STUDY ON SETTING TIME AND COMPRESSIVE STRENGTH PERFORMANCE OF ORDINARY PORTLAND CEMENT (OPC) PASTE CONTAINING SODIUM CHLORIDE

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ABSTRACT

The presence of impurities in concrete system can possess a damaging effect to the structural integrity of concrete material. Alkali silica reaction, scaling, and corrosion are among the deleterious effect caused by the ingression of internal or external substance to the concrete system. Sodium chloride or salt is one of the external agents involved in the process; hence its excessive amount in concrete system is strictly prohibited. Nevertheless, concrete containing salt water is generally known for the higher early-strength, yet lower ultimate strength than plain concrete. Meanwhile, study on the detail mechanism in early age properties are scarcely available, particularly at low salt concentration. Therefore this research was conducted to provide better understanding in the effect of salt water to the hardening mechanism of Portland cement paste and to obtain the optimum salt inclusion that contributes to the enhancement of concrete properties. Analysis on the performance of this reagent was conducted via setting time, compressive strength, and porosity test. Based on the setting time analysis, the inclusion of sodium chloride can extend the initial setting time of cement paste longer than control specimen. Obstruction on the formation of calcium silicate hydrate gel by sodium and chloride ion was one of the possible causes to this phenomenon. Acceleration on the compressive strength development by sodium chloride was also detected. It appears that sodium chloride was able to de-flocculate the coagulated cement particles and reduced the viscosities of cement slurries; hence resulted in faster early hydration process. Based on the results of this research, it can be concluded that sodium chloride inclusion is beneficial to the workability and compressive strength performance of cement paste binder at certain concentration. Improvement of compressive strength was achieved via faster dissolution and hydration of cement particle due to dispersant characteristic presented by sodium chloride.

Keywords: Sodium Chloride, Cement Paste, Setting Time, Compressive Strength

ABSTRAK

Kehadiran kekotoran dalam sistem konkrit boleh menberi kesan kerosakan kepada integriti struktur bahan konkrit. Reaksi alkali silika, bersisik, dan kakisan adalah antara kesan yang merosakkan disebabkan oleh bahan dalaman atau luaran kepada sistem konkrit. Natrium klorida atau garam adalah salah satu daripada agen luaran yang terlibat dalam proses itu; oleh itu jumlah yang berlebihan di dalam sistem konkrit adalah dilarang. Walau bagaimanapun, konkrit yang mengandungi air garam yang diketahui umum untuk kekuatan awal yang lebih tinggi, namun kekuatan muktamad yang lebih rendah daripada konkrit biasa. Sementara itu, kajian ke atas mekanisme terperinci dalam peringkat awal adalah tidak ada, terutamanya pada kepekatan garam yang rendah. Oleh itu kajian ini telah dijalankan untuk memberi pemahaman yang lebih baik dalam kesan air garam kepada mekanisme pengerasan Portland simen pes dan untuk mendapatkan kemasukan garam optimum yang menyumbang kepada peningkatan sifat konkrit. Analisis terhadap prestasi reagen ini telah dijalankan melalui menetapkan masa, kekuatan mampatan, dan ujian keliangan. Berdasarkan analisis masa penetapan, kemasukan natrium klorida boleh melanjutkan masa penetapan awal pes simen lama daripada spesimen kawalan. Halangan dalam pembentukan kalsium silikat hidrat gel oleh natrium dan klorida ion adalah salah satu sebab yang mungkin untuk fenomena ini. Pecutan kepada pembangunan kekuatan mampatan natrium klorida juga dikesan. Nampaknya natrium klorida dapat membentuk zarah simen beku dan mengurangkan kelikatan buburan simen; dengan itu menyebabkan proses penghidratan lebih cepat. Berdasarkan hasil kajian ini, dapat disimpulkan bahawa kemasukan natrium klorida memberi manfaat kepada kebolehkerjaan dan prestasi kekuatan mampatan simen tampal pengikat pada kepekatan tertentu. Peningkatan kekuatan mampatan dicapai melalui pembubaran cepat dan penghidratan simen zarah kerana ciri penguraian disampaikan oleh natrium klorida.

