

QUANTITATIVE ANALYSIS AND MAPPING OF
CONCRETE SCANNING ELECTRON MICROSCOPE
(SEM) IMAGES

ELLY NUR MYAISARA BINTI MAIZUL

SCHOOL OF CIVIL ENGINEERING
UNIVERSITI SAINS MALAYSIA
2018

Blank Page

QUANTITATIVE ANALYSIS AND MAPPING OF CONCRETE
SCANNING ELECTRON MICROSCOPE (SEM) IMAGES

By

ELLY NUR MYAISARA BINTI MAIZUL

This dissertation is submitted to

UNIVERSITI SAINS MALAYSIA

As partial fulfilment of requirement for the degree of

**BACHELOR OF ENGINEERING (HONS.)
(CIVIL ENGINEERING)**

School of Civil Engineering
Universiti Sains Malaysia

June 2018



**SCHOOL OF CIVIL ENGINEERING
ACADEMIC SESSION 2017/2018**

**FINAL YEAR PROJECT EAA492/6
DISSERTATION ENDORSEMENT FORM**

Title: **QUANTITATIVE ANALYSIS AND MAPPING OF CONCRETE
SCANNING ELECTRON MICROSCOPE (SEM) IMAGES**

Name of Student: **ELLY NUR MYAISARA BINTI MAIZUL**

I hereby declare that all corrections and comments made by the supervisor(s) and examiner have been taken into consideration and rectified accordingly.

Signature:

Approved by:

(Signature of Supervisor)

Date:

Name of Supervisor:

Date :

Approved by:

(Signature of Examiner)

Name of Examiner:

Date :

ACKNOWLEDGEMENT

In the name of Allah, the most gracious, the most merciful. O Allah, I am humbly grateful for all blessings, strength and patience that you had granted to me throughout the journey of completing this research.

My first and sincere appreciation goes to my supervising lecturer, Assoc. Prof. Sr. Dr. Mohd Sanusi S. Ahamad, for all I have learned from him and for his useful advices, motivation and continuous support in all stages of this thesis. His positivity and encouragement has always inspired me to do better in this study. Not to forget, his suggestion and immense knowledge in this research has also steered me in successfully presenting this dissertation.

Besides, I would also like to convey my honest thanks to my co-supervisor, Assoc. Prof. Dr. Norazura Muhamad Bunnori and her past master's degree student, Miss Nur Liyana Zainal for providing me the invaluable research materials and scholarly inputs. In the absence of these resources, this final year project would not be accomplished and thus, it will only remain as unrealistic ideas.

Getting through this “blood, sweat and tears” thesis writing required more than academic support. Nobody has been more important to me in the pursuit of this project than the members of my family. To my dearest Ayah, Maizul Misiran and beloved Mama, Rosita Abdullah, this is for both of you. My gratitude also goes to my sweetest sister, Puteri Umyaira and the three little brothers (Naqim Eiman, Putera Iskandar and Eqbal Ibtisyam) for always looking up to your big sister. Special thanks and appreciation are also extended to my besties (Siti Syahadah, Ain Amirah, Nur Adilla, Nor Azmira) for the constant moral support throughout this project.

ABSTRAK

Penilaian mikrostruktural bahan-bahan simen kompleks telah dimungkinkan oleh pencitraan mikroskopik seperti Mikroskop Pengimbasan Elektron (SEM) dan Mikroanalisis X-Ray. Khususnya, penggunaan pengimejan SEM konkrit dan analisis imej digital telah menjadi biasa dalam analisis dan pemetaan teknologi konkrit sama ada secara kualitatif atau kuantitatif. Dalam kajian ini, enam sampel imej SEM dua dimensi (2D) telah diubah secara spasi dengan menggunakan proses *resampling* standard untuk menghasilkan imej sampel SEM Geo-rujukan. Selepas itu, imej-imej SEM yang diletak semula ini dianalisis dan plot histogram intensiti dihasilkan untuk memudahkan penafsiran visual. Analisis imej digital yang dilakukan kemudiannya adalah proses penambahbaikan dan pengenyahbunyi yang menggunakan dua kaedah penapisan iaitu penapis median dan penapis penyesuaian kotak. Imej yang ditapis semula, seterusnya menjalani proses pengklasifikasian K-Means yang tidak diselia untuk memisahkan setiap piksel individu secara berasingan sepadan dengan data spektrum. Dengan segmen spasial algoritma K-Means, kumpulan kluster yang dihasilkan telah dikaji dengan teliti sebelum meneruskan analisis terakhir. Dari data yang dihasilkan, pemetaan pengagihan ruang k-cluster dan kuantifikasi retak mikro (lompong) dilakukan. Hasil akhir gambar SEM (sampel 1 hingga 4) memperlihatkan peratusan data k-cluster yang lebih tinggi yang menunjukkan korelasi yang baik dengan komposisi unsur utama analisis EDX, iaitu Oksida (O), Silikon (Si) dan Karbon (C). Sementara itu, penilaian visual subjektif sampel imej 5 dan 6 telah mengesahkan perkembangan retak mikro pada imej SEM konkrit di mana ketumpatan retak adalah 3.02% dan 1.30%, masing-masing.

ABSTRACT

The microstructural evaluation of complex cementitious materials has been made possible by the microscopic imaging tools such as Scanning Electron Microscope (SEM) and X-Ray Microanalysis. Particularly, the application of concrete SEM imaging and digital image analysis have become common in the analysis and mapping of concrete technology either in the qualitative or quantitative manner. In this study, six samples of two-dimensional (2D) SEM images were spatially transformed using the standard resampling process to produce Geo-referenced SEM sample images. Subsequently, these resampled SEM images were analysed and the intensity histogram plot was produced to facilitate visual interpretation. The consecutive digital image analysis performed was the enhancement and noise removal process using two filtering methods i.e. median and adaptive box filter. The filtered resampled images, then undergone the unsupervised K-Means classification process to collectively separate each individual pixel corresponds to the spectral data. By spatial segmentation of K-Means algorithms, the cluster groups generated were carefully reviewed before proceeding to the final analysis. From the resulting data, the mapping of the spatial distribution of k-cluster and the quantification of micro-cracks (voids) were performed. The final results of the SEM images (sample 1 to 4) showed higher percentage of k-cluster data indicating a good correlation with the major elemental composition of EDX analysis, namely Oxide (O), Silicon (Si) and Carbon (C). Meanwhile, the subjective visual assessment of the image sample 5 and 6 has confirmed the micro-crack developments on the concrete SEM images upon which the crack density was 3.02 % and 1.30 %, respectively.

