triaxial deformation characteristics of 3DP rocks, as well as the natural red sandstone. The 3DP rock specimens used in the triaxial testing are the same batch with the UCS test, i.e. 25 mm in diameter and 50 mm in length (Since the thermal curing effect on sample's strength has been investigated in UCS test, the specimens used in TCS are all uncured). The test results of TCS are summarized in Table 4, and the strength envelope, stress-strain curve, and image of failure mode are presented in Fig. 8.

Table 4. The results of triaxial compressive strength of 3DP specimens

Specimen	Confining pressure /MPa	Triaxial strength /MPa	Inter-friction angle /°	Cohesive /MPa
CSB-1	2	6.02		
CSB-2	4	12.13	23.07	0.86
CSB-3	6	15.41		

SS-1	2	7.23		
SS-2	4	11.74	21.14	1.08
SS-3	6	15.77		
GP-1	2	4.12		
GP-2	4	7.31	4.78	1.09
GP-3	6	9.33		
Natural 1	4	40.9		
Natural 2	8	51.2	29.15	7.82
Natural 3	12	61.4

Fig. 8 The failure envelope, Mohr's circle, and stress-strain curve of (a) CSB, (b) SS, (c) GP 3DP

specimens, and (d) natural sandstone

CSB and SS specimens have a larger inter-friction angle of 23.07° and 21.14° (for GP it is 4.78°), while the SS and GP specimens have a larger cohesive force value of 1.08MPa and 1.09MPa (CSB is 0.86MPa). The results indicate that the triaxial compressive strength of the 3DP rock increases linearly with increasing confining pressure. For the SS and CSB specimens, the volumetric strain increases with the increase of load (the volume decreases). However, for the SS specimen, before approaching the yield point, there is a slight volumetric expansion (Fig.8b). Once plastic deformation begins, cracks propagate until the specimen is broken into multiple fragments (particularly for the SS specimen). However, for the GP specimen, despite the volumetric compression at the initial stage, the radial strain increases rapidly after the yield point, which leads to a continuous increase of the sample volume. The GP specimen has a strong plastic rheological characteristic when compared to the CSB and SS specimens. The TCS of natural sandstone is obviously larger than that of 3DP rocks. The inter-fraction angle and cohesive force of natural sandstone are 29.15° and 7.82MPa, respectively. The natural sandstone shows shear failure of single inclined plane. Besides, there are no obvious tensile cracks near the shear failure surface, and the failure surface is relatively smooth and regular.

3.2 Microstructure and Transport Characteristics of 3DP Rocks

To understand the microstructure of 3DP rocks and reveal the mechanism behind strength reduction compared to natural rocks, micro-CT imaging was used to extract the pores and grains for a quantitative characterization of microstructure. SEM-EDS analysis was also used to identify the composition and bonding form of the 3DP specimens.

3.2.1 Pore-throat size and coordination number distribution

Micro-CT scanning has been regarded as a powerful approach to visualize the microstructure of porous media (Dong 2007; Song et al. 2017). In this section, images of 3DP rocks were captured via CT scanning (Table 5) and compared to the microstructure of Berea sandstone (from Dong 2007). The cross-sections of raw CT images, binarization images, extracted PNM, and calculated parameters (e.g. pore size, throat size, and coordination number) of 3DP rocks and Berea Sandstone were shown in Fig.9.

Fig. 9 Micro-CT images (a1, b1, and c1), segmented images(a2, b2, and c2), 2D view of extracted pore network (a3, b3, and c3), pore diameter (a4, b4, and c4), throat diameter (a5, b5, and c5), and coordination number distribution (a6, b6, and c6) of (a) CSB specimen; (b) SS specimen; and (c) Berea sandstone (Dong 2007). a7-c7 and a8-c8 are entire and sub volume 3D view of pore network used to give a very clear spatial structure.

Table 5. Detailed information of CT datasets of 3DP rock and natural sandstone

Category*	Resolution / μm	Size /pixel	Sub-volume
CSB	4.366	992*1012*992	800 ³
SS	5.398	992*1012*1012	800 ³
Berea			
sandstone	4.956	950*950*1020	400 ³
(Dong 2007)			

*note: the uncured specimens, i.e. original specimens are imaged to analyze microstructure.

