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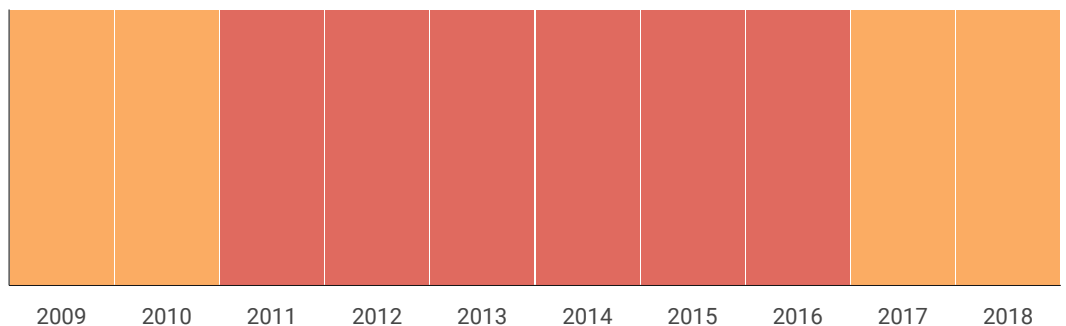
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H Index

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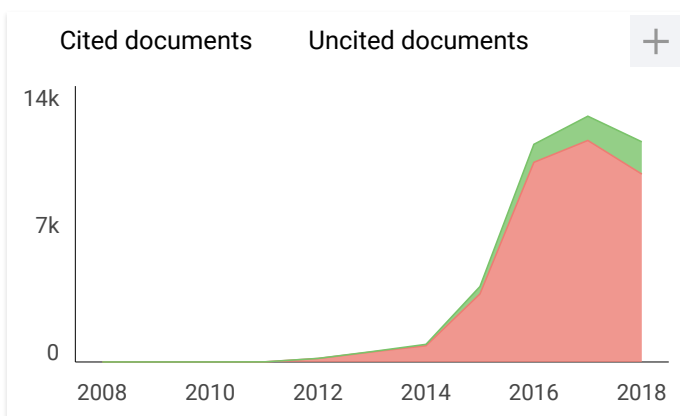
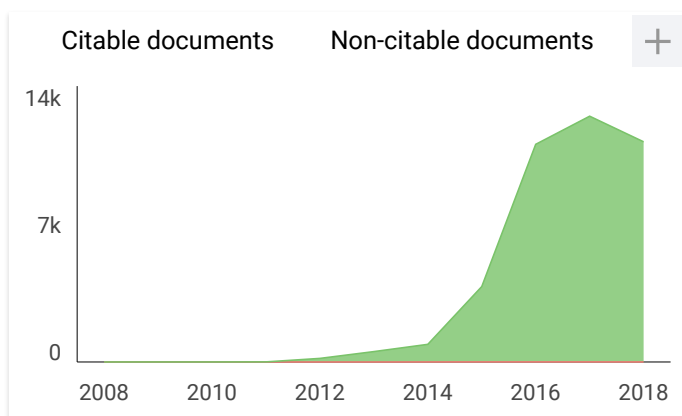
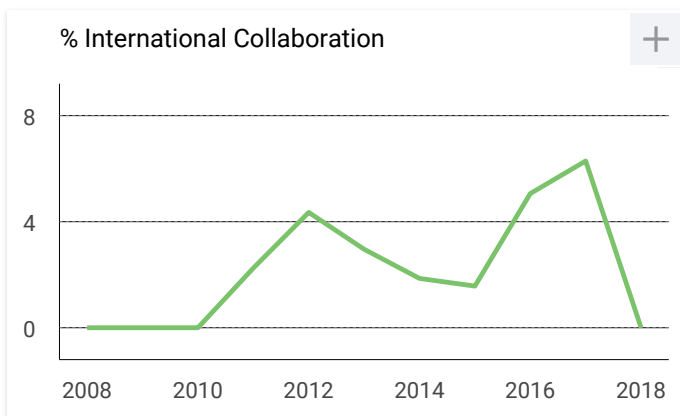
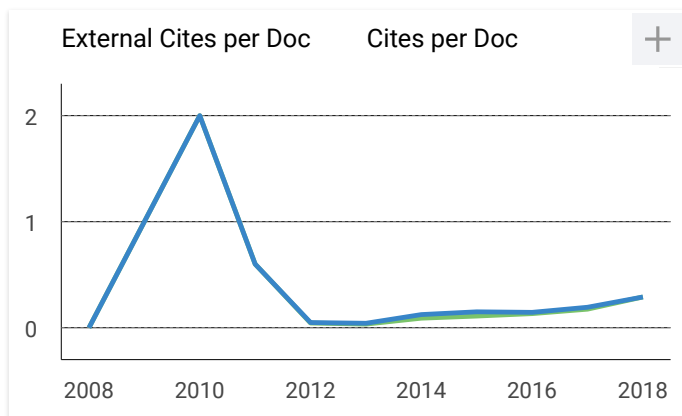
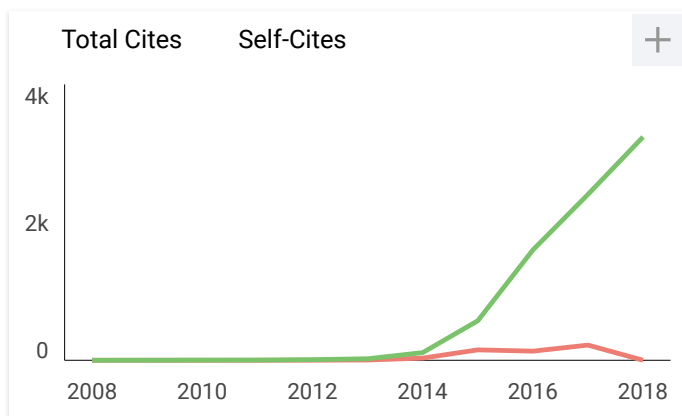
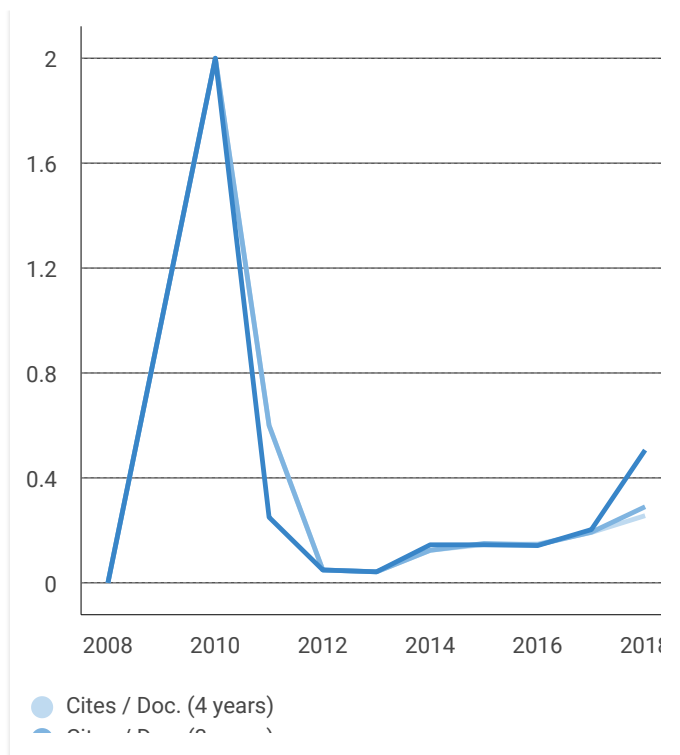
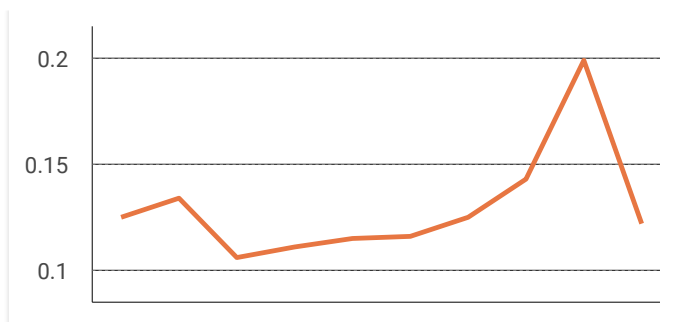