Keywords: Natrium Klorida, Cement Paste, Menetapkan Masa, Kekuatan Mampatan

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CHAPTER 1

INTRODUCTION

1.1 BACKGROUND

The main component of concrete contains of cement, aggregates and mixing water. In general, concrete is an artificial conglomerate stone made of Portland cement, water and aggregates. Meanwhile, admixtures are used to enhance the strength of the concrete in term of physic-chemical, physical, chemical and also mechanical effects. (A.B.Yilmaz, 2000). Table 1 shows the lists of ingredients or mixture percentages of a typical mix (Concrete Basics, 2010).

Table 1.1: Most common ingredients in concrete (Concrete Basics, 2010)

Ingredients	Percentages of concrete proportion
Air	6%
Cements	11%
Coarse aggregates	41%
Fine aggregates	26%
Water	16%

In overall, the percentages of each ingredients change, depending on the use of the concrete. The cement in the concrete acts as a binder when mixed with water. The cement hardens, or cures, through the process of hydration, a chemical change to be mixed. (Wasserman & Brian, 2011). This chemical reaction normally changes from a plastic to a solid state within 2 hours and concrete continues to gain strength as it cures.

In practical terms, about 90% of its strength is gained in the first 28 days. In general, concrete compressive strength depends on many factors which are quality and proportions of the ingredients.

1.2 PROBLEM STATEMENT

Issues concerning the durability and compressive strength of concrete containing Sodium Chloride solutions have been a great concern to civil engineers and material companies. Innovation in the use of admixtures and relevant cementations materials increases day by day due to these concerns. It is an important part to identify and determine how these materials will improve concrete strength and durability.

In the past few decades, an extensive research has obtained the properties and mechanism of saltwater attack on cement paste and concrete, and the result shown that Sodium Chloride solutions certainly causes deterioration of cement paste due to reaction with susceptible components of the paste such as gypsum, ettringite, and brucide. (A.Shayan, 2008). Although it is said that seawater –mixed concrete should be avoided to use. In the case of unavoidable situation where the access to clean water is limited, then the feasibility of seawater as a concrete mixing medium can be investigated.

The compressive strength of concrete is shown to be increased due to the presence of salts and sodium chloride in the process of mixing water. The strength was found to increase with increasing salinity even up to 7% by weight of water. (Michael A.Taylor, 1978). The relationship between elemental composition and degree of hydration should be studied to identify the factors that contribute increasing or decreasing of compressive strength by previous studies. Nevertheless, the effect of impurities particularly chloride in the mixing water should be studied also to provide better understanding in its influence to concrete strength and durability.

1.3 OBJECTIVES OF STUDY

Basically, there are 2 main objectives of this research. The main objectives of this study are:

1) To determine the workability, setting time, compressive strength, and porosity performance of cement paste made with salted water.
2) To determine the optimum percentage of Sodium Chloride content that can contribute to the enhancement of concrete properties.

1.4 SCOPE OF THE STUDY

Sodium Chloride used in this researched are obtained from commercial salts. The Sodium Chloride selected is 100% pure sea salt which contains no additives and preservatives. It's used with a certain amount of percentages to dilute with fresh water to produce Sodium Chloride solutions. Ordinary Portland Cement Type 1 are used in this researched.

Test that conducted are compressive strength test and porosity for hardened paste. Meanwhile, flow table and vicat apparatus are used in order to measure workability and setting time for fresh binder paste. The sample sizes of cement paste used in this research are 50mm x 50mm x 50mm. The entire sample is tested at the age of 1days, 28days, and 56 days. For the freshwater content in the cement paste, it will replace with sodium chloride solution with different percentage of 0%, 0.5%, 1%, 1.5%, 2%, and one proportion is replaced with fully seawater. Sample preparations will be done in Concrete Laboratory of University Malaysia Pahang to determine the parameters needs.

1.5 RESEARCH SIGNIFICANCE

The purpose of this research is to investigate the alternative ways to replace freshwater with saltwater since it is might increase the compressive strength of concrete

and durability. The relationship between setting time and compressive strength are analysed in this research. Optimum percentages of saltwater that contribute to the incremental process also investigated .Compressive strength are tested at the age of 1, 28, and 56 days .The results are obtained will compare with control samples having 0% saltwater in the mix.