The CT images indicate that the grain packing of 3DP rocks exhibits a relatively loose texture

(particularly the SS specimen) compared to Berea sandstone (Fig.9b and 9c). For the SS specimen, the matrix is silica sand with uniform grain sizes and angular shapes that leads to a relatively loose texture and high porosity (helium porosity is 52.18%) compared to the CSB specimen (helium porosity is 40.15%). For the CSB specimen, the matrix comprises silica beads with different grain sizes, which means a better gradation (the space between large grains can be filled by small grains) and can result in a tighter packing than in the SS sample (Fig. 9a). Despite the loose texture of the SS specimen, it has a closer affinity to Berea sandstone than that of the CSB specimen in terms of morphology, topology, shape of pores, and bonding between grains. For the CSB specimen, coated silica beads are a kind of artificially, post-processed material (a fully spherical granular medium), which have the surface roughness of grains different from natural rocks.

To quantify the microstructural characteristics of 3DP rocks including porosity, pore size, and connectivity, a pore network analysis was conducted on CT images (Fig. 9). The extracted pore network model indicated a good connectivity and high porosity of the SS and CSB specimens (CT image porosity is 47.37% and 38.2% for SS and CSB specimens, respectively). The average values for pore diameter, pore-throat diameter, and coordination number for the SS and CSB specimens were 92.35 μm , 40.17 μm , 9.45, and 43.87 μm , 17.81 μm , 8.13, respectively. The pore-throat size of the SS specimen was more than two times that of the CSB specimen, which confirmed that SS had a relatively loose texture (Table 6).

Table 6. Calculated pore-throat parameters of 3DP rock and natural sandstone

Category	Pore size / μm	Pore throat size / μm	Coordination number	Experimental porosity /%	Image porosity /%
CSB	43.87	17.81	8.13	40.15	38.2
SS	92.35	40.17	9.45	52.18	47.37
Berea sandstone (Dong 2007)	17.26	10.94	4.77	N/A	24.6

3.2.2 Permeability and wettability

The permeability and wettability have also been investigated to characterize the transport behavior of 3DP rocks. In our previous study (Song et al. 2020), the contact angle was measured by sessile drop method at the sample surface of 3DP rocks, which indicated that the 3DP rocks were strongly oleophilic and weakly hydrophilic (average contact angle test by oil and water was 23.87° and 69.29°). Previous study has also investigated the hydraulic properties of 3DP rocks. Ardila et al. (2019) reported the value of contact angel measured by water at the surface of printed sandstone with 10% binder saturation ranged from 73° ~93°, which was slightly higher than the value obtained in this study. Besides, the strong oil-wet behavior of 3DP rocks was confirmed both in this study and literature.

The absolute permeability of 3DP rocks was derived from a single-phase flow simulation which is directly computed on micro-CT images obtained in section 2.3. The direct flow simulation approach has been described in detail in our previous study (Song et al. 2019). The streamline-based velocity fields of the CSB and SS specimens are shown in Fig.10.

Fig. 10 Streamline based velocity field (a) CSB specimen and (b) SS specimen

The computed absolute permeability of the CSB and SS specimens was 9840.2mD and 12580.5mD, respectively. The absolute permeability of Berea sandstone was 3950mD. The computation results showed that 3DP rocks were more permeable than Berea sandstone, and that the SS specimen was more permeable than the CSB specimen. Besides, since the absolute permeability obtained in this study was derived from a single-phase flow simulation under unconfining pressure condition, so the value was much higher than those of permeability reported in Ardila (2018). (The maximum permeability was around 2000mD at the lowest effective stress condition, 500kPa). The results of permeability verified that the SS sample had a higher pore-throat size and coordination number than the CSB sample, and obviously was much higher than those in the Berea sandstone (Fig. 9). The streamline field normalized from local velocity confirmed the high connectivity and homogeneous pore structure of the SS specimen. With the same configuration of streamlines (e.g. number, length, width, and step size), the density of streamlines in the computation region of the SS specimen was much higher than that of the CSB specimen. The distribution of streamlines was also more uniform in the SS specimen (Fig. 10b). The 3DP rock had higher permeability than natural Berea sandstone, but also a higher oil recovery during waterflooding experiment which was confirmed in our previous pore-scale multiphase flow experiments conducted on 3DP rocks (Song et al. 2020).