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Area of Interest, Active vibration control, PID control, Self-tuning/Adaptive Control, Evolutionary Algorithms, Particle Swarm Optimisation, Differential Evolution, Genetic Algorithm, System Identification

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Area of research/interest: thermoelasticity, plasticity, cyclic loading, structural instability, buckling.

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Stresses.

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Area of Interest: Finite Element Method, Meshless Method, FE-Meshfree Methods, Mechanical Vibrations

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Area of Interest: Polymer, Vibrational Spectroscopy, Electrospinning, Polymer characterization.

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Area of Interest: Polymer Nanocomposites, Polymer/Plastic, Ionomers, Nanocomposites, Blends, Water Treatment, Plasticizers, Additives, Electroactive Materials, Smart Materials, Fuel Cell, Lithium Ion Battery, Sensors, Actuators, Artificial Muscles, Membranes, Conducting Polymer, Biocompatible, Drug Delivery.

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Area of Interest: Sustainable Development, Soils, Foundations, Geomaterials, Ground Improvement, Engineering Education, Leadership, Higher Education

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Area of Interest: Surface and pressurized irrigation, Drainage engineering, Relationship between energy and environment Agricultural water management, Mathematical and computer modeling and optimization Water resources, Hydrology, Hydrogeology, Hydrometeorology, Hydro informatics, Hydrodynamic Hydraulic, Fluid mechanics, Heat transfer in soil media

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Area of Interest: VLSI, Microelectronics, Device Physics.

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Area of Interest: Neural Networks, Image Processing, Artificial Intelligence, Data Mining & Data Warehousing, Computer Networks

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Area of Interest: Data Mining, VLSI Physical Design, Computer Networks, Embedded systems.

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Area of Interest: Environmental pollution, Environmental management, Environmental

toxicology, Environmental decision-making, Environmental policy, Waste management, and Sustainability Science.

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R. Manikandan, ICT Department, School of Computing, SASTRA University, Thanjavur, Tamil Nadu, India

Area of Interest: Data Mining, VLSI Physical Design, Computer Networks, Embedded systems

CONTENTS

PAPR Reduction in Wavelet based SCFDMA using Pulse Shaping Filters for LTE Uplink Transmission

pp. 6481-6492

Authors: Ishu and Naresh Kumar

Analysis of Combustion and Performance Characteristics of Low Heat Rejection Engine using DIESEL-DEE Blended Fuel

pp. 6493-6505

Authors: T. Mohanraj, S.Krishnamani and R.Suresh

Islanding Detection of Inverter Based DG Unit Using Vdc-Vpcc Characteristics

pp. 6507-6525

Authors: M. Divyasree and Dr. L. VenkataNarasimha Rao

Cost Effective Implementation of 3D Space Vector Modulation Using Matlab-Aurduino Interface

pp. 6525-6541

Authors: Ashok Kumar V, Narasimha Raju K and Dr. M Venu Gopala Rao

Common Fixed Point Theorem for Two Self Maps in a Cone Metric Space with ω -Distance

pp. 6543-6550

Authors: K.P.R. Sastry, A.Chandra Sekhar and K.Sujatha

Study on Selection and Satisfaction Level of Mobile Phone Service Users: A Study in Chennai City

pp. 6551-6559

Authors: Muthumani S and Krishna Priya V

User Authentication using Multimodel Face Recognition

pp. 6559-6570

Authors: Prashant Kumar Jain, Shailja Shukla and S. S. Thakur

On Identification and Modification of Verbose Queries for Effective Information Retrieval

pp. 6571-6580

Authors: K Hiba Sadia, Kallu Sahitya, A Antony Rebecca, Kirti V. Meshram, Praviya Bharati and Saravanakumar K

A Novel Architecture and PVT Analysis of 4-Bit Manchester Carry Chain Block

pp. 6581-6590

Authors: Ankit Mehta, Satyendra Kumar and Rohit Kumar

Mining Human Opinion Patterns Using Weighted Substructure DAG algorithm

pp. 6591-6605

Authors: K.Aparna and k .Venkataraju

Multivariate Statistical Quality Control Applications in Biomedical Industries

pp. 6607-6617

Authors: Mrs. G. Annalakshmi, Dr. S.P. Rajagopalan and Dr. A. Iyem Perumal

A New Concept of Simultaneous Voltage SAG or SWEEL and Load Reactive Power

Compensation Utilizing UPQC with ANN Technique

pp. 6619-6633

Authors: Bollapalli Anusha and K. Sarada

A Relative Performance Comparison of Double Output Asynchronous Generators Using Power Transfer Matrix and Direct Power Control Techniques

pp. 6635-6648

Authors: Viswanadha S Murthy K and Dr. G. R. K. Murthy

Study on Cactus Extract as a Hydrophobic Admixture in Lime Mortar

pp. 6649-6659

Authors: Ravi. R and Dr. Sekar S. K.

