

**IMPLICATION OF WATER PERMEABLE POROSITY AND
SORPTIVITY ON STRENGTH AND DURABILITY OF
HARDENED CEMENT BASED MATERIALS**

MANDALOZU RAJA



**DEPARTMENT OF CIVIL ENGINEERING
INDIAN INSTITUTE OF TECHNOLOGY DELHI**

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OCTOBER 2018

Dedication

Who has helped with my work

and

My supervisors Prof. B. Bhattacharjee &

Dr. Shashank Bishnoi

CERTIFICATE

This is to certify the thesis entitled “***IMPLICATION OF WATER PERMEABLE POROSITY AND SORPTIVITY ON STRENGTH AND DURABILITY OF HARDENED CEMENT BASED MATERIALS***” being submitted by **MR. MANDALOZU RAJA** to the Indian Institute of Technology, Delhi, India, for the award of the degree of “***DOCTOR OF PHILOSOPHY*** in civil engineering”, is a record of original bonafide research work carried out by him. *Mr. Mandalozu Raja* has worked under our guidance and supervision.

To the best of our knowledge, the thesis has reached the requisite standards. The material contained in this thesis has not been submitted, in part or in full to any other universities or institutes for the award of any degree or diploma.

Prof.B.Bhattacharjee
Professor

Dept. of Civil Engineering
Indian Institute of Technology
Hauz Khas, New Delhi - 11 0016
India

Dr.Shashank Bishnoi
Associate Professor

Dept. of Civil Engineering
Indian Institute of Technology
Hauz Khas, New Delhi - 11 0016
India

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MANDALOZU RAJA

ABSTRACT

The pore structure is the most important microstructural characteristic in cement-based materials as it influences the strength and durability of the material. The strength of cement-based materials is strongly affected by total volume of pores and their distribution within the solid. Materials with the same total pore volume can exhibit entirely different characteristics depending on size of pores i.e. less number of large pores or more number of small pores contained in the material. Therefore, estimation of representative pore size is extremely important for clear understanding of strength and durability properties.

There are several techniques like MIP (Mercury Intrusion Porosimetry), Adsorption, Microscopic etc., which are being currently used by researchers for measurement of pore structure parameters. The MIP technique is the most extensively used technique and sufficient data is available. All these available techniques are based on different assumptions and sample preparation techniques and size of sample. These techniques involve complex mathematical expressions for analysis. The size of the sample does not give fully the representation of the concrete mix in most these methods. These apparatuses are expensive and require special training to operate. To overcome all these difficulties, the available literature was surveyed for proposing the simple technique for measurement of representative pore radius.

The literature about the ingress of fluids in concrete was reviewed, and subsequently the possibilities for establishing the model based on these transport mechanisms was found to be one of the options. The strength and durability were solely dependent on the volume of pores and distribution in the concrete matrix. However, the durability depends on the inter connectivity of pores from concrete surface. Literature availability on estimation of pore structure parameters based on transport mechanism is very limited and very few researchers have studied the estimation of pore radius based on permeability.

The pores are mainly responsible for different transport mechanisms such as permeation, absorption and diffusion of concrete which allows the penetration of fluids into the concrete. Hence, estimating the representative pores radius by using any one of above techniques will help in clear understanding of both strength and durability properties of concrete.

Sorptivity is the most commonly used parameter for indicating the quality of concrete. The sorptivity is a function of mass of water absorbed in square root of time. Hence, it is easily correlated to Hagen-Poiseuille's and Lucas-Washburn's equation for determination of the representative pore radius. This was motivated for establishing simple model for determining of representative pore radius based on the sorptivity. The measurement of sorptivity requires a sensitive balance and climatic chamber, which are generally available in all material testing laboratories. There is no restriction on the size of sample and easily service conditions of the structure are simulated in sorptivity investigation. Initially, a simple case of cement paste is investigated for measurement of representative pore radius. Subsequently, paste with intrusion of sand (i.e. mortar) and concrete are investigated. Since the sorptivity indicates not only durability of concrete, it is also possible to estimate the strength of concrete from determined representative pore radius from established sorptivity model.