3.2.3 Pore type, grain bonding form and binder distribution

SEM scanning and EDS analysis were performed on 3DP rocks to investigate the pore type, binder distribution, and grain bonding (Fig. 11a, 11b, and 11c). By extracting the distribution of the grey values of a local zoomed-view SEM image (Fig. 11d and 11e), the grey value of the SS and CSB specimens had a similar distribution feature. The grey value of the pore phase was generally lower than 50, and the binder phase ranged from 100 to 150, while the grain phase was larger than 200. For the CSB specimen, the grey value of the sintered part of binder was significantly lower than those of un-sintered parts. However, the grey value distribution of the GP specimen was undisciplined compared to the other two specimens. There were no clear boundaries of grey value distribution between different phases shown in Fig. 11f. The determination of the critical threshold of grey value provided a reference for composition identification (Robinet et al., 2012; Ma et al., 2015, 2016).

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Fig. 11 SEM image of (a) SS, (b) CSB, and (c) GP 3DP specimens and grey value distribution of (d) SS, (e) CSB, and (f) GP 3DP specimens

To further analyze the binder distribution in 3DP specimens, the EDS test is performed simultaneously during SEM scanning to identify the binder phase. Since the binder used in this study is

made of organic materials (Furfuryl and Phenolic alcohol-based resin), carbon can be used as a marker to identify the binder to separate it from the pore and grain phases (Kong et al., 2019). Besides, the X-ray peak of carbon do not conflict with those of other elements during EDS test, which provides a convenience to detect binder distribution using EDS. The EDS test and segmentation results of the SS specimen are shown in Fig.12. The region of the selected SEM image (Fig.12d) indicates that the specimen experiences a slight compaction with a relatively loose texture. The segmentation results show that the binder mainly spreads on the surface of grains (Fig. 12e). The EDS test of selected points also indicates that the binder exhibits nonuniform distribution on the grain surface (Fig.12b, 12c, and 12h) (by comparing the relative concentration of C and Si). Since the phenomenon which is expected to fill the pore space by binder is not occurring, the intergranular pore is the main type in the SS specimen, as shown by the blue arrow in Fig.12a and 12g.

Fig. 12 Extracting of (e) binder distribution and (f) pore space of SS specimen combining the EDS analysis. (a) and (g) are selected areas to perform EDS test from (d) a large area; (b), (c), and (h) are the results of EDS test of the selected points P1, P2, and P3 in the image (a) and (g), respectively.

The EDS test results of the CSB specimen differ from the results of the SS specimen due to the matrix material (Fig.13). The silica beads are fully coated with phenolic resin, and the binder between grains is sintered by the laser. Hence, the main object to detect are the connecting bridges between adjacent grains, e.g., the sintered part of coated resin located in the red ellipse (Fig.13e). The results of four EDS test points indicate that the relative concentration of carbon is the highest at both grain surface and inter-grain (Fig.13b, 13c, 13h, and 13i). The lowest value of the test points is even higher than 57% (e.g. the relative atomic mass of carbon), thus there is no pore filling phenomenon that is expected to occur in the CSB specimen. The intergranular pore is also the main pore type (Fig.13f). However, the texture of the GP sample is tighter when compared to the SS specimen due to the different size of silica beads used. The space between large grains is filled by small grains (Fig.13a, 13d, and 13g). Hence, the tighter texture leads to a higher strength of the CSB specimen when compared to the SS specimen. Nevertheless, the rate of the strength improvement of the CSB specimen after thermal curing is the lowest

among three types of specimens. This is because the temperature of thermal curing is obviously lower than that of the laser, hence the contribution of binder curing on strength enhancement is limited. The increase of strength mainly comes from the decrease of moisture content in the specimen.