Segmentation Performance of Mosaic Textures Using KMeans with CC

pp. 6661-6673

Author: Dr. Shoba Rani

Security and Reliability using Wireless Sensor Network for Industrial Automation Using Harmony Search Algorithm

pp. 6675-6684

Authors: Meenatchi.S and Navaneethan.C

IDS and IPS Using ZigBee with GSM

pp. 6685-6698

Authors: Meenatchi.S and Navaneethan.C

A Novel Approach for Knowledge Mining from Graphs using Semantic pp.6699-6706

Authors: Hemamalini. S, MichaelRaj T.F, Prabu.M and Saravanan.N

Authenticated Data Transmission in Decentralized Wireless Mobile Ad-Hoc Network (;MANET)

pp. 6707-6714

Author: Mr. Vishal Rajput

WDM PON Long Haul System Design for 10 Gb/s using Switches for DBA

pp. 6715-6726

Authors: S. Rajalakshmi, Ananth Vijaya Venugopalen and Anirudh Kowtha

Detection of an Incognitos Intruder in Industries and Semantic Mapping of Emotions

pp. 6727-6734

Authors: Dr. R. Subhashini, E. Nagarajan and Niveditha.P.R

Finding Fuzzy Critical Path by Metric Distance Ranking Method Using Fuzzy

Numbers

pp. 6735-6745

Authors: S.Narayanamoorthy and S.Maheswari

Learning to Identify Bad Coding Practice

pp. 6747-6755

Authors: Gowtham Deivanayagam. K, Gayathiri. D, Manikandan. A, Raghul Karthik K R, Dr. G. Jeyakumar and Kriti. N

An Intelligent Technique to Detect ARP Spoofing in Local Area Network

pp. 6757-6764

Author: E. Vijayan and Ravi Chaurasia

A New Technique for the Reconfiguration of Radial Distribution Network for Loss Minimization

pp. 6765-6777

Authors: N. H. Shamsudin, M. S. Mamat, A.F.A.Kadir, M. F. Sulaima and H. I. Jaafar

An Efficient Operational Matrix based Approach for a Few Nonlinear and Fractional Differential Equations Arising in Engineering

pp. 6779-6797

Authors: M. Salai mathiselvi, G. Hariharan and B. Sripathi

Dynamic and Effective Stock Analyser using Data Mining Techniques

pp. 6799-6812

Authors: Muhammad Rukunuddin Ghalib, Senthil Kumar N C and Sasikumar Gurumurthy

A Key Management & Establishment Scheme in Heterogeneous Wireless Sensor Networks (HWSN)

pp. 6813-6821

Authors: Premamayudu B, Venkata Rao K and Suresh Varma P

Factorial Analysis of Ferulic Acid Extraction from Banana Stem Waste

pp. 6823-6833

Authors: S.N. Ismail and N. Zainol

Use of Non-thermal Microwave Plasma for Syngas Production from Dry Reforming of Compressed Biomethane

pp. 6835-6842

Authors: E. Chaiya, P. Khongkrapan and N. Tippayawong

Finding Fuzzy Critical Path by Metric Distance Ranking Method Using Fuzzy Numbers

pp. 6843-6854

Authors: S. Narayanamoorthy and S. Maheswari

Study of Reed Solomon Encoders and its Architectures

pp. 6855-6862

Authors: A. Deepa and C.N. Marimuthu

A New Approach to Improve the Efficiency of Photovoltaic Power through MPPT Techniques

pp. 6863-6872

Authors: Guddanti Gowthami and Dondapati. Ravi Kishore (Ph.D)

Development and Evaluation of Suitable Prototype Electrical Power System for Running an Air Conditioner using Solar Panel

pp. 6873-6882

Kondapi Sreedhar, Chandra Sekhar Garlapati and A. Seshu Kumar

Laser Cladding of Ti-6Al-4V Alloy Using TiC Particles

pp. 6885-6894

Authors: Essam R.I. Mahmoud and Hashem F. El-Labban

Development of Automatic Load-Shedding Strategy for Stand-Alone Photovoltaic System

pp. 6895-6906

Authors: M.N.M. Nasir, M. M. Farith, Mohd Hafiz Jali, M. S Jamri and H.I. Jaafar

FPGA Implementation of Back Propagation Algorithm for ANN using Verilog HDL

pp. 6907-6913

Authors: V V S Vijaya Krishna, K Sai Krishna and P Siva Prasad

Using Multivariate Statistical Quality Control Tool in Medical Industry

pp. 6915-6928

Authors: Mrs. G. Annalakshmi, Dr. S.P Rajagopalan and Dr. A. Iyemperumal

Incorporation of Radix- $\diamond\diamond\diamond\diamond$ Feed for Ward FFT and Adaptive Viterbi Decoder into OFDM for Wireless Applications

pp. 6929-6940

Authors: S. Prabu and E. Logashanmugam

Active and Reactive Power Control in Matrix Converter Based UPFC Fed Induction Motor Drive Using DPC Scheme

pp. 6941-6957

Authors: Katta Raja Sekhar and Mr. P.V. Pattabhiram

Circular Array Antenna Synthesis based on Element Spacing

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Sayavur I. Bakhtiyarov, New Mexico Institute of Mining and Technology,

Authors: Rekha.G.Pai and A.Kandasamy

Analyzing the Services and Privacy, Conflict Resolutions of Shared Data in OSN's
pp. 7103-7112

Authors: Doma Venkata Manasa, Dr. M. R. Narasinga Rao and A. S. Lalitha

EHMBA: An Efficient Heuristic-Based Multihop Broadcast Protocol for Asynchronous Duty-Cycled Wireless Sensor Networks
pp. 7113-7126

Authors: S. Pramatha, Dr. N. K. Sakthivel and Dr. S. Subasree

A Binary Schema and Computational Algorithms to Process Vowel-based Euphonic Conjunctions for Word Searches
pp. 7127-7142

Authors: Kasmir Raja S. V., Rajitha V. and Meenakshi Lakshmanan

Designing and Selection of Mixed Sampling Plans with Two Sided Complete Chain Sampling as Attribute Plan
pp. 7143-7148

Authors: K. Rebecca Jebaseeli Edna, V. Jemmy Joyce and S. Deva Arul

Secured Mining with Storage Efficiency and Privacy Guarantee on Outsourced Transaction Databases
pp. 7149-7158

Authors: A.Leo Paul and W.R. Helen

Estimation of Ku Band Satellite Signal Propagation Impairment Due to Rain in Tropical Environment Using ITU-R
pp. 7149-7168

Authors: Govardhani. Immadi, Sarat K Kotamraju, Habibulla Khan, M.Venkata Narayana, Hemavasavi.K, K. Pooja Naga Sai and N. Sirisha

Multi keyword Ranked search over Encrypted Cloud Data
pp. 7149-7176

Authors: Christal Joy.E and Indira.K

An Approach towards Defense of DDOS Attacks in Cloud Computing using Confidence based Filtering and Hop Count Filtering Techniques
pp. 7177-7190

Authors: P. Boominathan, K. Marimuthu, Apoorva Shenoy, Garima Hooda and Satish Reddy

Analysis of Ball and Roller Burnishing by Optimizing the Process Parameters for Surface Roughness Indices using Grey Based Taguchi Method
pp. 7191-7200

Network, Ajman, UAE

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pp. 7295-7304

Authors: Mr. Subba Rao, Dr. K. Venkata Rao and Dr. P. Suresh Varma

Designing Multidimensional Mixed Sampling Plans for Second Quality Lots

pp. 7305-7309

Authors: V. Jemmy Joyce, K. Rebecca Jebaseeli Edna and S. Deva Arul

Implementing Histogram Equalization and Retinex Algorithms for Image Contrast Enhancement

pp. 7311-7318

Authors: M. Prabhu, S. Rajarajan and M. P. Karthikeyan

Design of Renewable Energy Harvester using Smart IC and System

pp. 7319-7332

Authors: Venugopal. P, Sai Parimal Rohit, Dr. Suresh. Y and Modi Pandu Ranga Prasad