CHAPTER 2

LITERATURE REVIEW

2.1 CONCRETE

Concrete is known as composite materials consisting of building medium, cement and water in which particles of relatively inert filter materials such as sand and granite aggregate. Concrete also classified as an artificial conglomerate stone made of Portland cement, water and aggregates. ADMIXTURES are used to modify the existing materials through the variety application in order to increase the properties of concrete.

2.1.1 Strength of Concrete

The maximum stress or concrete load could withstand is referred to as its strength. The possibilities of concrete strength are determined by the composition and properties of its embedded material. In the construction industry, the compressive strength of concrete is used for specification and quality control. Meanwhile, the proportion of water cement ratio is an important to determine the quality, strength and the age of the concrete as well as the mix and shape of the specimen used to be tested.

2.1.2 Compressive Strength of Concrete

The compressive strength of concrete is an important properties that easily to be determined. It referred as the maximum compressive load that a concrete can carry per unit area. However, it depends on the constituents of mix proportions, type of cement, method of testing and also curing preparations. The results of compressive strength are primarily used to determine that the concrete mixture meets the requirements of the

specified strength. When the compressive strength needed could not be achieved, it shows errors during the process of fabrication, handling, curing and testing are not accorded the standard procedure.

2.1.3 Concrete Hydration

Concrete hydration can be divided into two processes which is dissolution and precipitation. Dissolution referred as the process when the cement dissolves and releasing ions into the mix water. The mix water could be not only pure fresh water, but an aqueous solution containing a variety of ionic species referred as pore solution. The use of cement minerals and gypsum which is highly soluble brings the mixture to dissolve quickly.

Therefore, the pore solutions increase rapidly as soon as the cement and water are combined due to the concentrations on ionic species. The second process referred as precipitation which it relieves the supersaturation of the pore solution. Hence, it allows dissolution of the cement minerals to continue. This cement hydration can be explained as a continuous process which the cement minerals are replaced by the new hydration products. The famous chemist Le Chatelier are the first person have been studied for hundred years regarding the reaction between Portland cement and water and the facts that the hydration process proceed with the dissolution and precipitation.

2.2 SALTWATER

Saltwater is water from a sea or oceans. The saltwater or saline water has characteristics most similar to fresh water with some noticeable differences because of the salts that are dissolved in water. It's measured in terms of dissolved materials per kilogram of sea water which it represents the amount of dissolved salt in seawater. On average, the salinity is a range of 3.5% or 35g/L containing of dissolved salts (sodium Na^{+}) and chloride (Cl^{-}) ions.

2.2.1 Physical Characteristics of Saltwater

In general, seawater or saltwater tends to have a higher density compared to freshwater. In fact, 35ppt concentration of seawater is equal to 1.0264 times as dense as freshwater at the same temperature and it has specific gravity 1.0264. This salinity or concentration can be measured by using hydrometers. Besides that, water that contains salts have an ability to refracts light which means bends light passing through compared to freshwater reaction. Meanwhile, it can conduct electricity due to more charged ions present in the water. This ion can lead to useful determinations of the concentration of Sodium Chloridein water.

2.2.2 Chemical Characteristics of Saltwater

Waters that contain salts have a variety of organic and inorganic chemicals which lead to provide chemical reaction. It referred as below:

I. Contains minerals – sodium (Na^+), Chloride (Cl^-), Magnesium (Mg^{2+}), Potassium (K^+), Sulfate (SO_4^{2-}) form salts
II. It's held together by ionic bonds that allows them to dissolve easily in water
III. Ph-Alkaline (Seawater slightly alkaline or slightly acidic probably due to pollution)
IV. Three main gasses dissolved in seawater
    i. Oxygen (O) 36% that's primarily used in respiration,
    ii. Nitrogen (N) 48% act as protein building,
    iii. Carbon dioxide (CO) 16% of photosynthesis.

