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Technical Paper

Analyze of scanning electron microscope images using deep learning for calcium carbonate polymorph detection

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Abstract

Calcium carbonate is a material involved in various types of industries, such as medical, electronics and environmental. In the oil and gas production, calcium carbonate precipitation can occur in the production well and also in upstream processing units, such as three-phase separator and heat exchanges. Calcium carbonate can occur in three main polymorphs: calcite, vaterite and aragonite. Scanning electron microscope (SEM) is used to investigate crystallization of calcium carbonate and to understand its mechanism for different environmental condition. Additionally, the calcite polymorph is often considered in the literature as a more tenacious and hard to remove scaling. In this work, we used convolution neural networks to detect crystals in the vaterite form of SEM images for an automatic identification that can be used to infer the ratio of vaterite to calcite in the sample.

Keywords: calcium carbonate. scanning electron microscope. deep learning. computer vision

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1. Introduction

Calcium carbonate is a material used in several types of industries, such as medical, electronics and environmental. In the oil and gas production, calcium carbonate precipitation can occur in the production well and also in upstream processing units, such as three-phase separator and heat exchanges. Calcium carbonate can occur in three main polymorphs: calcite, vaterite and aragonite. The calcite polymorph is often considered in the literature as a more tenacious and harder to remove scaling. Hence, understanding the operational conditions that stabilizes a certain calcium carbonate polymorph is an interesting topic of research in the petroleum industry.

Scanning electron microscope (SEM) is commonly used for characterization of solid materials as a method to gain insights and understand the phenomena under investigation. This characterization technique allows a quick manner for mineralogical assessment and identification. The method is based on an electron microprobe that uses electrons to scatter or reflect off the sample to produce its image, which is processed by image analysis software to produce the final image. Features as mineral abundance and chemical composition, grain size, grain morphologies, textures, liberation and association can be investigated. Example of applications in the petroleum industry are: analyze of encrustation material and investigation of failed equipment, such as gears and bearings, and for wear damage as abrasion, adhesion and corrosion. The measurement equipment provides high-resolution images which is often inspected in a visual manner by the experimenter. As the number of images to be inspected is increased, this procedure can be time demanding. Additionally, the captured images can contain features and patterns that may not be identified by the investigator.

Traditional image processing involves preprocessing, feature extraction, feature reduction and classifier detection and are largely dependent on experts in image processing to incorporate insights and nuances for each particular application in the processing pipeline. Convolutional Neural Networks are an emerging technique that accelerated the progression of computer vision applications (Huang et al., 2020). General applications as face recognition, object detection and classification in a scene and self-driving cars are now allowed. More specific use cases in industrial environment have also being developed, such as surface defect detection (Huang et al., 2020), crystallization monitoring (Gao et al., 2018) and industrial egg production classification (Shimizu et al., 2017). The computer vision applications falls mainly in the following algorithm categories (Elgendy, 2019): (i) image classification, which labels imagens from a set of predefined categories; (ii) object detection and localization, a task that have received great attention and established important networks, such as YOLO, SSD and Faster R-CNN; (iii) semantic and instance segmentation, in which the task is to classify the image in the pixel level; (iv) image captioning, which uses Recurrent Neural Network; and (v) style transfer and image creation for enhanced art generation.

In this work, we used convolution neural networks for calcium carbonate polymorph identification based on crystal morphologies and textures, detecting crystals in the vaterite habit of SEM images for an automatic identification that can be used to infer the ratio of vaterite to calcite in the sample. This tool can auxiliate an analyst in the evaluation of elevated number of images. Additionally, compared with the reference polymorph quantification method of DRX measurement with Rietveld refinement algorithm, the sample is preserved and further information can be retrieved, as mean particle size.

2. Methodology

The present work is composed of experiments for the generation of calcium carbonate crystal by mixing of calcium chloride and sodium bicarbonate and by the image processing for vaterite crystal detection.

2.1. Material and Methods

Experiments consisting of calcium carbonate precipitation were performed by mixing calcium chlorine with sodium bicarbonate. Sodium bicarbonate solutions (NaHCO3) at 0.15 mol/L and calcium chloride dihydrate (CaCl2 · 2H2O) at 0.05 mol/L were diluted in deionized water by reverse osmosis and stored in glass vessels with open contact to the atmospheric air. The open system allows the thermodynamic balance for the carbonate chemical species. The prepared solutions are stored at a controlled temperature of 21 to 24˚C and with monitored pH, conductivity and temperature variables. When each solution reaches the pH close to the open system equilibrium, which is 7.5 for the calcium chloride and 8.6 for sodium bicarbonate, they are ready for the mixture and start of the experiment. The saturation index for this mixture is calculated as 3.4.