A Comprehensive Behavior Analysis of TOR versus I2P

pp. 7333-7345

Authors: Karthigeyan A, Robinson Joel M, Manikandan S P, Raja Guru P and Sri Raman S

Indian Sign Language Recognition using Skin Colour Detection

pp. 7347-7360

Authors: Ashok Kumar Sahoo and kiran Kumar Ravulakollu

I Safe: Secure Global Money Transfer with Automated Currency Converter

pp. 7361-7370

Authors: Shivramvelseethapathi, Hariprashanth and Viswanathan. R

The Role of Environmental Engineers in Management of Natural Disaster

pp. 7371-7376

Authors: Akshaya Kumar Sabat and Saroj Kumar Dash

A Robust Watermarking Technique Based on DCT and Image Normalization

pp. 7377-7383

Authors: E. Vijayan, S. Hemalatha, Senthilkumar N C Kuldeep Singh Yadav, Pankaj Kumar Nayak and Parul Gupta

An SPR_SD Model Used to Predict Dengue Fever

pp. 7385-7394

Authors: V.Srinivasan, A.Iyem Perumal, S.P.Rajagopalan and S.Dheva Rajan

Naturalistic Traffic Data Collection Using Stereo Vision Systems

pp. 7395-7410

Authors: Salvatore Cafiso and Alessandro Di Graziano

Authors: Doradla Raja Ramesh, A. M. K. Prasad and A. C. S. Kumar

Experimental Study of Adaptive Power and Modulation-A Cognitive Radio Application

pp. 7201-7212

Authors: Vijaya Kumar Ponnusamy and Malarvizhi .S

Modeling of Air Quality Index in the Eastern Urban Areas of Thailand using Neural Network Method

pp. 7213-7220

Authors: K. Saithanu and J. Mekparyup

Sans Douleur Continuous Glucose Scrutinizer System

pp. 7221-7225

Authors: B. Bharathi and Aathilakshmi

Power Quality Improvement Using Neuro-fuzzy based Custom Power Devices (UPQC) in Wind Farm Connected to Weak Grid

pp. 7227-7240

Authors: Kopella Saiteja and R. B. R. Prakash

Novel Intellegent Control Technique based on Space Vector Modulation Direct Torque Control for Induction Motors

pp. 7241-7256

Authors: Srinivasulu Budharapu and Bhavani Juppali

Multi-Channel E-Learning System based on Semantic Web Service Architecture

pp. 7257-7264

Authors: R.Sethuraman and Dr.T.Sasiprabha

A Study of Adaptive Modulation for Space-Time Trellis Coded Multiple Input Multiple Output Systems with Imperfect Channel State Information

pp. 7265-7273

Authors: Anusree. L and Prof. (Dr.) Sheeja. M. K

Complete Analysis of Joint Inference in Information Extraction Using Markov Logic

pp. 7275-7285

Authors: J. Refonaa and Dr. Lakshmi

Performance Behaviour of Cryptography Algorithms in Aspect based Web Services

pp. 7287-7293

Authors: S. Murugan and B. Muthukumar

Communication and Computational Cost Efficiency in Automatic Test Generations

Catalytic Performance of Al-HDTMA Bentonite Impregnated Fe on Phenol Hydroxylation

pp. 7521-7529

Authors: Restu Kartiko Widi, Arief Budhyantoro and Emma Savitri

An Authentication Method for Secure Web Services Access with Preventing Tautology Type SQL Injection

pp. 7531-7543

Authors: M.D.Anto Praveena, R. Joseph Manoj and Dr.V.Shanthi

Reduction of I/O Delay by Incorporation of Reconfigurable Processing Units into the High Level Synthesis of DSP Applications

pp. 7545-7560

Authors: Awni Itradat and M.O. Ahmad

Design, Simulation and Performance Analysis of Digital FIR Filter based on Low-Power Reversible Gates

pp. 7561-7574

Authors: W.Sujan and A.V.M.Manikandan

Energy Efficient Coverage Estimation for Wireless Sensor Networks from Real Time RSSI Measurements for Indoor Localization

pp. 7575-7588

Authors: K.Vadivukkarasi and R.Kumar

Proposing a Model for User Satisfaction in Electronic Human Resource Management

pp. 7579-7595

Authors: S.K.Manivannan and Dr.A.Chandra Mohan

Cross-Layer Optimization for Multichannel Multiradio Wireless Network with Network Coding

pp. 7597-7608

Authors: Geunseok Choi and Wonsik Yoon

Tamil Word Sense Disambiguation using Support Vector Machines with Rich Features

pp. 7609-7620

Authors: Anand Kumar M, Rajendran S and Soman K.P

Studying the Impact of HSS based Drills and Pre-drills on Delamination during Drilling Chopped Strand Mat Glass Fiber Reinforced Polymer

pp. 7621-7632

Authors: Panneerselvam T and Raghuraman S

Student Monitoring Using Opencv

pp. 7411-7418

Authors: R. Sethuraman and E. Vaitheeswaran

Numerical Study on Effect of Axial Gap in the Flow Dynamics of Gas Turbine

pp. 7419-7433

Authors: Aji M Abraham and Suresh.M.S

A Secured Cloud System and Log Records based on 2LE

pp. 7435-7451

Authors: K Marimuthu, D Ganesh Gopal, Ginni Malik and P Boominathan

Estimation of Body Segment Weights for Prosthetic Legs suitable to Indian Amputees

pp. 7453-7462

Authors: Y.Kalyan Chakravarthy, D.Tarun and Dr.A.Srinath

An Optimized Algorithm for Generating Subsets

pp. 7463-7468

Authors: K. Arulmani, P. Swaminathan and K. Chandrasekhara Rao

A Study on Big Data and its Importance

pp. 7469-7479

Authors: Duvvuri.B.S.Suresh Kumar, D.Bala Krishna Kamesh and Dr. Syed Umar

The Influence of Flow Steering Angle on the Performance a of Cup-Bladed Kinetic Turbine

pp. 7481-7489

Authors: Nita C.V. Monintja, Rudy Soenoko, Slamet Wahyudi, Yudy S. Irawan

Quantification of Environmental Services on Hydro Power Plant of Jelok and Timo

pp. 7491-7498

Authors: Purboseno, S., Bambang, A.N, Suripin and Hadi, S.P

Performance of a 3-Phase Asymmetrical Cascaded Subcell Multilevel Inverter

pp. 7499-7508

Authors: Dhanamjayulu C, Dr. Y Suresh, Ponnambalam Pathipooranam and Rashmi Ranjan Das