Secondly, in order to validate the determined representative pore radius, the availability of pore radius models in the literature was investigated. Kondraivendhan and Bhattacharjee model, which is based on the extensive study on MIP for calculation of pore entry radius was found and adopted for comparison purposes.

Thirdly, sorptivity sharply reduces with increase of degree of hydration and hence the degree of hydration of cement pastes and cement mortars are studied in detail and some empirical relationships for estimation of degree of hydration are proposed. The continuous pores are mainly responsible for moving liquids or gas through the structure of concrete. Therefore, the

measured water permeable porosity is compared with the estimated porosity by using Powers' Model.

Sorptivity mainly depends on the water-cement ratio, volume of the paste and age. It is also observed that sorptivity reduces with curing age and with degree of hydration. The increase in paste volume significantly increases the water permeable porosity and sorptivity. Therefore, an empirical relationship for estimation of sorptivity is proposed for cement paste and concrete.

Fourthly, the strength of concrete is related to the water-cement ratio, and the degree of hydration, which in turn are related to the porosity or the pore volume. The researchers have proposed different forms of strength-porosity-pore radius model for estimation of strength (compressive/splitting tensile). In order to validate the measured pore radius from sorptivity, one of these models is used for estimated strength of concrete. A consistent relation between measured and estimated is observed.

Durability of concrete structures is addressed by an engineering approach based on scientific principles called Durability Indices. This approach has been developed from the point the material characterization, durability specifications on a performance basis, as a means of site quality control, and as a basis for long-term predictions.

Correlations are required between indices, durability test results, and actual structural performance, such that the index tests can be used as:

1. A means of controlling a specific property of concrete, usually the quality of the cover concrete,
2. A means of assessing the quality of construction for compliance with a set of criteria, and
3. A means of predicting the performance of concrete in the design environment.

Finally, the data generated during investigation of sorptivity was used for estimation of other forms of durability indices such as coefficient of water absorption, percentage of water

absorption and apparent hydraulic diffusivity. Apparent hydraulic diffusivity is useful in predicting service life of a structure. The main scope of present study is to establish methodology for representative pore radius. Therefore, present study is restricted to presentation of other forms of durability indices from sorptivity data.

सार

छिद्र संरचना सीमेंट-आधारित सामग्रियों में सबसे महत्वपूर्ण सूक्ष्म संरचनात्मक विशेषता है क्योंकि यह सामग्री की ताकत और स्थायित्व को प्रभावित करती है। सीमेंट-आधारित सामग्रियों की ताकत ठोस रूप से छिद्रों की कुल मात्रा और ठोस के भीतर उनके वितरण से प्रभावित होती है। एक ही कुल पोर मात्रा वाली सामग्री छिद्रों के आकार के आधार पर पूरी तरह से अलग-अलग विशेषताओं को प्रदर्शित कर सकती है यानी बड़ी संख्या में बड़े छिद्रों या सामग्री में निहित छोटे छिद्रों की संख्या। इसलिए, ताकत और स्थायित्व गुणों की स्पष्ट समझ के लिए प्रतिनिधि पोर आकार का आकलन बेहद महत्वपूर्ण है।

एमआईपी (बुध घुसपैठ पोरसिमेट्री), एड्सोरप्शन, माइक्रोस्कोपिक इत्यादि जैसी कई तकनीकें हैं, जिन्हें वर्तमान में शोधकर्ताओं द्वारा पोयर संरचना मानकों के माप के लिए उपयोग किया जा रहा है। एमआईपी तकनीक सबसे व्यापक रूप से उपयोग की जाने वाली तकनीक है और पर्याप्त डेटा उपलब्ध है। ये सभी उपलब्ध तकनीक विभिन्न मान्यताओं और नमूना तैयारी तकनीकों और नमूने के आकार पर आधारित हैं। इन तकनीकों में विश्लेषण के लिए जटिल गणितीय अभिव्यक्तियां शामिल हैं। नमूना का आकार इन तरीकों से कंक्रीट मिश्रण का पूरी तरह से प्रतिनिधित्व नहीं करता है। ये उपकरण महंगी हैं और संचालित करने के लिए विशेष प्रशिक्षण की आवश्यकता है। इन सभी कठिनाइयों को दूर करने के लिए, उपलब्ध साहित्य को प्रतिनिधि पोर त्रिज्या के माप के लिए सरल तकनीक का प्रस्ताव देने के लिए सर्वेक्षण किया गया था।