TABLE OF CONTENTS

ACKNOWLEDGEMENT	II
ABSTRAK.....	III
ABSTRACT	IV
TABLE OF CONTENTS.....	V
LIST OF FIGURES.....	VIII
LIST OF TABLES.....	IX
LIST OF ABBREVIATIONS.....	X
NOMENCLATURES	XII
CHAPTER 1 INTRODUCTION	1
1.1 Research Background.....	1
1.2 Problem Statements.....	2
1.3 Research Objectives	3
1.4 Research Scope and Limitations	4
1.5 Dissertation Outline.....	4
CHAPTER 2 LITERATURE REVIEW	6
2.1 Introduction	6
2.2 Scanning Electron Microscope (SEM).....	6
2.2.1 EDX Analysis on the SEM	10
2.3 The Concept of Digital Image Processing.....	12
2.3.1 Intensity Histogram.....	13
2.3.2 Image Enhancement.....	14
2.3.3 Image Classification.....	17

2.4	Microstructure Properties Evaluation through Digital Image Processing.....	19
CHAPTER 3 METHODOLOGY		24
3.1	Introduction	24
3.2	Software	24
3.3	Research Data.....	26
3.4	Image Processing Technique.....	28
3.4.1	IDRISI Macro Command.....	28
3.4.2	Digital Image Acquisition.....	29
3.4.3	Image Resampling	30
3.4.4	Generation of Histogram.....	31
3.4.5	Noise Filtering	31
3.4.6	K-Means Clustering	32
3.5	Mapping of Concrete SEM Images	33
3.6	Identification and Quantification of Micro-cracks and Voids	35
CHAPTER 4 RESULTS AND DISCUSSION.....		37
4.1	Overview	37
4.2	SEM Image Processing and Analysis.....	37
4.2.1	Interpreting the Intensity Histogram.....	42
4.2.2	Comparison of Median Filter and Adaptive Box Filter	44
4.3	Spatial Distribution of Concrete SEM Images	46
4.4	Micro-cracks and Voids Analysis	49
4.5	Summary	54
CHAPTER 5 CONCLUSIONS AND RECOMMENDATIONS		55
5.1	Conclusions	55
5.2	Recommendations for Future Research	57

REFERENCES 58

APPENDIX A

APPENDIX B

APPENDIX C

APPENDIX D

APPENDIX E

APPENDIX F

APPENDIX G

LIST OF FIGURES

Figure 2.1: Main signal emitted as a result of interaction between the electron beam and the sample (Goldstein et al., 2011)	7
Figure 2.2: The differences in SEM imaging techniques for (a) topographic contrast in SE mode and (b) atomic number contrast in BSE mode (Australian Microscopy & Microanalysis Research Facility, 2014).....	9
Figure 2.3: EDX spectrum from the magenta inspection field of powder sample and the figure table summarized the elemental compositions present with IL being the most abundant (Sabatini et al., 2015)	12
Figure 3.1: The flow of methodology	25
Figure 3.2: Print screen of macro command in IDRISI.....	29
Figure 3.3: Cropped window of both study areas in concrete Sample 1	35
Figure 4.1: Spatial transformation of image processing onto the SEM image starting from (a) original TIFF file, (b) IDRISI formatted image, (c) resampled image, (d) filtered image and lastly, (e) after k-means clustering	41
Figure 4.2: Percentage of intensity distribution for individual k-cluster group.....	48
Figure 4.3: Identification flow of possible deterioration evidences in concrete SEM specimens.....	51
Figure 4.4: The quantification of crack density for Sample 5 and Sample 6.....	52
Figure 4.5: Quantitative analysis and mapping of grayscale concrete SEM images	53

LIST OF TABLES

Table 2.1: Filtering techniques according to Shenbagavadivu and Devi (2013).....	16
Table 2.2: Grey levels range for image analysis (Zhao and Darwin, 1992)	20
Table 2.3: Lengths of noise, micro-cracks, and voids at the three different magnification factors (Soroushian et al., 2003)	22
Table 2.4: A review summary of digital image processing techniques used in analysing concrete digital images	23
Table 3.1: Primary information of raw concrete SEM samples (details interpretation)	27
Table 3.2: Metadata of all concrete SEM image.....	27
Table 4.1: Metadata changes for SEM specimen before and after spatial resampling..	38
Table 4.2: Summary statistics for resampled SEM images	42
Table 4.3: Comparison of minimum/maximum values of resampling images and after filtering process.....	45
Table 4.4: Micro-crack density for concrete SEM images	50

LIST OF ABBREVIATIONS

BMP	Bitmap Image
BSE	Backscattered Electrons
DIP	Digital Image Processing
EDX/EDS	Energy-Dispersive X-Ray Spectroscopy
EDXA	Energy Dispersive X-Ray Analysis
EDXMA	Energy Dispersive X-Ray Microanalysis
EPMA	Electron Microprobe
ESEM	Environmental Scanning Electron Microscope
GIF	Graphics Interchange Format File
GIS	Geographic Information System
GCPs	Ground Control Points
HPC	High-Performance Concrete
ITZ	Interfacial Transition Zone
JPEG	Joint Photographic Experts Group File
LoG	Laplacian of Gaussian
MPC	Magnesium Phosphate Cement
MRF	Markov Random Field
MCT	Micro-Computed Tomography
MIP	Mercury Intrusion Porosimetry

NC	N ormal C oncrete
OM	O ptical M icroscopy
OPC	O rdinary P ortland C ement
POGR	G rinded Concrete
POSB	S and B lasted Concrete
RAW	R aw File
SE	S econdary E lectrons
SEM	S canning E lectron M icroscopy
SVM	S upport V ector M achine
TIFF	T agged I mage F ile F ormat
UHPFRC	U ltra- H igh P erformance F iber R einforced C ementitious C omposite
USM	U niversiti S ains M alaysia
WDS	W avelength D ispersive S pectroscopy