Fig. 13 Extracting of (e) binder distribution and (f) pore space of CSB specimen combining the EDS analysis. (a) and (g) are selected areas to perform EDS test from (d) a large area; (b), (c), (h) and (i) are the EDS results of selected points P1, P2, P3 and P4 in the image (a) and (g), respectively.

The microstructure of the GP specimen is different from the SS and CSB specimens. Due to the extremely small particle size, the binder distribution in the GP specimen is quite different from that of the SS and CSB specimens (Fig.14d and 14e). The EDS test has also confirmed this phenomenon (Fig.14b and 14c), in which the concentration of carbon is over 95%. The GP specimen has an extremely low porosity of 9.27%, and the pores have a highly irregular “honeycomb-like” shape (Fig.14a).

The microstructural characterization of the GP specimen has given a reasonable explanation of the strong plastic expansion during compression. Due to the well-mixed binder and gypsum powder, the moisture content of the GP specimen is the highest among the three types of specimens. Therefore, the decrease of moisture content after thermal curing is also the highest, which results in a substantial improvement of strength. Moreover, the high moisture content can explain why the uncured GP specimen has the lowest compressive strength, and with the lowest helium porosity (9.27%).

Fig. 14 Extracting of (e) binder distribution and (f) pore space of GP specimen combining the EDS analysis. (a) is the selected area to perform EDS test from (d) a large area; (b) and (c) are the EDS results of selected points P1 and P2 in the image (a), respectively.

The SEM and EDS test results indicate that different microstructure, binder distribution and bonding form of 3DP rock analogs can lead to different mechanical and failure response. In this study, the bonding type is the main reason for the failure response of the three samples. As shown in the Fig. 15, the bonding neck broken marked in red arrow of CSB and SS samples will lead to a brittle failure. The blue arrow marked in the GP sample shows many irregular “honeycomb-like” pores. In addition, compared to the silica sand and coated sand beads which are rigid granular materials, the porous gypsum powder will lead to a strong compression aggregation and plastic expansion.

Fig. 15 Different bonding form of 3DP rock analogs

3.3 Effect of Micro Failure Mode on Compressive Strength of 3DP Rocks

To investigate the effect of the different microscopic failure modes on macroscopic compressive strength, specimen fragments were collected after the UCS test to capture the microstructural change via SEM imaging (Fig.16). Since the rock has been non-conducting, platinum (Pt) was deposited on the surfaces to improve imaging via a multifunctional ion sputtering system (LEICA EM ACE 200) at Southwest Petroleum University in Chengdu, China.

Fig. 16 SEM images of (a) SS, (e) CSB, and (h) GP 3DP fragments after UCS test.(b-d), (f-g), and (i-j) represent different microscopic failure mode, i.e. grain cracking, grain stripping, and exfoliating; (k) the failure pattern of the GP specimen observed in the previous study (Zhou and Zhu, 2018)

A noticeable grain agglutination can be observed in the red dashed box in the SEM images of the SS and CSB specimens (Fig.16a and 16e). The GP specimen exhibits exfoliation in a slice or wedge shape along with grain agglutination (Fig.16h), which leads to its strong rheological plasticity. The grain cracking and stripping phenomena are observed in the SS specimen. As mentioned above, the binder in the SS specimen accumulates on the grain surface. Therefore, the binder cracking between grains results in the microscopic failure mode via grain stripping, while the squeezing and dislocation between grains lead to grain cracking (Fig.16d and 16c). For the CSB specimen, grain stripping is the main failure mode, which is determined by the bonding form of grains (Fig.16f and 16g). The microscopic failure mode of the SS and CSB specimens determines the macroscopic failure mode of tensile failure caused by grain cracking and stripping. Grain stripping results into the multiple micro-cracks, and with the evolution and propagation of the local cracks, where a main failure plane throughout the specimen is formed. This is in good agreement with the failure pattern observed in the UCS testing.