कंक्रीट में तरल पदार्थ के प्रवेश के बारे में साहित्य की समीक्षा की गई, और बाद में इन परिवहन तंत्र के आधार पर मॉडल की स्थापना की संभावनाएं विकल्पों में से एक मानी गई। ताकत और स्थायित्व पूरी तरह से ठोस मैट्रिक्स में छिद्रों और वितरण की मात्रा पर निर्भर थे। हालांकि, स्थायित्व ठोस सतह से छिद्रों की अंत कनेक्टिविटी पर निर्भर करता है। परिवहन तंत्र के आधार पर पोयर संरचना मानकों के आकलन पर साहित्य उपलब्धता बहुत सीमित है और बहुत कम शोधकर्ताओं ने पारगम्यता के आधार पर पोयर त्रिज्या के आकलन का अध्ययन किया है।

छिद्र संरचना सीमेंट-आधारित सामग्रियों में सबसे महत्वपूर्ण सूक्ष्म संरचनात्मक विशेषता है क्योंकि यह सामग्री की ताकत और स्थायित्व को प्रभावित करती है। सीमेंट-आधारित सामग्रियों की ताकत ठोस रूप से छिद्रों की कुल मात्रा और ठोस के भीतर उनके वितरण से प्रभावित होती है। एक ही कुल पोर मात्रा वाली सामग्री छिद्रों के आकार के आधार पर पूरी तरह से अलग-अलग विशेषताओं

को प्रदर्शित कर सकती है यानी बड़ी संख्या में बड़े छिद्रों या सामग्री में निहित छोटे छिद्रों की संख्या। इसलिए, ताकत और स्थायित्व गुणों की स्पष्ट समझ के लिए प्रतिनिधि पोर आकार का आकलन बेहद महत्वपूर्ण है।

एमआईपी (बुध घुसपैठ पोरोसिमेट्री), एड्सोरप्शन, माइक्रोस्कोपिक इत्यादि जैसी कई तकनीकें हैं, जिन्हें वर्तमान में शोधकर्ताओं द्वारा पोयर संरचना मानकों के माप के लिए उपयोग किया जा रहा है। एमआईपी तकनीक सबसे व्यापक रूप से उपयोग की जाने वाली तकनीक है और पर्याप्त डेटा उपलब्ध है। ये सभी उपलब्ध तकनीक विभिन्न मान्यताओं और नमूना तैयारी तकनीकों और नमूने के आकार पर आधारित हैं। इन तकनीकों में विश्लेषण के लिए जटिल गणितीय अभिव्यक्तियां शामिल हैं। नमूना का आकार इन तरीकों से कंक्रीट मिश्रण का पूरी तरह से प्रतिनिधित्व नहीं करता है। ये उपकरण महंगी हैं और संचालित करने के लिए विशेष प्रशिक्षण की आवश्यकता है। इन सभी कठिनाइयों को दूर करने के लिए, उपलब्ध साहित्य को प्रतिनिधि पोर त्रिज्या के माप के लिए सरल तकनीक का प्रस्ताव देने के लिए सर्वेक्षण किया गया था।

कंक्रीट में तरल पदार्थ के प्रवेश के बारे में साहित्य की समीक्षा की गई, और बाद में इन परिवहन तंत्र के आधार पर मॉडल की स्थापना की संभावनाएं विकल्पों में से एक मानी गई। ताकत और स्थायित्व पूरी तरह से ठोस मैट्रिक्स में छिद्रों और वितरण की मात्रा पर निर्भर थे। हालांकि, स्थायित्व ठोस सतह से छिद्रों की अंत कनेक्टिविटी पर निर्भर करता है। परिवहन तंत्र के आधार पर पोयर संरचना मानकों के आकलन पर साहित्य उपलब्धता बहुत सीमित है और बहुत कम शोधकर्ताओं ने पारगम्यता के आधार पर पोयर त्रिज्या के आकलन का अध्ययन किया है।