NOMENCLATURES

$f(x, y)$	Input image
$g(x, y)$	Output (processed) image
T	Function operator on f (set of processed input data)
d_{ik}	Distance from the i -th pixel to the k -centroid
X_{in}	Vector of the i -th pixel
C_{kn}	Vector of the k -th centroid
n	Number of bands
C	Carbon
O	Oxide
Na	Sodium
Mg	Magnesium
Al	Aluminium
Si	Silicon
S	Sulphur
K	Potassium
Ca	Calcium
Fe	Iron

CHAPTER 1

INTRODUCTION

1.1 Research Background

Concrete is undeniably the prime construction material of any man-made structural applications. It is a composite material comprised of a mixture of aggregates and cement paste. From the conventional concrete recipe, many researchers have creatively modified the design mix in order to innovate higher compressive strength, better durability and more sustainable concrete. Traditionally, the assessment of concrete performance is done via its physical mechanical behaviour. However, the complexities of concrete microstructure have led to various advanced study with respect to their influence in the engineering properties of concrete. According to Akçaoğlu et al. (2004), under a microscopy perspective, concrete is defined as a three-phase composite mass comprises mortar matrix, aggregate and the interfacial transition zone (ITZ) between them. The concrete microstructure can be analysed by numerous investigation techniques such as Mercury Intrusion Porosimetry (MIP), Light Optical Microscopy (OM) together with digital image analysis, Scanning Electron Microscopy (SEM) and X-ray Micro-computed Tomography (MCT) with image processing (Hilal, 2016). However, Stutzman (2000) indicated that the modern SEM imaging and X-ray microanalysis methods can contribute to a greater finding in the complex concrete microstructure imaging. In addition, the implementation of digital image processing (DIP) on miscellaneous microscopic images has provided information quantitatively which then beneficial for any objective comparison.

Over the past years, the utilization of SEM in concrete studies is becoming more well-known. Due to the vast development in concrete technology, the SEM imaging study on the microstructure of concrete has extensively accomplished by countless researchers (Diamond, 2004; Stutzman, 2004; Paiva et al., 2017). Besides that, these researches has likewise focused on the evaluation of the ITZ of concrete specifically in the bonding strength (Diamond and Huang, 2001; Zhang et al., 2017; Vargas et al., 2017). Wang et al. (2005), in his study has observed that the micro-crack initiation of concrete exposed to elevated temperature can be further analysed by digital image processing tool. In this regard, the evaluation of high-performance concrete (HPC) is best observed under SEM that will incorporate other advance image analysis (Wang et al., 2015; Jing jun Li et al., 2016; Shen et al., 2017). Therefore, the findings from the past research, have shown that the SEM study of micro features of concrete can offer a deeper understanding of concrete nature and also, in diagnosing the concrete complexities.

1.2 Problem Statements

The SEM image is a 2D intensity map in digital domain where each image pixel on the display corresponds to a point on the sample, which is proportional to the signal intensity captured by the detector at each specific point in 8 bits (256 grey levels). Typically, these characteristics are very similar to Raster Geographic Information System (GIS) images. If the SEM can be geo-referenced or assigned coordinates (in sub millimeters), then standard GIS quantitative mapping will be possible. Likewise, quantitative measurements such as area, length, breadth and perimeter of the SEM sample contents can be performed. In addition, standard image segmentation,

enhancement and classification tools in the GIS image processing technology have been frequently used in the context of quantitative analysis of cementitious materials, on images acquired with Scanning Electron Microscope (SEM).

Although the identification of the components of the cement in SEM images can be rather accurately performed visually by an expert but for micro-scale images, there will be a need to spatially automatize the procedure. Therefore, the interest will be to investigate the capability of GIS image analysis tools in studying concrete microstructure properties with regards to mapping the spatial pattern of its clustered properties and the quantifying the existence of micro-cracks (voids) on specific SEM cement samples. Consequently, the components present in cement samples can be identified and mapped in quantitative manner.

It is anticipated that the experimental analysis of concrete SEM, incorporated by digital image processing, will optimistically leave a significant impact in the present day of concrete technology studies.

1.3 Research Objectives

The objectives of this research are as follows:

1. To apply the spatial resampling process on the concrete SEM specimens from different magnification to produce geo-referenced images.

2. To conduct a sequential digital image analysis such as intensity histogram, image enhancement, classification and segmentation on the coordinated concrete SEM images (quantitative analysis).
3. To produce classified micro-structure property distribution and micro-crack maps of sand blast and grinded concrete SEM images.

1.4 Research Scope and Limitations

This research comprises the utilization of image processing software to analyse particular concrete digital images. Two different type of concrete SEM specimens were examined, namely, sand blasted (POSB) concrete and grinded (POGR) concrete images. These image samples represent secondary data as it was obtained from a specific SEM research work that studies the effects of bonding between normal concrete (NC) and green ultra-high performance fiber reinforced cementitious composite (UHPRFC). In evaluating the secondary resources, some limitations are declared. The initial process of capturing images in the Scanning Electron Microscope (SEM) is beyond the scope of this research. However, the comprehensive understanding on the background principle of obtaining SEM data, has been thoroughly examined.