Before the beginning of the experiment, the pH, conductivity and temperature values of each solution are measured with a Metrohm 914 pH/Conductivity meter. The experiment is started with the transfer of 50mL of NaHCO3 solution to a graduated cylinder. The CaCl2 solution was transferred to a beaker containing a submerged Salobetter pump connected to a plastic tube with a diameter of 1/8 ”and a length of 130 cm. After the end of the mixture and precipitation, the suspended particles were filtered and the filter paper with the moisted material is placed in a dehumidifying chamber, where it remains for 1 hour until the entire sample is dried. Finally, the paper is removed and the sample is collected in eppendorf tubes and properly labeled.

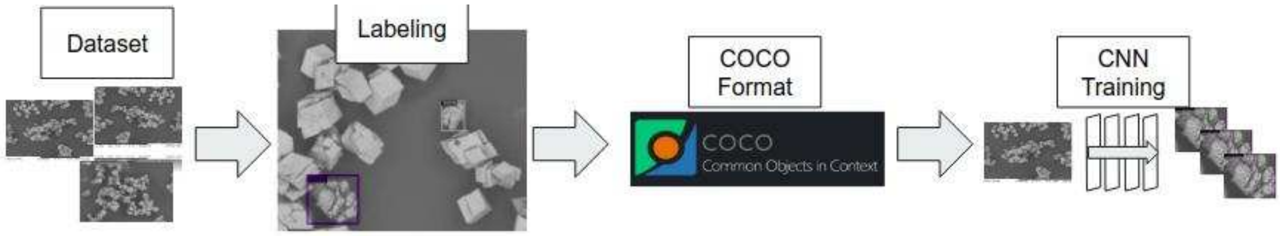
The collected solid undergoes Scanning Electron Microscopy (SEM) analysis by the Hitachi TM3030 Tabletop Microscope equipment. The analysis of electron microscopy begins with the preparation of the sample where a small piece of carbon paper is separated and coupled with the equipment's sample holders, its surface being suitable for the crystal not to detach from it. Then, a small amount of sample is inserted into the equipment to start capturing the micrographs in 3 randomly chosen regions. The equipment, which uses high energy electron beams to generate high definition images, was operated in a low-voltage of 5kV for higher constant and with a 500x amplification for the image capture.

2.2. Image Detection with Convolutional Neural Networks

Image detection and classification using Convolutional Neural Networks have disrupted computer vision applications that was not possible using traditional methods of feature engineering. One of the recent developments of convolution neural networks is the method Mask R-CNN. It consist in main two steps: the first scans the input image and generates proposals using a less computational demanding neural network layers denoted as Region Proposal Network, which reduces the detection scope; the second step is used for refinement and classification and can output the results as masks, bounding box and labels. In general applications, it can for instance detect and classify persons, cars and objects in a scene, giving a confidence factor for each detected item.

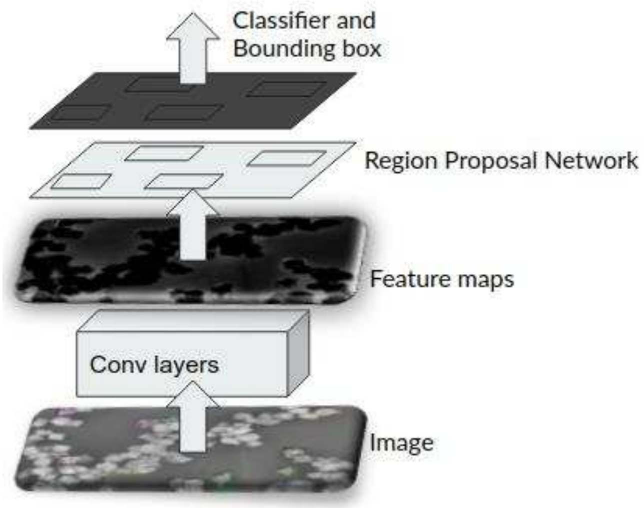
The image processing pipeline used in this work is schematized in Figure 1. The image dataset is composed of 23 original images, which were augmented with rotation and flipping to 276 total images and 4101 total object instances. The dataset was splitted as 75% for training and 25% for testing. The vaterite instances were labelled in the Supervesity platform. The vaterite polymorph was the only category defined, because the SEM images were in the majority predominantly composed of aggregated calcite which dispersed vaterites, hence allowing a simpler labelling procedure. The labelled images and their annotations were exported and a conversion script was created to conform to the Common Object in Context (COCO) standard. COCO is a large-scale dataset collection for computer vision applications and a standard for deep learning packages. In our study, the annotated COCO dataset was used as input to the Detectron2 library (Wu et al., 2019).