पावर के मॉडल का उपयोग करके मापा गया पानी पारगम्य porosity अनुमानित porosity के साथ तुलना की जाती है।

गुप्तता मुख्य रूप से पानी-सीमेंट अनुपात, पेस्ट और आयु की मात्रा पर निर्भर करती है। यह भी देखा गया है कि सशक्तता इलाज की उम्र और हाइड्रेशन की डिग्री के साथ कम हो जाती है। पेस्ट वॉल्यूम में वृद्धि में पानी की पारगम्य porosity और sorptivity बढ़ जाती है। इसलिए, सीमेंट पेस्ट और कंक्रीट के लिए गुप्तता के आकलन के लिए एक अनुभवजन्य संबंध प्रस्तावित है।

चौथा, कंक्रीट की ताकत पानी-सीमेंट अनुपात से संबंधित है, और हाइड्रेशन की डिग्री, जो बदले में porosity या pore मात्रा से संबंधित हैं। शोधकर्ताओं ने ताकत (संपीड़न / विभाजन तन्यता) के आकलन के लिए विभिन्न प्रकार के ताकत-पोरोसिटी-पोर त्रिज्या मॉडल का प्रस्ताव दिया है। सशक्तता से मापा पोर त्रिज्या को सत्यापित करने के लिए, इन मॉडलों में से एक का उपयोग

कंक्रीट की अनुमानित ताकत के लिए किया जाता है। मापा और अनुमानित के बीच एक सतत संबंध मनाया जाता है।

ठोस संरचनाओं की स्थायित्व को स्थायित्व सूचकांक नामक वैज्ञानिक सिद्धांतों के आधार पर एक इंजीनियरिंग दृष्टिकोण द्वारा संबोधित किया जाता है। इस दृष्टिकोण को साइट गुणवत्ता नियंत्रण के साधन के रूप में, और दीर्घकालिक भविष्यवाणियों के आधार के रूप में, प्रदर्शन के आधार पर भौतिक विशेषता, स्थायित्व विनिर्देशों के बिंदु से विकसित किया गया है।

सूचकांक, स्थायित्व परीक्षण परिणामों और वास्तविक संरचनात्मक प्रदर्शन के बीच सहसंबंध की आवश्यकता होती है, जैसे इंडेक्स परीक्षणों का उपयोग इस प्रकार किया जा सकता है:

1. कंक्रीट की एक विशिष्ट संपत्ति को नियंत्रित करने का साधन, आमतौर पर कवर कंक्रीट की गुणवत्ता,
2. मानदंडों के एक समूह के अनुपालन के लिए निर्माण की गुणवत्ता का आकलन करने का साधन, और
3. डिजाइन पर्यावरण में कंक्रीट के प्रदर्शन की भविष्यवाणी करने का एक साधन।

अंत में, स्रावशीलता की जांच के दौरान उत्पन्न डेटा का उपयोग टिकाऊपन सूचकांक के अन्य रूपों के आकलन के लिए किया गया था जैसे जल अवशोषण के गुणांक, पानी का प्रतिशत

अवशोषण और स्पष्ट हाइड्रोलिक diffusivity। एक संरचना के सेवा जीवन की भविष्यवाणी करने में स्पष्ट हाइड्रोलिक विसारकता उपयोगी है। वर्तमान अध्ययन का मुख्य दायरा प्रतिनिधि पोर त्रिज्या के लिए पद्धति स्थापित करना है। इसलिए, वर्तमान अध्ययन सोरेंटता डेटा से स्थायित्व सूचकांक के अन्य रूपों की प्रस्तुति तक ही सीमित है।

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LIST OF SYMBOLS

A simplified notation is used when describing cement compounds, the cement shorthand notation:

A	=	Surface area of concrete in contact with water in cm^2 or area occupied by one gas molecule in $\text{m}^2/\text{molecule}$ or Al_2O_3 = Aluminium oxide (Alumina) or Wetted area in m^2
$\text{\AA}$	=	Angstrom ($1\text{\AA} = 10^{-10}\text{ m} = 10^{-4}\text{ }\mu\text{m} = 0.1\text{ nm}$)
A_p	=	Cross sectional area of pore in mm^2
A_s	=	Total surface area of pores in mm^2
<i>ACI</i>	=	American Concrete Institute
<i>ASTM</i>	=	American Society Testing Materials
B	=	Bolzmann variable in $\text{m/sec}^{0.5}$ or Fluid penetration coefficient
<i>BWS</i>	=	Boiling Water Saturation
$C(\text{CaO})$	=	Calcium oxide (Lime)
<i>CWS</i>	=	Cold Water Saturation
C_3S	=	(Oxide Formula = $3\text{CaO}.\text{SiO}_2$, Mineral Name = Alite, Chemical Formula = Ca_3SiO_5) = Tricalcium Silicate
C_2S	=	(Oxide Formula = $2\text{CaO}.\text{SiO}_2$, Mineral Name = Belite, Chemical Formula = Ca_2SiO_4) = Dicalcium Silicate
C_3A	=	(Oxide Formula = $3\text{CaO}.\text{Al}_2\text{O}_3$, Mineral Name = Aluminate/Clite, Chemical Formula = $\text{Ca}_3\text{Al}_2\text{O}_6$) = Tricalcium Aluminate
C_4AF	=	(Oxide Formula = $4\text{CaO}.\text{Al}_2\text{O}_3.\text{Fe}_2\text{O}_3$, Mineral name = Ferrite, Chemical Formula = $\text{Ca}_2\text{AlFeO}_5$) = Tricalcium Aluminoferrite
CaSO_4	=	Calcium sulphate (Anhydrite)
$\text{CaSO}_4.1/2\text{ H}_2\text{O}$	=	Calcium sulphate hemihydrates
$\text{CaSO}_4.2\text{ H}_2\text{O}$	=	Calcium sulphate dehydrate
CH	=	{Oxide Formula = $\text{CaO}.\text{H}_2\text{O}$, Mineral name = Portlandite, Chemical Formula = $\text{Ca}(\text{OH})_2$ = Calcium hydroxide}
$C-S-H$	=	Calcium silicate hydrate (sometimes called as Tobermorite gel)
$C_6\bar{A}\bar{S}_3H_{32}$ or $C_6(A, F)\bar{S}_3H_{32}$	=	Ettringite or Tricalcium sulfoaluminate {AFt phase, t = Tri, where (A, F) indicates aluminum with variable substitution of Iron}
$C_4\bar{A}\bar{S}_3H_{32}$ or $C_4(A, F)\bar{S}_3H_{32}$	=	Mono calcium sulfoaluminate {AFm Phase, m = mono, where (A, F) indicates aluminum with variable substitution of Iron}
$3\text{CaO}.\text{Al}_2\text{O}_3.\text{CaCl}_2.10\text{H}_2\text{O}$	=	Friedel's Salts (Calcium chloroaluminate hydrate)
$2c$	=	Elliptical crack of length
$c(\theta)$	=	Specific water capacity
D	=	Average cement particle size
$D(\theta)$	=	Capillary diffusivity or water diffusivity

d	Pore diameter or probability of occurrence of pores of a diameter d
$d_{0.5}$	= Median pore size corresponding to 50% cumulative pore volume
d_o	= Lower limit of distribution
d_u	= Upper limit of distribution
d^*	= Distribution parameter
$\frac{d}{d_o}$	= Relative pore diameter
$\frac{dh}{dt}$	= Velocity of the liquid rise in capillary
$\frac{dQ}{dt}$	= Volumetric flow rate
$\frac{dr}{dv}$	= Differential pore size
E	= Modulus of elasticity of material
$F (Fe_2O_3)$	= Iron oxide or Ferric oxide or Mass flux in kg/m^2
f	= Estimated compressive strength
$f(d)$	= Generalised lognormal distribution function
g	= Acceleration due to gravity in m/sec^2
$H = H_2O$	= Water
HCP or hcp	= Hydrated Cement Paste
h	= degree of hydration or distance penetration by the liquid (i.e. length of tube)
I	= Hydraulic gradient in m/m
ITZ	= Interfacial Transition Zone
i	= Mass of water per unit area in mm
$K = K_2O$	= Potassium oxide or Coefficient of permeability or hydraulic conductivity in m/s
K or $K(\theta)$	= Coefficient of permeability or hydraulic conductivity in m/s
k	= Intrinsic permeability of material in m^2 or constant equal to $\sqrt{\frac{\sigma}{2\mu}}$
L	= Thickness of the sample
LTC	= Low Temperature Calorimetry
l_c	= Mean tortuous length of the capillary
$M (MgO)$	= Megnesium oxide or Magnesia or Molar mass of the gas in cm^3/g mole
MIP	= Mercury Intrusion Porosimetry
m	= Mass of water gain in gm or mass of material in gm or mass of flowing substance
m_d	= Dry mass of material in kg
m_s	= Saturated surface dry mass of material in kg
m_{sub}	= Submerged or immersed mass of material in kg
$N (Na_2O)$	= Sodium oxide or Avogadro's Number (6.023×10^{23} molecules/g mole) or Number of pores in volume V
$NaCl$	= Sodium chloride
NMR	= Nuclear Magnetic Resonance
OPC	= Ordinary Portland Cement