1.5 Dissertation Outline

In total, this dissertation comprises of five chapters. The overview of each chapter are as follows:

1. Chapter 1 starts with the background information of the research topic and identification of related problem. Likewise, this chapter includes the three main research objectives, research scope and limitations.
2. Chapter 2 consists of literature review on the principle of SEM and the incorporating Energy-dispersive X-ray spectroscopy (EDX) analysis. In addition, this chapter reviews the general concept of digital image processing and analysis techniques associated such as image resampling, intensity histogram, image enhancement, image classification and image segmentation. Lastly, a summary on implementation of digital image processing for microstructure properties evaluation was included.
3. Chapter 3 provides the background information regarding the image analysis software and the research data used for this study. Additionally, this chapter describes the digital image processing techniques which start from the acquisition of digital images and resampling process, followed by the procedure in generating the intensity histogram, image enhancement, image classification and image segmentation. The general procedure in quantitative analysis and mapping of concrete SEM images is also included.
4. Chapter 4 presents the results from the outcome of the digital image analysis. Subsequently, it includes critical discussion on the applied techniques. It also deliberated the output from the quantification and mapping of the processed concrete SEM images.
5. Chapter 5 includes the conclusion and recommendation for the improvement of future research work.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

This chapter consists of three main sections. The first section reviews the basic principle of Scanning Electron Microscopes (SEM) and the attachment component of Energy-dispersive X-ray spectroscopy (EDX). The second section described the general concept of digital image processing and analysis associated with microscope imaging. Meanwhile, the following sub-section delivers an overview of the quantitative analysis and mapping methods proposed, namely, intensity histogram, image enhancement, image classification and image segmentation. Lastly, the final section provides a review summary of past research work related to microstructure properties evaluation using the image processing tool.

2.2 Scanning Electron Microscope (SEM)

The Scanning Electron Microscope (SEM) is one of the most important electron beam technology inventions available particularly when it is for the examination and analysis of the microstructural characteristics of solid objects. Essentially in concrete technology, Mehta and Monteiro (2006) stated that concrete microstructure comprises of the type, amount, size, shape and distribution of phases present in a solid. The evident elements of a microstructure of a material can be vividly seen from a cross section of the material itself, whereas the finer elements are usually observed with the aid of microscope. According to Wang and Shi (2009), SEM is generally a type of electron

microscope that captured the sample surface images by scanning it with high-energy beam of electron in raster scan pattern. The process is described in Figure 2.1. Amongst the signals emitted by SEM during the interaction of the electron beam with the sample includes electrons (Auger electrons, secondary electrons and backscattered electrons), X-rays (Characteristic X-ray and Bremsstrahlung X-ray), light, heat, conducted electrons and absorbed electrons by the sample (Goldstein et al., 2011).

Consequently, SEM uses these focused beams of electrons to render high resolution, three-dimensional images that contain information on sample's surface topography, morphology, composition and other properties such as electrical conductivity (de Oliveira et al., 2017; Khursheed, 2011; Wang and Shi, 2009). Typically, SEM imaging can be characterized using secondary electrons, backscattered electrons, photoelectrons, Auger electrons and ion scattering. Two major types of imaging inclusive secondary electrons and backscattered electrons were mainly discussed and studied in many literatures.

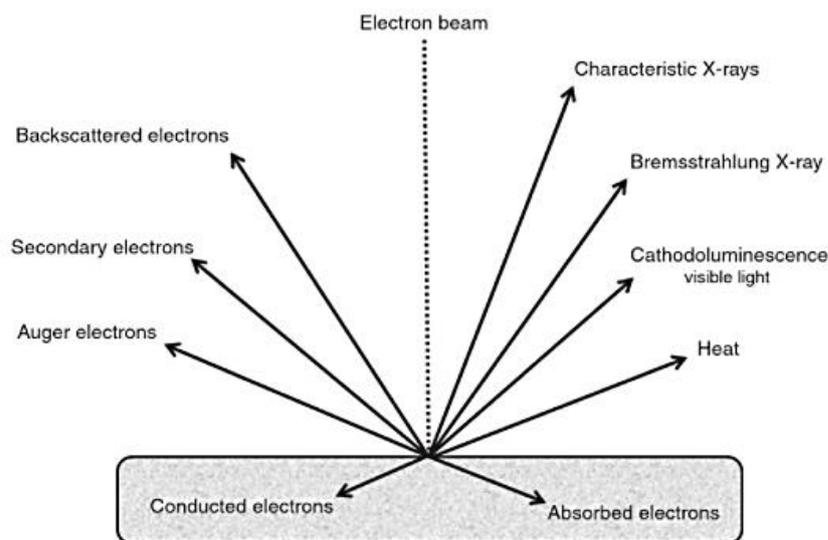


Figure 2.1: Main signal emitted as a result of interaction between the electron beam and the sample (Goldstein et al., 2011)

Secondary electrons (SE) are low energy electrons generated as ionization products from primarily high energy radiation in the forms of ions, electrons or photons. These electrons are emitted by atoms near the surface of a sample material as it becomes excited and has sufficient energy to escape the sample surface. SE imaging is beneficial for the inspection of sample topography, size and size distribution of nanoparticles as it provides good edge detail. These pointy parts are often appeared brighter in SE images than the rest of image because it produces more electrons. However, the drawback of SE imaging is that the position of SE detector plays the vital role in determining the topographical contrast differences of an image. Because of SE comes from a very close to the specimen and if the SE detector is off to the side, the resulting image will produce a shadowed picture giving a three-dimensional effect.

Backscattered electrons (BSE) are high-energy electrons (>50 eV) originating in the electron beam, that are reflected or back-scattered out of the specimen interaction volume by elastic scattering interactions with specimen atoms. BSE imaging are performed when examining for metal voiding or high atomic number impurities. The backscattered electron has an energy up to the incident beam energy and is usually very near that energy. The higher energy of BSE as compared with SE, signifies that BSE produced from deeper within the interaction volume is able to escape from the sample and collected by the BSE detector. Therefore, BSE images have lower spatial resolution than SE images. According to Scrivener (2004), the utilization of BSE imaging for cement and concrete study has shown significant potential in future research particularly on microstructure of cement hydration, the development of microstructure related to atypical hydration processes and lastly, the differences in microstructure between cement paste produced in isolation, cement paste, and in the interfacial transition zone (ITZ).

In comparison, BSE is used because their emission is sensitive to the atomic number, contrarily to the SE signal. Figure 2.2 displayed the same region observed with the two signal types. The bright regions in the SE image correspond to hills, whereas the bright regions in the BSE image relates to the presence of heavier elements. Heavier elements scatter electron more efficiently so it appeared brighter in the images. This atomic number contrast is a major advantage of the BSE detector. While as mentioned, SE detector is primarily good in defining the topographical contrast of the image.