Figure 1 – Image processing pipeline used for vaterite identification in SEM images (author's own production).



The Detectron2 package (Wu et al., 2019) is provided as an open source tool from Facebook Artificial Intelligence Research (FAIR) built on top of pytorch framework (Paszke et al., 2019). The package uses state of the art deep neural networks, such as Faster Regional Convolutional Neural Networks (R-CNN) and Mask R-CNN both for object detection and pixel level segmentation. In our case, the Faster R-CNN method was employed for object detection. This technique can be separated into the stages: (i) initially a CNN is used for feature map; (ii) a Region proposal network provides region of interest proposals; (iii) the proposals are normalized to the same size by a region of interest pooling layer; and, finally, (iv) the proposals are classified and localized with bounding box by a fully connected layer with softmax and regression layers (Sharma, 2018). A schematic representation of Faster R-CNN is shown in Figure 2.

Figure 2 –Schematic representation of Faster R-CNN (adapted from Sharma (2018)).

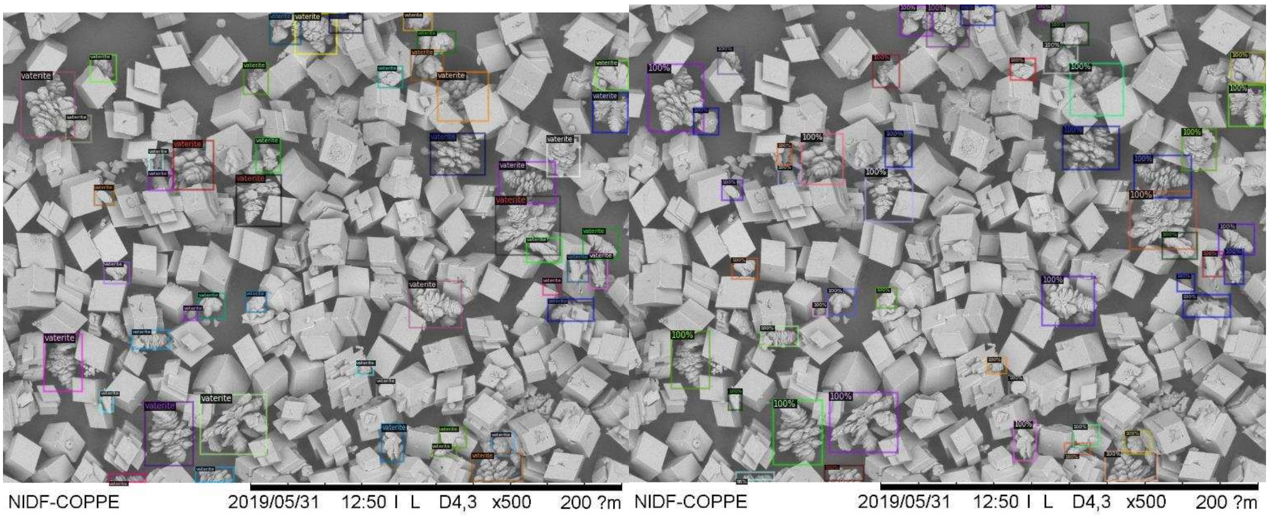


The object detection and localization is performed using standard Detectron2 configuration of a Faster R-CNN with: a ResNet backbone network with a convolution layer and 4 residual blocks, a Region Proposal Network with 256 batch size per image and a Region of Interest pooler layer using feature maps from last residual block of the backbone. The network was training with default detectron2 trainer, which was built to simplify standard model training workflow and uses a stochastic gradient descent optimizer. We have used a base learning rate of 0.01 with a nesterov accelerated gradient momentum of 0.9. The neural network was trained using the Google Colaboratory platform with NVIDIA T4 Graphics Processing Unit.

3. Results

Figure 3 illustrates a training input and prediction output for a image in the training set. Figure 3-left presents the labelled vaterite instances and Figure 3-right the prediction. The predictions were well adjusted for the training image, as all the instances were determined.