<i>PSD</i>	=	Pore size distribution
<i>PC</i>	=	Plain Portland Cement
p_{cap}	=	Capillary Porosity
p_{gel}	=	Gel Porosity
p	=	Applied external pressure
p_o	=	Saturated vapour pressure
p_c	=	Capillary tension
p_e	=	Hydraulic pressure
$p(x)$	=	Compound density function
P_p	=	Perimeter of the pore cross section in mm
p_1, p_2, p_3	=	Atmospheric pressure, sample cell pressure and equilibrium pressure in N/mm ² respectively
Q	=	Flow rate
R	=	Universal gas constant (8.31 J/K.mol)
r	=	Radius of tube
r_{eff}	=	Effective pore radius
r_h	=	Hydraulic radius in mm
r_k	=	Kelvin Radius
r_m or $r_{0.5(cap)}$	=	Mean pore radius or Mean capillary pore radius
r_{max}	=	Maximum pore radius
r_{min}	=	Minimum pore radius
$S(SiO_2)$	=	Silicon dioxide (Silica) or sorptivity based on mass in mm/sec ^{0.5} or specific surface in m ⁻¹ or m ² /g
<i>SCM</i>	=	Supplementary Cementitious Materials
$\bar{S}(SO_3)$	=	Sulfur trioxide or alternative spelling Sulphur trioxide (Sulfate)
<i>SAXS</i>	=	Small angle X –ray scattering
<i>SEM</i>	=	Scanning Electron Microscopy
S_{BET}	=	Specific surface area
T	=	Temperature in Kelvin degree or Specific surface energy
T_o	=	Temperature of triple point water in K
t	=	Age in days or time in sec
U	=	Elastic strain energy
V	=	Total volume of the material or Molar volume of the absorbate or Volume of fluid absorbed in mm ³
<i>VAS</i>	=	Vacuum Saturation
V_b	=	Bulk volume of material in mm ³
V_m	=	Mass of the absorbed gas when the surface of the material is covered by monolayer molecules
V_p	=	Total pore volume in bulk material in mm ³
v	=	Cumulative pore volume or apparent velocity of flow or volume of water per unit area
v_c	=	Volume of the sealed empty sample cell
v_m	=	Molar volume of water in m ³ /mol
v_R	=	Volume of calibrated reference cell
v_{tot}	=	Total volume of material in m ³
v_{tr}	=	True volume of material in m ³
W	=	Total surface energy
W/C or w/c	=	water to cement ratio

x	= Variation
α	= Aspect ratio
σ	= Surface tension of mercury or water or surface tension of liquid-gas interface of the gas or geometric standard deviation or shape parameter
θ	= Contact angle of mercury- solid interface
ρ	= Density of water
ρ_{app}	= Apparent density (bulk density)
ρ_{tr}	= True density or real or skeletal density
$\emptyset$ or P	= Pore volume or porosity or water permeable porosity (are simply water porosity)
ΔH_m	= Change in the molar heat of the ice-water transition
ΔP	= Pressure drop
σ_{sl}	= Surface tension of mercury or water or surface tension of liquid-gas interface of the gas
μ	= Viscosity of the absorbing liquid and location parameter
θ	= Volume water content
ψ	= Capacity potential in m