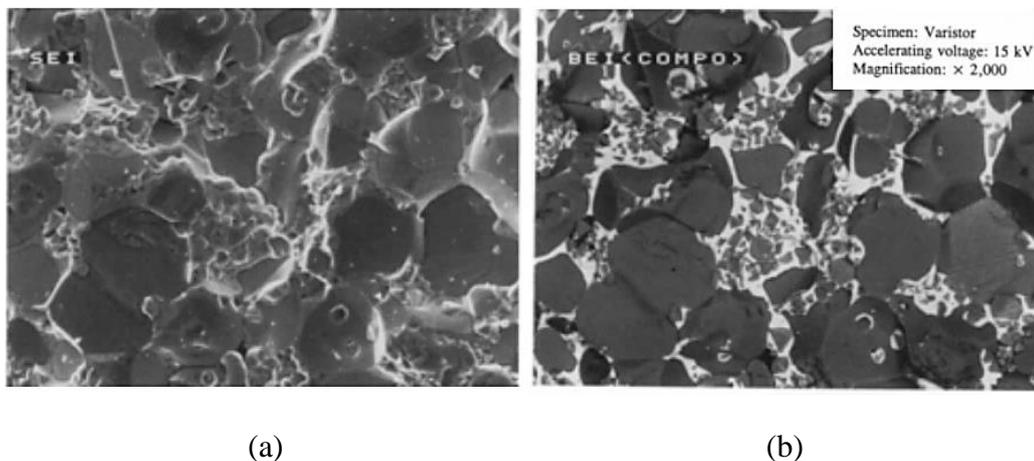


Figure 2.2: The differences in SEM imaging techniques for (a) topographic contrast in SE mode and (b) atomic number contrast in BSE mode (Australian Microscopy & Microanalysis Research Facility, 2014)

In overall, the basic idea of SEM image is the formation of 2D intensity map in analogue or digital domain where each image pixel on display corresponds to a point on the sample. This SEM imaging is a well-known non-destructive technique that generates monochrome images. The image formation is from the reflection of electron resulting from beam/specimen interaction that is proportional to the signal intensity captured by

the detector at every local point. Hence, it is vital for the user to familiarize with the fundamental principles behind SEM to set the working condition as per application.

2.2.1 EDX Analysis on the SEM

According to Goldstein (2012), the conventional components of SEM consists of the lens system, electron gun, electron collector, visual and recording cathode ray tubes and the electronics associated with them. Nevertheless, over the past years, the traditional SEM has evolved steadily with association of advanced component. Energy-dispersive X-ray spectroscopy (EDX or EDS), sometimes named as energy dispersive X-ray analysis (EDXA) or energy dispersive X-ray microanalysis (EDXMA) is the standard accessory attached on most Scanning Electron Microscopes. In general, EDX is based on detection of characteristic x-rays emitted of an element as a result of the de-excitation of core electron holes created by a high energy electron beam. EDX can simultaneously investigates the entire surface under the electron beam and thus present qualitative analysis of the vital elements on the surface as mentioned by Balendran et al. (1998). Subsequently, the analysis of characteristic X-rays emitted from the sample will then provide more quantitative elemental data.

Initial EDX analysis usually involves the generation of an X-ray spectrum with peaks at the characteristic energies for the elements present in the inspection field (Figure 2.3). Similarly, resulting spectrum is also accompanied by a tabulation of semi-quantitative data of the elemental composition in units of both weight and atomic percentage. In some cases, the EDX analysis also presents the x-ray mapping of each localised elements where the positions of specific elements emitting characteristic x-rays

within an inspection field can be assigned by unique colour. Dempere et al. (2013) studied the utilization of SEM and microanalysis particular in EDX to determine the spatial distribution of chloride in cementitious materials. The resulting data were also compared to the use of electron microprobe (EPMA) equipped with Wavelength Dispersive Spectroscopy (WDS). Similarly, De la Varga et al. (2018) examined the microstructural analysis on quantification of porosity incorporating BSE imaging by using SEM/EDX instrument. It is concluded that after image treatment process based on the EDX mapping, the mechanism behind the enhanced bond performance between grout and concrete is significant. The SEM images allow better evaluation and understanding in the microstructural inter-relation between two cementitious materials.

Meanwhile in another research, Qin et al. (2018) investigated the bond behaviour and interfacial micro-characteristics of magnesium phosphate cement onto old concrete substrate by means of SEM/EDX results. It is then concluded that the morphologies and EDX analysis of the interface between ordinary Portland cement (OPC) substrate and magnesium phosphate cement (MPC) substrate was best studied to understand the development of the bond properties between them. To summarize, the greater depth in SEM/EDX analysis have make it a powerful instrument in determining the exact elemental compositions of inspected specimen.