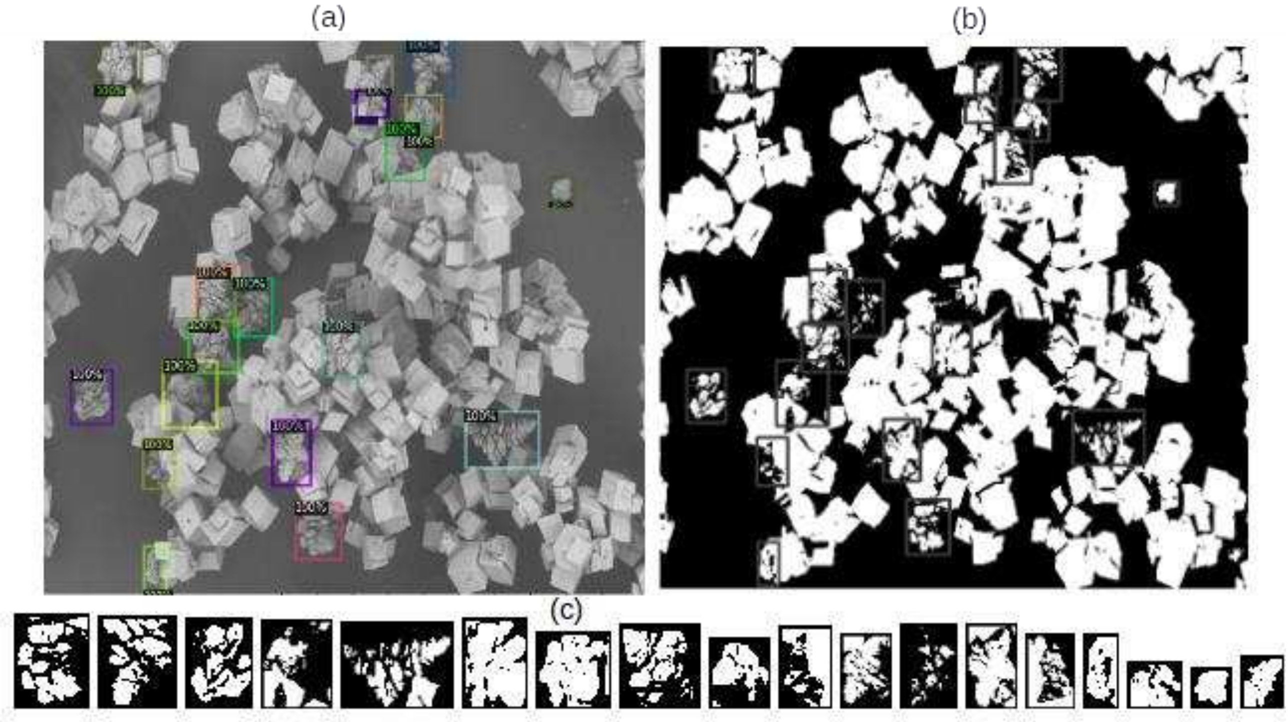
Figure 3 –Example of labeled vaterite instances (left) and bounding box prediction (right) (author's own production).



The detection was tested against a dataset of 69 images with a total of 925 vaterite instances. The Average Precision metric in this unseen dataset was 74.96 for intersection over union (IoU) from 0.5 to 0.95 with a step size of 0.05, which is denoted as COCO mAP metric. The Average Precision for IoU=0.5 was 92.61 and for IoU=0.75 was 86.09. The average prediction time for a single image was 1s on the Google Colaboratory platform.

The quantification of vaterite ratio was performed using the image from the test dataset depicted in Figure 4-a. The total amount of particle is estimated by a background removal using a black or white binary transformation in which the white pixels corresponds to particles. The amount of vaterite is obtained by analysing each predicted bounding box and performing background removal. The total amount of vaterite is considered as the sum of the number of white pixels in each bounding box. Over estimation of number of vaterite pixels can occur since the bounding box may contain both vaterite and calcite, but for simplicity we have neglected this extra pixels in a bounding box, as errors can be mitigated by part of vaterite not included in the bounding box or disregarded in the black and white transformation. An alternative would be with the use of semantic segmentation, however the labelling for this case is cumbersome and can be prohibitive for large number of images. Figure 4-b shows the image with the bounding box after the transformation and Figure 4-c enumerates the detected vaterite instances, which were used in the quantification ratio. The calculated vaterite ratio was equal to 11%.

Figure 4 – Quantification of vaterite ratio: (a) is the original image with the predicted bounding box; (b) is the image after black and white transformation; (c) is the predicted bounding box. (author's own production).



5. Concluding Remarks

This work consisted in the application of image processing techniques based on convolution neural networks to an automatic identification of calcium carbonate polymorphs in the vaterite form. An object detection Faster R-CNN network was employed and acceptable average precision was obtained in the test dataset

This approach can be a tool for investigators allowing several images to be treated in short period of time. Future developments of validation is required and can allow the quantification of vaterite to calcite ratio in the sample.

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