2.2.3 Chemical Action of Saltwater On Concrete

According to (Bryant.M, 1964), concrete that exposed to saltwater might be attacked by mainly due to existing of sodium chloride and magnesium sulphate ($MgSO_4^-$) in the concrete. This gives impact to wetting and drying process by chemical reaction with chlorides, sulfates, or alkali obtains in the cement and another mixture of concrete. (A.M.Badike & A.U.Elinwa, 2011) founds that based on his research, the

results obtained that the concrete strength will decrease while the presence of chlorides and sulphates in concrete approximately by 8%.The seawater can be used to mix concrete, but its strength will be decrease 10 to 20 percent compared of using freshwater. The solution is by using less water in the mix or the amount of cement is increasing. The steel reinforcement in the concrete mix with seawater will not deteriorate when it's exposed to air. Combination air and saltwater will cause concrete to spall and corrosion to the steel reinforcement. (Schwartz, 2000)

2.2.4 Concentration of Saltwater Measurement

There are two methods to identify the amount of Sodium Chloride in water. It referred as Total Dissolved Salts (or Solids) and Electrical Conductivity. Total Dissolved Salts (TDS) measured by evaporating a known volume of water to dryness and weighing the solid residue remaining. Meanwhile, Electrical conductivity (EC) is measured by passing an electric current between two metal plates called electrodes in the water sample. (Anderson & Cummings, 1999)

The more dissolved Sodium Chloridein the water, the stronger the current flow and give higher value of the EC. Unit measurement for TSS is in milligrams of dissolved solid in one liter of water (mg/L) and the EC will measure the charge carrying ability of liquid in a specific dimension. According to Victorian Salinity Program and Murray Darling Basin Commission, the standard EC unit used is microSiemens per centimeter (μS/cm) at 25^0.

EC can also be converted to TDS by the following relationship as in Eq. (2.1)

$$\text{TDS (mg/L)} = \text{EC (}\mu\text{S/cm at } 25^{\circ}\text{)} \times 0.6 \qquad (2.3)$$

2.2.5 Saltwater and Compressive Strength of Concrete

The most usually considered for measurement of concrete is its compressive strength, even in other research cases, they may use other characteristics such as permeability, durability, volume and stability may in fact be more important. The most

important part in mixing of concrete is the water used in that process and also for curing purposes. Generally, there are a few conditions and extra special consideration when concrete for coastal structures is cured with seawater. It needs to maintain a moist environment by sealing materials used in order to keep continuously wet. Although the saltwater has been used in the past as the replacement of freshwater in the curing process, recent research still concerning the corrosion issues of the reinforcing steel indicates that only fresh water can be used in curing process.

Previous researchers carried out and studied regarding the effects of saltwater on the compressive strength of concrete either by replacing the freshwater with saltwater as mixing or curing or even both might increase the strength in the final results. Reports obtained in the past studies revealed that by using saltwater will decrease the strength, meanwhile recent studies obtained increasing of compressive strength. There have found the possibilities that other factors contribute to the results provided such as changing chemistry and or grinding of cements by the action of salts. The concentration of Sodium Chlorideis also identified as one of the factors responsible for the strength differences recorded.

(Taylor & Kuwairi, 1978) and (Bryant.M, 1964) has been carried out an experiment regarding the effects of ocean salts on the compressive strength of concrete either cast or cured in saltwater. Hence, the result shows the main factors of increasing or decreasing the strength are depended of types of cement and Sodium Chlorideused. Types of possible salts referred to NaCI, $MgCI_2$, Na $_2SO_4$, and CaCI2. They observed the strength of the concrete will increase with increasing the amount of ocean salts to 7% by weight of water.

A few years ago, a researcher namely (Lateef, 2010) carried out an experiment regarding the effect of seawater concentration on compressive strength of concrete. Based on the research it shows that the strength increment for cubes cast with seawater and it noted that the strength will continually increase at 7, 14, 21 until 28 days. They also observed that some reaction or chemical processes have taken place.

Based on the inconclusive research results of TayloR, Kuwairi and Briant. M, it was suggested that further test is required in order to investigate if any ascertain influence, basic mechanisms and other important characteristics of concrete that may involve.

2.2.6 Effect of Saltwater on Concrete

In general, direct action of saltwater may results in Sodium Chlorideattack. However, it depends on the type of soluble Sodium Chlorideused that possess different deterioration mechanism in terms of concrete. Sodium Chlorideattacks on concrete can be in the following ways:

I. Physical attack
II. Chemical attacks
III. Corrosion of reinforcement

Table 2.1 below shows different effects of Sodium Chloride solutions on concrete by previous researchers.