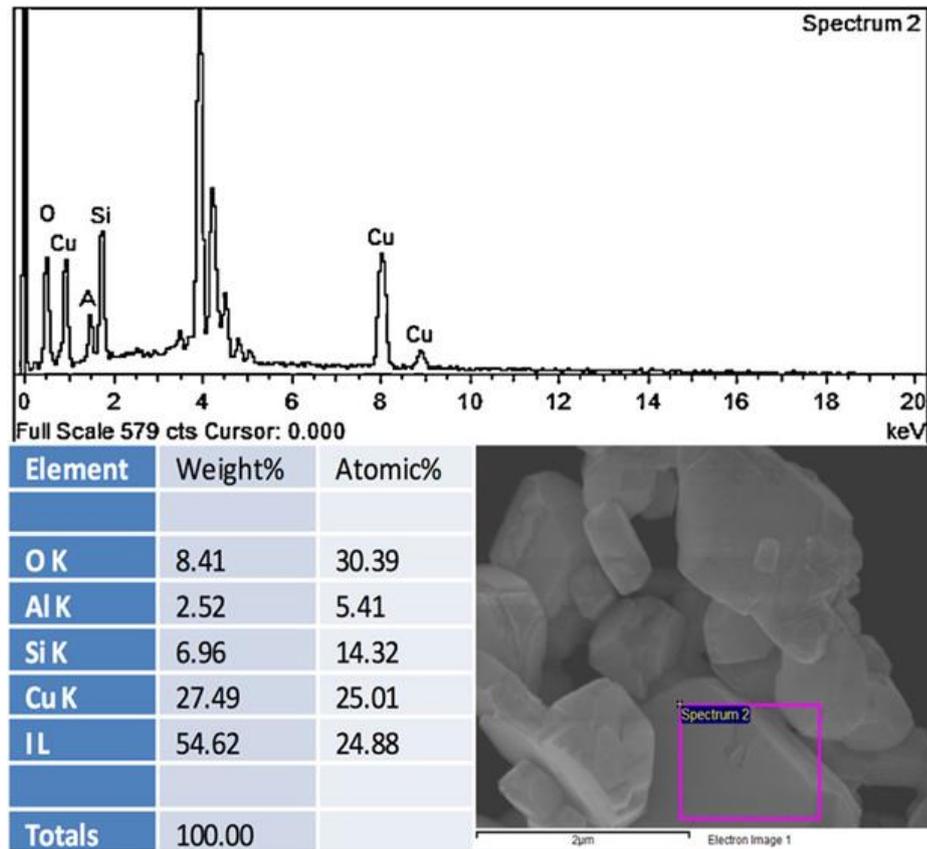


Figure 2.3: EDX spectrum from the magenta inspection field of powder sample and the figure table summarized the elemental compositions present with IL being the most abundant (Sabatini et al., 2015)

2.3 The Concept of Digital Image Processing

A digital image is a finite and discrete quantities of two-dimensional function of $f(x,y)$, where x and y are the spatial (plane) coordinates and the amplitude f is called the intensity of the image at that level. A digital image is composed of a finite number of elements named pixels, each of which has its own location and value. Images obtained from any sensors are stored according to image file formats such as RAW, BMP, GIF, JPEG and TIFF. Primarily, these are designed to preserve the high image quality with low storage consumption, quick transference, simple transformation to other formats and

straightforward editing/processing (Bhabatosh and Dutta, 2011). By looking at the modern computerization and image sensor technologies, digital image processing has become more vital in the sense of substituting the conventional film-based photomicrograph in microscope imaging (Wu et al., 2010). According to Annadurai (2007), digital image processing technique is used in two major application areas which are the enhancement of pictorial information for human interpretation and organizing of scene data for autonomous machine perception. In simpler words, image processing is a method to perform some operations on the image, in order to get an enhanced image or to extract some useful information from it. Essentially, the sequence in image processing includes steps such as importing, processing, analysing, manipulating, and reporting based on output processed image upon analysis. The computational approach in image analysis include various processing techniques, namely enhancement, segmentation, detection of the region of interest, pre-filtering method, thresholding technique and morphological operations (Rajeswari and Jagannath, 2017).

2.3.1 Intensity Histogram

Histogram plot of a digital image is a distribution of its discrete intensity level in the range. This histogram is basically a graph displaying the number of pixels from 0 (dark) to 256 (light) in an image at each vary intensity value found. In the context of image processing, the distribution of level intensity will provide the user information on global appearance of the image and its properties. Moreover, the intensity histogram is also useful for deep apprehension while performing further image processing such as image enhancement, image compression, and image segmentation (Das, 2018).

Histograms can assist in detecting image acquisition problems such as image exposedness (over or under exposure), brightness, contrast or dynamic range (Agu (2014).

Typically, the result of level histogram will follow by the contrast enhancement and brightness preservation of the image in which it improves the image quality, developing informative and visually pleasant image (Agarwal and Mahajan, 2018). In most cases, histogram equalization is seen to be widely adapted in digital image analysis as it can generalize the intensity value such that the output image formed has an equal number of input pixels fall into each ordered class (Tang and Mat Isa, 2017; Singh and Kapoor, 2014; Aгаian et al., 2007; Buzuloiu et al., 2001). However, one must not confuse as the plotted histogram after histogram equalization will appear flat since the histogram nature has already been altered and thus, losing some information on the image characteristics. This is because histogram equalization is rather an enhancement tool in image processing, though the usefulness of intensity histogram in image analysis is far beyond it.

2.3.2 Image Enhancement

Image enhancement is an action of rendering digital images for better human visual interpretation and/or the suitability of the processed images to further computer analysis. Image enhancement techniques have been widely applied in every of pre-processing digital imaging where the subjective of image quality is crucial. According to Gonzalez and Woods (2008), image enhancement approach falls into two main categories: spatial domain method and frequency domain method. The term ‘spatial

domain' refers to an aggregate of pixels comprising in a digital image. Hence, the process action is directly based on the manipulation of image's pixel. On the other hand, the frequency domain processing is constructed from manipulating Fourier transformation to an image. Furthermore, as mentioned by Gonzalez and Woods (2008), spatial domain process can be mathematically defined by the expression:

$$g(x, y) = T[f(x, y)] \quad (2.1)$$

where $f(x, y)$ is the input image, $g(x, y)$ is the output (processed) image, and T is the operator on f (set of processed input data).

In image processing, a digital image can be enhanced by removing noise, refining, or brightening the image. These enhancement techniques are either linear or non-linearly applied in pre-processing stage of image analysis in order to effectively display or record the image data for subsequent visual interpretation.

2.3.2.1 Random Noise Removal

In signal processing, noise is an unwanted information present in a digital image that created during the process of storage, transmission, processing, or conversion (Tuzlukov, 2002). During SEM image collection, the stability of an image could be interrupted at multiple stages where each of them contribute to its own noise component to the detector signal noise. According to Timischl et al. (2012), primary emission, secondary emission, scintillator, photocathode, and photomultiplier are the sources from where the noise pulses generated. In many cases, the removal of background noise in SEM images is crucial as it showed significant advantages in image analysis (Marturi et

al., 2014; Oho et al., 1996; Mazhari and P R Hasanzadeh, 2016). Without altering much the nature of SEM images, filtering the noise is most effective way to preserve and minimize the uncertainties of digital images. A study by Shenbagavadivu and Devi (2013) has investigated several techniques in noise removal used in spatial domain image processing as summarised in Table 2.1. It was concluded that the median filter may be the best enhancement method if the image contained low noise but relatively high magnitude. Meanwhile, Eliason and McEwen (1990) researched the utilization of adaptive box filtering to remove random bit errors (pixel values with no relation to the image scene) and smooth noisy data (pixels related to the image scene but with an additive or multiplicative component of noise) from digital images. This technique is proved to effectively filter the random noise without degrading much the fine image detail.