Data Clustering using Principal Component Analysis and Differential Evolution

pp. 7509-7520

Author: Rajashree Dash

Authors: B. Priyadarshini and S. Sivasundarapandian

An Analysis on 1-Step Transition Probability Matrix and 2- Step Transition Probability Matrix of Markov Passwords

pp. 7745-7753

Authors: S.Vaithyasubramanian and A. Christy

Data and Information Storage Security from Advanced Persistent Attack in Cloud Computing

pp. 7755-7768

Authors: J.Vijaya Chandra, Dr. Narasimham Challa and Dr. Mohammed Ali Hussain

A Survey of Research Dimensions in Complex Event Processing

pp. 7769-7780

Authors: C.Imthyaz Sheriff and Dr.Angelina Geetha

Advancement in Vehicle Airbag Deployment System

pp. 7781-7789

Authors: Mr. Ishpreet Chawla, Mr. Mandeep Rana and Mr. Yash Parikh

Priority Based Traffic Light Controller with IR Sensor Interface using FPGA

pp. 7791-7800

Authors: B Murali Krishna, K Gopi Vasanth Kumar, A Gnandeep Reddy, N Madan Gopal, K Varun Chowdary, B T P Madhav

Minimizing the Distribution of Ready-Mixed Concrete with "Out of Kilter" Algorithm

pp. 7801-7812

Authors: Mir Heydar Hashemi and Orhan Yuksel

The Strategy to Coordinate Contractors' Statement of Work with Scheduling

pp. 7813-7820

Authors: Mir Heydar Hashemi, Elaheh Sharifi and Mobin Sameie Paghaleh

Size Effect on Deformation Characteristic of Aluminum under Impact Loading Condition

pp. 7821-7833

Authors: Hairul Arsyad, ING Wardana, Wahyono Suprpto and Anindito Purnowidodo

Minimizing the Distribution of Ready-Mixed Concrete with "Linear Programming"

pp. 7835-7846

Authors: Mir Heydar Hashemi and Orhan Yuksel

Employees' Commitment and Credibility towards Job and Organization in Indian

Secure and Competent Information for Mobile and Desktops

pp. 7633-7638

Authors: B. Bharathi and L.K. Joshila Grace

Lateral Load Resisting Capacity of RC Ductile Framed Structure Using Non Linear Static Analysis

pp. 7639-7652

Authors: R. Ponnudurai, K. Swaminathan and Dr. S. Nagan

Automated Anti-theft and Accident Detection System for the Elderly

pp. 7653-7666

Authors: Rohan Kulkarni, Sahil Karkhanis, Abhishek Tripathi and Yokesh Babu Sundaresan

Classification of Flooding Attacks using Severity Labeling based Machine Learning Techniques

pp. 7667-7678

Authors: Prathibha R.C and Smt. Rejimol Robinson R.R

Optimized Regression Testing using Genetic Algorithm and Dependency Structure Matrix

pp. 7679-7690

Authors: J. Albert Mayan and T. Ravi

Flow of a Micropolar Fluid in an Inclined Channel Bounded by Permeable Beds

pp. 7691-7704

Authors: K. Nandagopal, S. Sreenadh K. Chakradhar and P. Lakshminarayana

Rule Based Labeling and Maxentropy based Learning for Mining Wishes from User Reviews

pp. 7699-7704

Authors: Shruthi Raveendran Nair and Chitharanjan K.

Mining and Predicting Customer Transactions– A Novel Approach

pp. 7705-7716

Authors: Karthika Surendran and KuttyMalu V.K

Surfing Large Websites based on User Behaviour

pp. 7717-7726

Authors: D.B.K Kamesh, J.K.R. Sastry and M. Devi Kavya Priya

A Miniaturized Circular Microstrip Patch Tri Band Ring Antenna for SATCOM Applications

pp. 7735-7743

Facilitation of Scientific and Technical Big Data Sharing

pp. 7959-7970

Authors: Sang-Gi Lee and Eui-Kyeong Hong

The Effects of Self-esteem, Depression and Stress on Students' Adjustment to College

pp. 7971-7980

Authors: Young-Sook Kwon

A Study on Improving the Batch Registration Process for Collecting National Research and Development Information

pp. 7981-7992

Authors: Tae-Hyun Kim, Myung-Seok Yang, WonKyun Joo, MinWoo Park, NamGyu Kang and Kwang-Nam Choi

Development of User Interface for Game Developer using NGUI and Mecanim Technique

pp. 7993-8002

Authors: Sung-Su Kim, Shin-Jin Kang, Seok-Hun Kim and Soo-Kyun Kim

Two Prospects on Nowadays: Smart Convergence & Big Data

pp. 8003-8011

Author: Byung-Tae Chun

An Effective P-Peak Detection Algorithm for HRV Analysis

pp. 8013-8020

Authors: Wen Hai Jin, Bo Yeon Kim and Yun Seok Chang

Smart System for Food Safety to Advance Feed Service Culture

pp. 8021-8028

Authors: Ki-Bong Kim and Sung-Han Lee

Decision Factors for Supplier Selection

pp. 8029-8039

Authors: Hyun Gi Hong

Real Time Signal Analysis for Modeling the Performance of UHF/VHF Transceivers

pp. 8041-8066

Authors: A. Jhansi Rani, K. Ch. Sri Kavya, Sarat K Kotamraju, G. Sree Teja and K.Snigdha

Library Book Recommendation System Using CF-Apriori Algorithm

pp. 8067-8074

Authors: Shriladha B, Suganya Magudeswaran, Sini Raj P and P Subathra

Moving Objects Detection for Video Surveillance Applications

pp. 8075-8082

K. Eswar Chaitanya, G. Harish and B. Harish

Improved Page Rank Algorithm Using Efficient Damping Factor

pp. 8083-8091

Authors: Dr. V. Vaithiyanathan, M. Rajasekhar Reddy, Anishin Raj M M, B. Karthikeyan, G. VThanushree, Diana Baby and S. Nesha (a) Sindhu

Effects of Participation in Volunteering Activities on Nursing Professionalism and Self-Esteem among Nursing Students in Korea

pp. 8121-8128

Authors: Soyoun Yim

Design and Analysis of a Novel Multifractal Multiband Antenna using 3D-FDTD Method

pp. 8129-8139

Author: Vivek Dhoot

Comparison of Self-esteem and Successful Aging According to Social Networks among Elderly Men in Korea

pp. 8141-8154

Authors: Hee Kyung Kim

Academic Achievement of Blended Problem Based Instruction for University Students in Korea

pp. 8155-8164

Authors: Myeong-Hee Shin and Eunpyo Lee

Estimation of the Congestion Rate of Local Public Goods in Korea

pp. 8165-8174

Author: Sung Tai Kim

The Relations between the Status and Needs of Full Dentures and Fixed or Removable Partial Dentures, and Periodontal Disease for Korean Adults

pp. 8175-8188

Authors: In-Ho Jeong, Jong-Hwa Lee and Myung-Ja Park

A Study of Dental Caries Condition and a High Caries Risk Group for 12-years-old Korean Children

pp. 8189-8198

Authors: In-Ho Jeong, Jong-Hwa Lee, Myung-Ja Park and Hyun-Kyun Yun

The Effect of Eating Habit and Oral Health behavior on Subjective Perception of

Oral Health for Korean Adolescent

pp. 8199-8210

Authors: In-Ho Jeong, Jong-Hwa Lee, Myung-JaPark and Jin-Yeong Yoo

Luteolin Suppresses Nitric Oxide (NO)-induced Dedifferentiation and Enhances Cyclooxygenase-2 (COX-2) Expression in Rabbit Articular Chondrocytes

pp. 8211-8220

Authors: Seong-Hui Eo and Song-Ja Kim

Catalytic Performance of Al-HDTMA Bentonite Impregnated Fe on Phenol Hydroxylation