Table 2.1: Past observations of effects of saline water

Salts	Observations	References
Ocean salts	Strength increase with salinity of mixing water	Cement and Concrete Association, London
3.5%	Water cured : it stronger at 3 and 7 days but not at 28 days	Abrams A. D. Anons
Synthetic sea	Increase the compressive strength of the concrete in the presence of NaCI in the mixing water	Abrams A. D. Anons

Table 2.1: Continued

Salts	Observations	References
Chloride	When the chlorides reach the reinforcement bar, it may increase the risk of corrosion. But, it depends on the concrete mix proportions, type of cement used and other mix constituents	Cement Concrete & Aggregates Australia
Seawater	Based on the research it shows that the strength increment for cubes cast with seawater and it noted that the strength will continually increase at 7, 14, 21 until 28 days.	Lateef & Raimi
Ocean salts	The research has done a few decades before and it's obtained that the strength of the concrete will increase with increasing the amount of ocean salts to 7% by weight of water.	Taylor & Kuwairi

2.2.6.1 Physical attack

When concrete is exposed by a Sodium Chloride solutions solution, with long periods of drying during which pure water evaporates, some of the Sodium Chloride dissolved in the Sodium Chloride solutions solution are left behind in the form of crystals (mainly sulfates) in the concrete pores and surface of the concrete unit. These crystals re-hydrate and grow upon subsequent wetting, and thereby exert an expansive force on the surrounding hardened cement paste within the concrete unit when this growth occurs. This expansive force is greatly affecting the ability of the Sodium Chloride crystals to grow rapidly to many times their original crystal dimension upon wetting. This rapid growth causes the concrete paste surrounding the crystals to "burst", exposing the aggregate in the concrete masonry unit.

2.2.6.2 Chemical attacks

The existence of chloride and sulfate ions in saltwater might change the composition and microstructure of the concrete. However this change may vary in type or severity but may include:

I. Extensive cracks
II. Expansion
III. Loss of bond between the cement paste and aggregate

Alteration of paste composition, with monosulfate phase converting to enttringite and may cause gypsum formation. The necessary additional calcium is provided by the calcium hydroxide and calcium silicate hydrate in the cement paste.

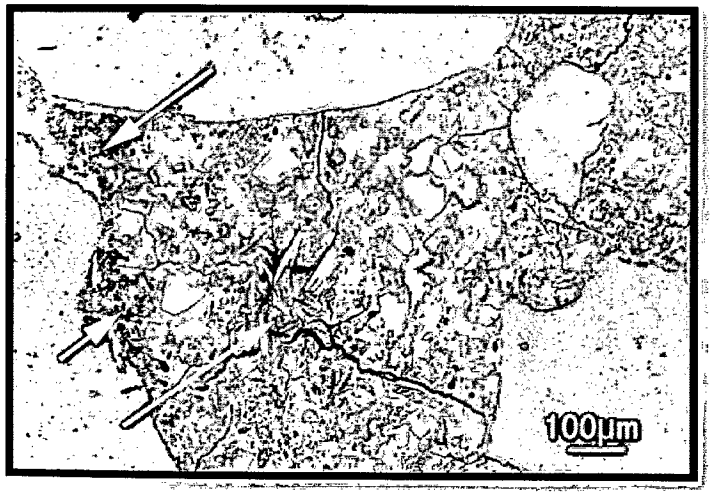

Figure 2.1: Scanning electron microscope image of sulfate attack in concrete.

Source: Nicholas B. Winter 2003

It causes the cement paste expanded and a gap has formed between the aggregate and the cement paste. (Winter, 2005). However, this attack can be classified as "moderate" because they attack a greater number of the concrete constituents.

2.2.6.3 Corrosion of reinforcement

The reinforcement bar in concrete will preserve and protected with highly alkaline environment by forming a strong impermeable oxide layer on its surface. This automatically protects the steels from corrosion. In general, corrosion of reinforcement in concrete will occurs in two phases, namely *initiation and propagation.* The initiation phase can be defined as the concrete alkalinity is reduced by carbonation or ionization.