Table 2.1: Filtering techniques according to Shenbagavadivu and Devi (2013)

Filter Name	Filter Type	Noise Type & Performance
Mean Filter	Linear Smoothing	Gaussian Noise
Weiner Filter	Linear Smoothing	Additive Noise
Gaussian Filter	Linear Smoothing	Gaussian Noise
Max filter	Nonlinear Smoothing	Salt Noise (Brightest Point)
Mean Filter	Nonlinear Smoothing	Pepper Noise (Darkest Point)
Midpoint Filter	Nonlinear Smoothing	Gaussian and Uniform Noise
Median Filter	Nonlinear Smoothing	Impulsive Noise
Rank Order EV Filter	Nonlinear Smoothing	Reduction of White Noise
Rank Order ER Filter	Nonlinear Smoothing	Complicated Noise with Impulsive Component
Rank Order KNV Filter	Nonlinear Smoothing	Reduction of White Noise and Speckle Noise
Laplacian Filter	Nonlinear Sharpening	Edge Detection
Gradient Filter	Nonlinear Sharpening	Noise Reduction

2.3.3 Image Classification

In many image processing applications, the size, shape and pattern of particle such as aggregates, molecules, drops, bits of pigment and cell nuclei etc. must be taken into consideration during any microstructural analysis. In these cases, classification in image processing refers to the assignment of extracting relative spectral information classes from a multiband raster image. The objective of image classification is to categorize each individual pixel correspond to the spectral data by assigning a unique value and/or its own grey level or colour. Consequently, the resulting raster image can be then manipulated to generate thematic maps. In general, there are two main classification methods which are supervised and unsupervised.

With supervised classification, one requires a reference or information classes of interest in the image which is also referred as the “training sites”. In image processing, training samples train the classifier to characterize the classes and are used to set the “rules” that eventually permit the assignment of class label to each pixel in the image (Sarath et al., 2014). Prior to that, many researchers have successfully applied this object-based image classification method in analysing remote-sensing image data (Laliberte et al., 2012; Ma et al., 2017; Rougier et al., 2016).

Unsupervised classification or commonly known as clustering is a method used to examine large number of unknown pixels and separates into homogenous classes based on particular characteristics present in the image values (Subbiah and C, 2012). Contrary from the supervised classification, this technique requires no preliminary training dataset which basically a more flexible approach to understand an image. The resulting information from this technique are spectral classes which based on natural

groupings of the image values, however there is a need of identification process of the spectral class by evaluating them to some form of reference data to accurately determine the informational values of the spectral classes (Sarath et al., 2014). The uncertainty found in spatial imaging is best studied by unsupervised classification. Many literatures on typical unsupervised clustering algorithms such as K-means, Fuzzy C-Means, hierarchical clustering, and mixture of Gaussians have been discussed accordingly. In a study by Abbas (2008), it is concluded that partitioning algorithms such as K-Means and Expectation Maximization clustering are best used for huge dataset while conversely, hierarchical clustering algorithm is suggested for small dataset.

2.3.2.1 Unsupervised Segmentation by K-Means Algorithm

Image segmentation is the classification of an image into different groups. In computer vision, Uddin (2014) defined image segmentation as a process of subdividing a digital image into numerous segments (sets of pixels, which also known as super-pixels). The aim of segmentation is to simplify and/or alter the representation of an image into a more significant and easier to analyse (Shapiro and Stockman, 2001). In general, image segmentation and classification process are inter-related, a classifier implicitly segments an image and on the other hand, a segmentation implies a classification (Brady, 2005). K-means algorithm is one of the simplest and most popular unsupervised learning algorithms that solve the clustering problem. In K-means clustering, it divides a dataset comprising of n points embedded in m -dimensional space into K clusters such that the data points within a cluster are more 'similar' among the other data points in another cluster. The term 'similar' implies as it means closer by some similarity measures such

as the pixels with the same label share certain characteristics like objects and boundaries (lines, curves, etc.). Therefore, the results of the K-means clustering algorithm are the centroids of the K clusters, which can be used to label new data and the labels for the training data (each data point is assigned to a single cluster). According to Dhanachandra et al. (2015) whom analysed the medical images, summarized that the proposed K-clustering algorithm has better segmentation and when used with median filter will enhance the segmented image. Meanwhile, Angelin et al. (2017) identified voids in rubberized mortar SEM images and they concluded that the challenges of partitioning the right group to find the pixel's seeds are possible using the K-means algorithm, combined with the watershed algorithm.

Conversely, there are some minor limitations of K-means clustering involved in data organization. The K-means clustering process required the user to specify the initial number of clusters intended. Ultimately, K-means dealt with the assumption related to spherical clusters and that each cluster has roughly equal numbers of observation (Keppe and Schmalz, 2017). In short, this satisfies the Expectation Maximization (EM) algorithm and predicts that all clusters are evenly sized and have the similar variances. In most of the cases this assumption is not satisfied as clusters will differ in their size, density and variance. Careful consideration and interpretation of analysis must be conducted to bring out the best result in K-means clustering.

2.4 Microstructure Properties Evaluation through Digital Image Processing

Nowadays, microscope imaging and image analysis are of the greatest interest in scientific and engineering societies. The applications of digital image processing are

continuously expanding particularly in the study of machine vision, materials science, medicine, metallography, microscopy, optical character recognition, remote sensing, robotics etc. As SEM images are the result of data collection over a selected area of sample's surface, hence this approach is especially useful in qualitatively or semi-quantitatively mapping the composition and particle distribution in concrete microstructure-property relations. In the earlier study, Zhao and Darwin (1992) has performed a quantitative analysis on BSE cement paste image. During the image acquisition, the Backscattered Electron Detector (BSD) adjustment was fixed to two silicon/magnesium standard of mean video scope signal intensities namely (i) -3 for (Si) and -15 for (Mg) to differentiate between the five major phases within cement paste (treating unhydrated materials as a single phase) and (ii) -8 for (Si) and -18 for (Mg) to distinguish between phases within the unhydrated particles. Table 2.2 presents the threshold file utilized for the Si-Mg standard for representing the grey levels ranging from 0 to 255. An upper limit of 240 was set in the study.