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ABSTRACT

This paper describes the addition of Fe into modified-bentonite layer by impregnation method. Natural bentonite (from Pacitan, Indonesia) was intercalated with HDTMA-Br 1,5 % solution before pillared with Al metal to give Al-HDTMA bentonite forms. The ratio of bentonite and intercalating agent or pillaring agent was 1 gr/50 ml. The mixture was agitated, and then the solid phase was washed with distilled water. Then it was dried and calcined at 723 K for 4 hours. This modified bentonite was then impregnated with Fe solution. The Fe concentrations were 0.01 M, 0.05 M, and 0.1 M. All the materials were characterized using FT-IR and X-ray diffraction. X-ray diffractogram showed that the higher Fe penetrates into bentonite, the lower the crystallinity of bentonite. Their catalytic activity and selectivity were studied for phenol hydroxylation using H₂O₂ (30%). The reaction conditions of this reaction were as follows: ratio of phenol/ H₂O₂ = 1:1 (molar ratio), concentration of phenol = 1 M, reaction temperatures were 333 K, and ratio of catalyst/phenol was 1:10. The best catalytic performance to convert phenol and produce hydroquinone by phenol hydroxylation reaction is on PILB HDTMA-Al,Fe 0.05 M.

Keywords: bentonite, impregnation, catalyst, phenol hydroxylation

INTRODUCTION

Clay is one of the most abundant material found in nature. Bentonite, the material which is used in this research, is natural bentonite from Tulakan, Pacitan in east Java. Bentonite consists of montmorillonite mineral as the major component. Up to now, bentonite can be utilized as filler, ion exchanger, catalyst, adsorbent etc.

Bentonite has characteristics such as moisture content as high as 30-40 %, and easy to be swelling. The characteristics cause unstable structure and make low selectivity for both adsorbent and catalyst. Otherwise, the interlayer spacing of the swollen clay is sufficient to receive the Al and Al/Fe polyoxocations [1]. Because of that, bentonite has to be modified to get more stable structure. One of the ways to modify bentonite is by pillarisation process [2]. The application fields of these substrates are very broad because of the possibility to intercalate different types of pillars making them suitable for specific adsorption and catalytic processes.

The introduction of inorganic pillars in the natural bentonite improves its resistance and stability, increases its micro porosity and provides larger surface area and accessibility to its acid sites (Brönsted and Lewis sites) [3,4]. Pillared bentonite can be utilized as catalyst because of the reason that pillared bentonite has active sites such as silica group (SiO_4) and alumina group (AlO_4). The presence of active metal which is penetrated into bentonite structure during pillarisation process can enhance the catalytic performance of pillared bentonite. Other wise, the presence of pore which has definite size will give the ability to select both of reactants and products.

To increase its selectivity, some modifications of bentonite have been done. One of the modifications is clay intercalation using organic molecules (anionic, cationic and non-ionic molecules). This process cause the distance between clay molecules becomes longer, so that the pore size becomes bigger. As a result, the bigger molecules (organic molecules usually have big molecular size) can be laid on the bentonite surface where the adsorption and/or catalytic process is occur [5-7]. In addition, the other modification is clay pillarisation using metal oxides [5,6,8]. Various works have been reported on the catalyst synthesis [9-13]. They show that catalytic activity depends on several factors such as chemical composition, preparation method, calcinations temperature and characteristic of catalyst surface.

In environmental catalysis there is a great interest in obtaining pillared clays containing iron (oxides or oxyhydroxides) species use in the catalytic wet peroxide oxidation of toxic organic compounds since many industrial processes yield a variety of organic contaminants which are poured into natural water sources with negative impact on ecosystem and humans (toxicity, carcinogenic and mutant properties) [14]. Among these pollutants contained in industrial residual water we can highlight phenol, substituted phenols (chlorophenol, nitrophenol), oxalic acid, acetic acid, pesticides and herbicides [14]. Phenol is particularly considered as one of the most toxic organic contaminants and it is commonly chosen as model molecule on catalytic oxidation of organic compounds studies in diluted aqueous medium [15]. Several processes of catalysis industrial importance have been reported over pillared clays [16-18].

This work shows the synthesis of solid catalyst, which is bentonite was intercalated using Cetyl-Tetramethylammonium Bromida, CTMA-Br or HDTMA then pillared by Al. Al-HDTMA bentonite was then pillared with Fe by impregnation method, in which iron is introduced as active phase (activation sites for hydrogen peroxide as well as organic molecules) in order to develop the catalyst material to the complete oxidation of organic molecules.

EXPERIMENTAL SECTION

Materials

Clay (natural Pacitan bentonite), FeCl_2 (Merck), Cetyl-Tetramethylammonium Bromida, CTMA-Br or HDTMA-Br (Merck), NaOH (Merck), $\text{Al}(\text{OH})_3$ (Merck).

Instrumentation

Fourier Transform Infra Red Spectrophotometer (FTIR, Bruker Tensor 27, Germany), X-ray diffraction (XRD, Shimadzu XRD 1000)

Procedure

The starting clay was a natural Pacitan bentonite, extracted from Pacitan region, East Java. Cationic polyoxy Fe solution is prepared by dissolve and stir Fe salt and NaOH in water. Clay suspension is prepared by dissolve 1 gr of bentonite in water. Polyoxy solution and suspension are mixed and stirred. The solid is washed and then dried in the oven at 373 K. Intercalation is carried out by adding 1 gr bentonite suspension into 50 ml surfactant (Cetyl-Tetramethylammonium Bromida, CTMA-Br or HDTMA) solution. The materials were calcined at 723 K for 4 hours.

The pillaring agent solution was prepared by mixing NaOH and Fe which has molar ratio OH to Al = 0.8. Pillarisation process of the clay was carried out by mixing HDTMA-bentonite and pillaring agent solution with ratio [gram bentonite/volume of solution] = 1 gram/50 mL. The mixture was heated at 343 K. After 5 hours, the mixture was cooled, and washed with aquadest. The obtained solid was dried in the oven at 373 K. This modified bentonite was then impregnated with Fe solution. The Fe concentrations were 0.01 M, 0.05 M, and 0.1 M. After impregnation process, Al-HDTMA bentonite was calcined at 773 K for 4 hours with nitrogen and oxygen stream.