The sources of carbonates are groundwater or carbon dioxide in the air. Meanwhile, ionization occurs where there is a higher concentration of reactive ions such as chlorides. The Chlorides sources include acid etching, atmosphere, soil, groundwater, admixtures, or the water, aggregate and sand used to make the concrete. After that, the propagation phase of the corrosion continues to depend on the amount of oxygen, moisture, reactive ions and remaining alkalinity. (Department of Environment & Climate Change, 2003)

2.2.7 Types of Saltwater

In general, the water used in the concrete mix should be clean, fresh drinking water that's free from any impurities such as salt. However, some waters that are not fit for drinking may be suitable for use in mixing concrete. ASTM C109 or AASHTO T106 already stated that any water can be used for making concrete if the mortar cubes when it have 7 day strengths equal to at least 90% of companion specimens made with drinkable or distilled water. Meanwhile, guideline provided by ASTM C191 (AASHTO T131) tests should be made to ensure the impurities in the mixing water do not extend or adversely shorten the setting time of the cement.

The effect of impurities in mixing water not only will affect setting time and strength of concrete, but it might cause staining, corrosion of reinforcement, efflorescence, volume instability and reduced durability. Table 2.2 and 2.3 show the acceptance criteria of water quality and types of salts and its effect that might be used in mixing concrete respectively

Table 2.2: Acceptance Criteria for Questionable Water Supplies (ASTM C94 or AASHTO M 157)

	Limits	**Test method**
Compressive strength, minimum percentage of control at 7 days	90	C 109* or T 106
Time of set, deviation from control, Hr: min	From 1:00 earlier to 1:30 later	C 191* or T 131

*Comparisons should be based on fixed proportions and the same volume of test water compared to a control mixture using distilled water.

Source: Design and Control Concrete Mixtures EB01

2.3 TESTING

Variety method in determining the testing involves in the concrete field to measure rheological properties of cements such as compressive strength, workability, degree of hydration and porosity.

2.3.1 Compressive strength

There are varieties of methods to measure the compressive strength of cement paste by the previous studies. The main factor affecting the compressive strength of a concrete structure is its w/c ratio. (R.Zoughi, 1995) used microwave compressive strength estimation of cement paste by using monopole probes. It's an alternative ways for a non-destructive and in-situ technique for evaluating the compressive strength. Microwave non-destructive methods become interesting and attractive for use in evaluating various properties of concrete members. It also can be used to determine the degree of hydration of the cement paste from the initial mixture to its final cured state.

Table 2.3: Possible Sodium Chloridein mixing concrete

Type of salts	Observation
Alkali Carbonate and Bicarbonate	Both of the salts have different effects on the setting times of different cements. Sodium carbonate cause very rapid settings, meanwhile bicarbonates can be accelerated or retard the set. However, when it's in large concentrations, these salts can materially reduce the strength of concrete.
Chloride	High amount of chloride contents in the mixing water process may cause due to the possible adverse effect of chloride ions on the corrosion of reinforcing steel or prestressing strands. This is because the highly alkaline in which the pH greater than 12.5 chemical environment present in concrete. (Taylor,Whitting, and Nagi 2000)
Sulfate	High sulphate content in mix water is due to possible expansive reactions and deterioration by sulfate attack, in areas the concrete will exposed to high sulfate soils or water.
Iron salts	Natural ground waters contain more than 20 to 30ppm of iron. These types of salts that have concentrations up to 40000 ppm do not usually affect concrete strengths adversely.
Sea water	35000 ppm of dissolved salts in sea water is generally suitable as mixing water for concrete not containing steel. Although concrete made with seawater may have higher early strength than normal concrete, strengths at later ages which is 28 days may be lower. However, it depends on the water cement ratio used.

Source: Design and Control Concrete Mixtures EB01

2.3.2 Workability

The previous researchers standardized flow test ASTM C124-71 in 1973 in determining workability of mortars. It is because the use in the field was deemed cumberstone compared to the standard described in ASTM C143. Later, it was revived as ASTM C1437-01. The method is upgrading again in ASTM C230 for its unique advantages in term of standardization and productivity. However, the flow test results remain difficult to interpret. Therefore the test is currently used in determining workability of cement paste and mortars.

2.3.3 Porosity

The total porosity and pore size distribution plays important roles in characterizing the pore structures in porous materials. Previous researchers use mercury intrusion porosimetry (MIP) method to evaluate pore size distribution in cement-based materials. Another method to evaluate the porosity is the quantitative SEM-BSE image analysis. It can be evaluated by the procedures of image analysis. However, it should be noted that the pore structure revealed by those MIP does not show the real structure in concrete due to inappropriate assumption to be made to the complex network of pores. (S.Igarashi, 2004).