The SEM investigation of the 3DP fragments collected from triaxial testing is also conducted. The results show that the microstructure and microscopic failure behaviors of 3DP rocks after triaxial compression differs from that of uniaxial compression. First, the phenomenon of particle compression and aggregation is more obvious than that of uniaxial compression (highlighted by red circle in Fig. 17). Second, the cracking phenomenon of bonding neck between particles is much more serious (highlighted by red arrow in Fig. 17). In the uniaxial compression, the bonding neck between particles tends to peel off at one cracking point. However, there are more than one cracking points between particles in the triaxial compression, which also explains that the samples after triaxial compression have much higher crushing degree and more stripping particles around sample.

The microscopic failure mode of the GP specimen is different from the SS and CSB specimens. Due to the strong rheological plasticity caused by the unique bonding form of the powder and high bounding capacity between the binder and gypsum powder. The microscopic failure mode furtherly confirms the strong rheological plasticity of the GP specimen. Besides, it can also reveal the unique failure pattern of

the GP specimen observed in Zhou and Zhu (2018), where the X type conjugate shear occurred in the middle area of the specimen (Fig. 16k). Moreover, due to the limited effect of temperature on the curing depth during thermal processing, the moisture content inside the specimen is still much higher than in the areas near the specimen surface. Therefore, the differential on moisture content leads to failure primarily near the surface of the specimen.

Fig. 17 SEM results of 3DP rock fragments after triaxial compression

4. Conclusions

A comprehensive experimental investigation of mechanical behavior, microstructure, and transport characteristics of 3DP rocks was conducted using UCS and triaxial testing, micro-CT imaging, and SEM-EDS analysis. 3DP rocks had properties favorably comparable to natural rocks in terms of mechanical behavior (compared to weak-cemented red sandstone and low-buried Sydney sandstone) as well as microstructural and transport characteristics (compared to Berea sandstone):

(1) Among the original 3DP rocks, the CSB specimen (i.e. manufactured by SLC technique with coated silica beads) had the highest compressive strength (6.16MPa). Thermal curing could effectively enhance the compressive strength of 3DP specimens, and the increase rate was 32.4%, 43.4%, and 145% for CSB, SS, and GP specimen, respectively.

(2) The failure modes of the SS and CSB specimens were the tensile failure and local crippling, while the GP specimen was mainly experiencing shear damage and showing plastic-rheology behavior. In addition, the impacts of the microscopic cracking mode on the macroscopic failure of 3DP rocks were investigated and analyzed by SEM images. The CSB and SS specimens had a similar microscopic failure mode, i.e. binder cracking between grains, which resulted in a tensile and compressive-shear damage.

(3) The PNM analysis indicated that the CSB and SS specimens had a higher porosity (40.15% and 52.18%), pore-throat diameter (43.87 μ m/17.81 μ m and 92.35 μ m/40.17 μ m), and connectivity (8.13 and 9.45) compared to Berea sandstone (24.6%, 17.26 μ m/10.94 μ m, and 4.77 for porosity, pore-throat diameter, and coordination number, respectively). The intergranular pores were mainly developed in the SS and CSB specimens, while the GP specimen formed irregular “honeycomb-like” pores. 3DP rocks were more permeable than Berea sandstone.

(4) Considering the mechanical and pore-structure properties of 3DP rocks, the CSB and SS specimens are suitable for validating transport behavior since the microstructural characteristics are similar to highly permeable sedimentary rocks. Due to the strong plasticity, the GP specimen with specific post-treatment can be used to simulate the large deformation and creeping failure mode of highly stressed soft rock reported in Wu et al. (2019).

Despite the 3DP rocks have a lower compressive strength, this property can help decline the load requirements, especially in the physical modeling experiments when considering a similarity theory when a de-scaling treatment between the experimental model and the engineering prototype is needed. However, the properties related to the mineral composition of the 3DP rock, e.g. the wettability, water-absorbing property, need to be improved in future studies.

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Declaration of Competing Interest

The authors declare that there are no competing financial interests with any other people or groups regarding the publication of this manuscript.

Availability of data and material

All the data and materials used in this paper are available from the corresponding author upon request.

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