BSD Settings (Standard)	Grey Levels						
	Si	Mg	Unhydrated Cement Particles	Calcium Hydroxide	Non-Calcium Hydroxide Inner Product	Calcium Silicate Hydrate	Cracks and Voids
-3 (Si) and -15 (Mg)	102-138	24-62	212-240	159-211	107-158	16-106	0-15
-8 (Si) and -18 (Mg)	146-188	27-97	-	-	-	-	-

Table 2.2: Grey levels range for image analysis (Zhao and Darwin, 1992)

Similarly, Yue Li et al. (2016) have examined the original image of SEM backscattered electron in quantitatively analysing the ground blast furnace in cement-slag paste. Concurrently, Vidal et al. (2016) investigated the image processing technique in quantifying cracks area in electrodeposits. The study uses the second derivative of the histogram obtained with Laplacian of Gaussian (LoG) together with Prewitt vertical edge detector to generally access the spatial cracked area by comparing the data with the subjective visual assessment. Likewise, Zhang and Ye (2011) studied the images captured by environmental Scanning Electron Microscope (ESEM) by segmentation and binary image processing to quantify the volume fractions of hydration products and porosity. Meanwhile, in the related study done by Drumetz et al. (2015) on the new segmentation and classification method of SEM images analysis, the research has provided an automatic segmentation and classification maps involving the usage of an advanced image processing tools.

On a different note, Gopan and Wins (2016) experimented the image processing method such as global thresholding and noise filtering technique onto the actual digital images of the wheel surface as the study aimed to have a quantitative analysis of wheel loading. Campbell et al. (2018) also proposed a new method in image processing for automating the measurement of microstructural SEM images where it utilized the filtering technique, watershed transformation, region merging, phase separation and measurement process. In addition, Meulenyzer et al. (2013) studied the multispectral image datasets (backscattered electron (BSE) image combined with X-ray microanalysis elemental maps) of cementitious paste. The study has explored the application of merged image processing algorithms such as Support Vector Machine (SVM) and Markov Random Field (MRF) algorithms in quantifying the supplementary cementitious

materials particularly fly ash, slag and natural pozzolans. Also, Soroushian et al. (2003) established the pixel length criterion for filtering noise and other highlighted features of concrete microstructure at three different magnification factors (Table 2.3). The study has concluded that the automated quantitative microstructural analysis of concrete micro cracks and voids systems required a proper selection of image processing operations.

From the past research studies, it can be presumed that with the aid of modern technology imaging such as Scanning Electron Microscopy (SEM), incorporated by the image processing and analysis allows many researchers to have a better judgement and critical investigation in defining the concrete microstructure properties. Lastly, Table 2.4 provides the review summary of the past research work related to microstructure properties evaluation using the image processing tool.

Table 2.3: Lengths of noise, micro-cracks, and voids at the three different magnification factors (Soroushian et al., 2003)

Length (Pixels)	125× magnification			250× magnification			500× magnification		
	Noise	Micro-cracks	Voids	Noise	Micro-cracks	Voids	Noise	Micro-cracks	Voids
Minimum	2	18.4	8	2.82	29	6.72	2	29	35.5
Maximum	106	478	612	82	756	906	165	948	745
Mean	15.5	97.7	107	16.8	130	205	24.4	198	271

Table 2.4: A review summary of digital image processing techniques used in analysing concrete digital images

Author (reference)	Digital Image Types	Spatial Processing Techniques			
		Histogram Analysis	Image Enhancement	Image Classification	Image Segmentation
Zhao and Darwin (1992)	BSE Images	✓	✗	✗	✗
Yue Li et al. (2016)	SEM-BSE Images	✓	Filter	✗	✗
Vidal et al. (2016)	SEM Images	✓	Laplacian of Gaussian (LoG)	✗	Prewitt Vertical Edge Detection
Zhang and Ye (2011)	ESEM-BSE Images	✓	✗	✗	✗
Drumetz et al. (2015)	SEM-BSE Images	✓	Block Matching 3D (BM3D) Algorithms	Support Vector Machines (SVM) + Markov Random Field (MRV) Regulation	Binary Partition Tree (BPT)
Gopan and Wins (2016)	Microscope Digital Images	✗	✗	✗	Global Thresholding
Campbell et al. (2018)	SEM and Optical Microscope (OM) Images	✗	Gaussian Filtering, Sobel Filters	Watershed Algorithm, Globular Alpha Segmentation	✓
Meulenyzer et al. (2013)	BSE Image + X-Ray Microanalysis Elemental Maps	✗	Fourier Transformation	SVM-MRF Algorithms	✗
Soroushian et al. (2003)	ESEM-BSE Images	✗	✗	✗	Automated Thresholding
Dhanachandra et al. (2015)	Medical Digital Images	✗	Median Filter	✗	K -means Clustering Algorithm, Subtractive Clustering Algorithm
Angelin et al. (2017)	SEM Images	✗	Median Filter	✗	K-Means, Watershed algorithms

CHAPTER 3

METHODOLOGY

3.1 Introduction

This chapter explains the methodology used in the process of quantifying and mapping the concrete SEM images. It covers the background information on the image analysis software used, the details on the research data, and the digital image processing and analysis techniques applied in the study. In addition, a further description on the identification and quantification process of micro-cracks (voids) and the map analysis of SEM images were provided. The experimental methods and procedures adopted are all summarized and presented in Figure 3.1.

3.2 Software

The image processing software used in this study is GIS- IDRISI Selva Version 17. This is an integrated and complete GIS and Image Processing software that provides practically 300 modules to tackle digital spatial information analysis. In general, the GIS- IDRISI software offers spatial solution for land change and graphical modelling, time series analysis, neural network analysis, decision support, and spatial statistics. In addition, the image processing software tools comprises of complete restoration, transformation, enhancement, supervised and unsupervised classification process to support the spatial imaging work.