The chemical analysis of the solids was carried out by Fourier Transform Infra Red Spectrophotometer (FTIR, Bruker Tensor 27, Germany) to characterize bentonite structure. The X-ray diffraction (XRD) study was done in Shimadzu XRD 1000 device. It identified crystalline structure and d_{001} spacing of pillared bentonite.

The catalytic oxidation reaction of phenol in a diluted aqueous medium was carried out in a batch glass reactor equipped with thermometer, and reflux condenser. It is open to the atmosphere, and thoroughly stirring with magnetic stirrer. Firstly, the reactor was loaded with phenol solution and catalyst and heated up until 333 K. The reaction was conducted at 333 K for 4 hours. The hydrogen peroxide solution was added stepwise during a reaction time of 4 hours. The molar ratio of phenol to hydrogen peroxide = 1:1. The experiment variables are the weight ratio of catalyst to phenol = 1:10, and 5:10. The course of the phenol conversion and selectivity was followed by high performance liquid chromatography (HPLC) by means of Knauer.

RESULTS AND DISCUSSION

Characterization

FTIR spectra

Characterization was conducted to Al pillared HDTMA-bentonite (PILB HDTMA Al)

without Fe impregnation and with Fe impregnation in various Fe concentration. The experiments were conducted in Fe concentrations 0.01 M (PILB HDTMA Al, Fe 0.01M), 0.05 M (PILB HDTMA Al, Fe 0.05M), and 0.1 M (PILB HDTMA Al, Fe 0.1M). The results were shown by figure 1.

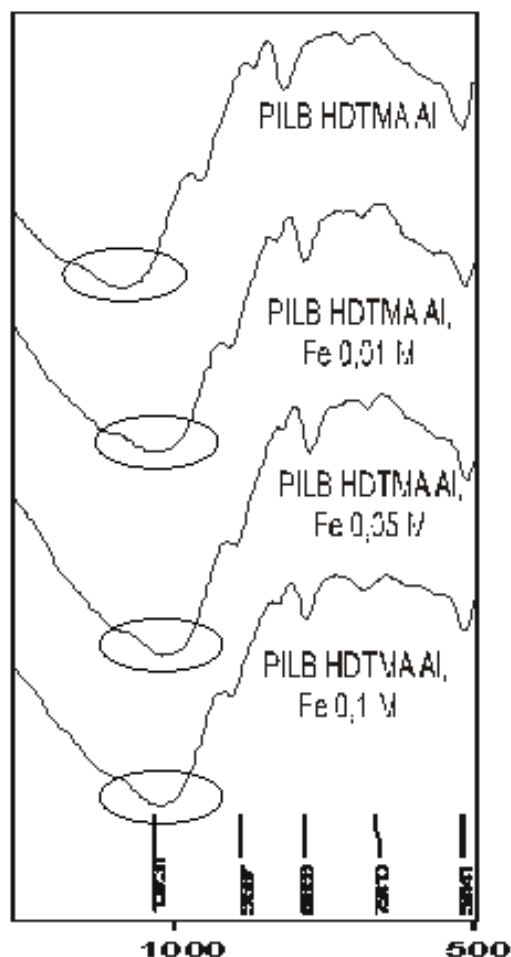


Fig. 1. FTIR Spectra of PILB HDTMA-Al dan Fe impregnated PILB HDTMA-Al in various Fe concentration

Fig. 1 displays the absorption peak in the region $1035\text{--}1050\text{ cm}^{-1}$ which is indicated that Fe has introduced into Al-HDTMA pillared bentonite. It can be observed by the change of the peak in that region. The higher Fe was impregnated into Al pillared bentonite, the lower the intensity of the absorption. It shows that more Fe was introduced into pillared bentonite, the crystallinity of it was decrease (amorph). It was caused that pillar structure in previous material (O-Al-O) becomes disorder because of the formation of Fe oxide.

The absorption peak in the range $526\text{--}650\text{ cm}^{-1}$ of PILB HDTMA Al-Fe in various concentration was appeared different. The absorption peak at 526 cm^{-1} of PILB

HDTMA Al-Fe 0.05 was appeared narrower than that of PILB HDTMA Al-Fe in other concentration. It is proved that the number of double range structure of PILB HDTMA Al-Fe 0.05 M is relatively higher and more stable than that of other pillared bentonite. It suggests that the number of created pores of PILB HDTMA Al-Fe 0.05 M was higher and the pore was bigger than those of other pillared bentonite. The form of the absorption peak in that region was shown smooth in line with increasing Fe concentration. The absorption peak in the region $910 - 920 \text{ cm}^{-1}$ show that the amount of Fe impregnated was higher result in the peak was getting smaller and weaker. It was occurred because the octahedral structure TO_6 (AlO_6) was pushed by Fe which is introduced into bentonite structure. It caused O-Al-O bonding in octahedral structure TO_6 broken down. The damage of octahedral structure results in decreasing of crystallinity of bentonite structure.

Based on the analysis, it can be suggested that increasing number of Fe will cause the damage of bentonite crystalline structure and decreasing the number of pores in bentonite structure. It is validated by the absorption peak of PILB HDTMA Al-Fe in various concentrations at 525.8 cm^{-1} was smoother and narrower than that of PILB HDTMA Al.

X-Ray Diffraction characterization

The success of pillarisation and impregnation process of bentonite by using Al and Fe metal can be characterized by X-ray diffraction. By observing the shifting of the diffraction peak and the change of d_{001} spacing as shown by figure 2, it can be suggested the size of created pores of bentonite.

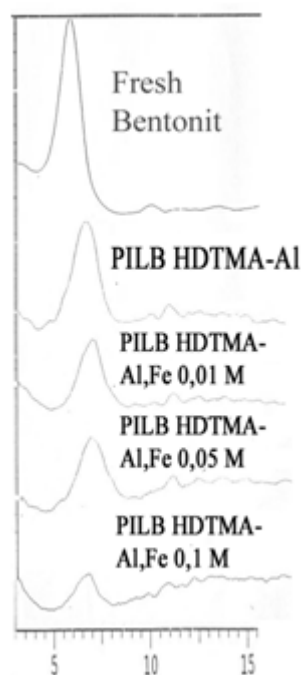


Fig. 2. X-ray diffractogram of PILB HDTMA in various Fe concentrations

The main peak of fresh bentonite is appeared at $2\theta = 5.828^\circ$ and d_{001} spacing = 15.1524. The smaller the value of 2θ , the bigger the layer distance of bentonite is. It suggests the created pillars are higher and the pores are bigger. Figure 2 shows that the peak at 2θ of PILB-HDTMA-Al shifts from 5.828° to 6.25° with the value of d_{001} spacing = 14.1302, it means that interlayer spacing of bentonite is getting smaller. Al metal which is penetrated into interlayer of bentonite will be crowded horizontally and results in creating low pillar.

Figure 2 shows that last three of impregnated bentonite almost has no main peak. It is suggested that most of the crystalline structure of Al pillar bentonite was broken. The higher Fe concentration used as impregnating agent, the lower the main peak in diffractogram. If the amount of Fe in bentonite structure is higher, the existing pillars because of pillarisation process become damage after calcinations. The phenomenon is occurred because Fe is easier to attack oxygen atom of $-O-Al-O$ -group. It is proved by the evidence that the I-count value of main peak is getting lower in line with increasing Fe concentration (Table 1).

Table 1. The effect of Fe concentration to layer distance of PILB HDTMA-Al

Sample	2θ (degree)	d_{001} spacing ($^\circ A$)	I-count	The amount of Fe (mg/gr)	The amount of penetrated Fe (mg/gr)
Fresh bentonit	5.828	15.152	695	-	-
PILB HDTMA-Al	6.25	14.130	295	1.2679	-
PILB HDTMA-Al,Fe 0,01 M	6.38	13.842	194	2.0412	0.7734
PILB HDTMA-Al,Fe 0,05 M	6.283	14.055	192	4.2095	2.9416
PILB HDTMA-Al,Fe 0,1 M	6.213	14.213	95	5.9258	4.6579

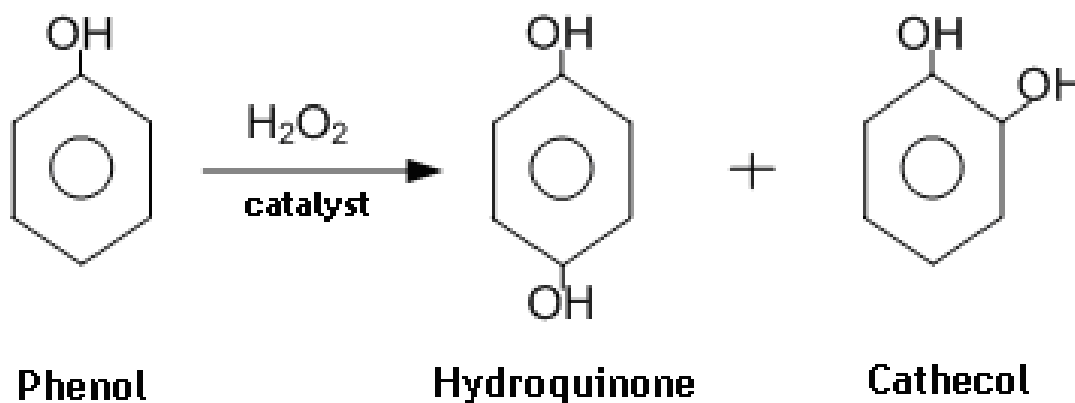
PILB-HDTMA-Al and PILB-HDTMA Al-Fe has lower crystallinity than fresh bentonite. Fresh bentonite has d_{001} spacing = 15.1524 while PILB-HDTMA-Al has d_{001} spacing = 14.1302. It means that the existing pillar has broken and decreases the crystallinity of bentonite. It also occurs on Fe-impregnated bentonite. The higher Fe penetrates into bentonite, the lower the crystallinity of bentonite. It is validated by IR spectrum in the range $900-1100\text{ cm}^{-1}$. The absorption peak is getting weaker in line with increasing Fe concentration.

Catalytic Activity Test and Selectivity for Phenol Hydroxylation Reaction

Catalytic activity test for synthetic catalysts is conducted for phenol hydroxylation reaction. Table 2 shows phenol conversion and selectivity of hydroquinone, cathecol, and benzoquinone. It describes that by using modified bentonite catalyst in the system, % conversion of phenol is higher than those of fresh bentonite and without catalyst system. It happens because the number of active site and suitable pore size is increase, and it gives effect on the amounts of phenol which can be converted into

product. Percentage conversion and reaction selectivity were determined by HPLC analysis

The reaction scheme as shown below:



The catalytic reaction produced hydroquinone (HQ), cathecol (CAT) and benzoquinone (BEQ). The most expected product is HQ but it is produced in small amount. It is shown that the catalyst has not been selective enough to produce the expected product. The system also produced isomer product (CAT) in high amount and very small amount of benzoquinone. Phenol hydroxylation has been known to proceed via redox mechanism involving $\text{Fe}^{3+}/\text{Fe}^{2+}$ redox pair. It also required generation of $\text{OH}\cdot$ radicals by decomposition of H_2O_2 . Transition metal ion was needed to initiate the decomposition of H_2O_2 [17,18]. Because of the fresh bentonite was devoid of any transition metal ions, it was inactive in hydroxylation. Table 2 shows that transition metal ion Fe was successfully loaded in catalyst. Fe^{2+} might coordinate to H_2O_2 strongly enough to decompose it. However, it seems that surface area also plays the important role in the catalytic activity for phenol hydroxylation. It can be seen in table 2 that the intercalation samples (PILB HDTMA-Al) which has lower surface area values than that of impregnated bentonite (PILB HDTMA Al, Fe), was less active for phenol hydroxylation.

Table 2 Hydroxylation of phenol to hydroquinone and cathecol

Catalyst	Conversion (%)	Selectivity (%)		
		hydroquinone	cathecol	benzoquinone
Without catalyst	1.2	49.1	50.3	0.6
Fresh bentonite	4.4	47.0	52.4	0.6
PILB HDTMA-Al	58.8	8.4	91.6	0.0
PILB HDTMA-Al,Fe 0.01 M	72.8	23.9	74.8	1.3
PILB HDTMA-Al,Fe 0.05 M	73.8	27.6	71.6	0.8
PILB HDTMA-Al,Fe 0.1 M	69.7	27.5	72.1	0.4

Reaction condition: Catalyst/Phenol/ H_2O_2 = 1/10/10

The results also show that the concentration of impregnation of Fe into modified bentonite plays the important role in the catalytic activity for phenol hydroxylation. Table 2 indicates that adjustment of Fe concentration causes variation of phenol conversion. Table 2 also depicts that the most excellent Fe concentration for impregnation of modified bentonite is 0.05M. This is supported by the results that phenol conversion is as high as 73.8%, and the selectivity of HQ reaches 27.6 %, and on contrary benzoquinone selectivity is very low. This result also states that pore size of Fe pillared bentonite has not been appropriate for HQ molecule but it is suitable for CAT molecule. HDTMA is expected to increase pore size and be able to support two bentonite layers before penetrating pillaring agent, but the experiment did not get optimal condition of material. It seems that the calcinations process was not successful to remove the organic surfactant molecules. Hence it blocked the porosity.

CONCLUSION

The intercalation of bentonite using HDTMA and followed by impregnation with Fe is successfully presented. However, method for preparing the catalytic material with large pores, namely mesopores, is still lacking. This paper showed that the pillaring bentonites have good potential to catalyze phenol hydroxylation reaction. The best catalytic performance to convert phenol and produce hydroquinone by phenol hydroxylation reaction is on PILB HDTMA-Al,Fe 0.05 M.

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