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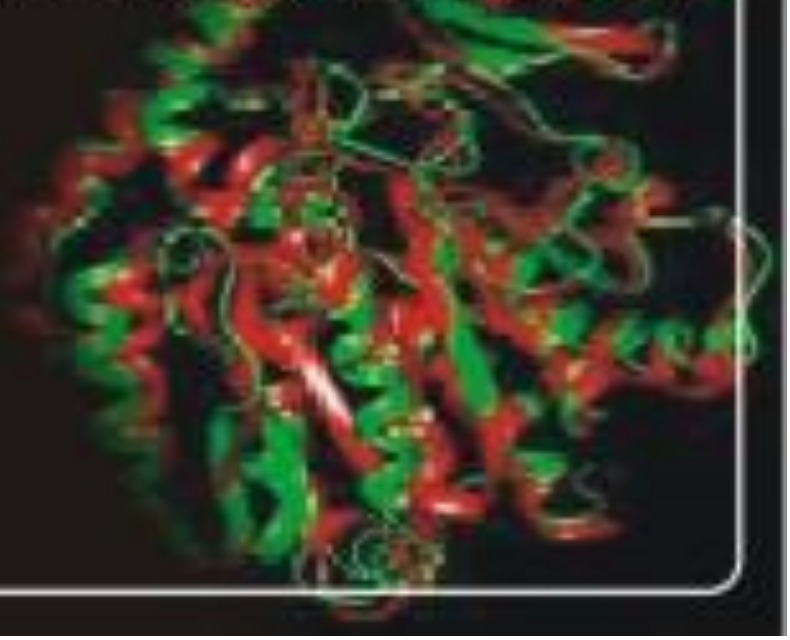

Journal of Chemical and Pharmaceutical Research (ISSN : 0975-7384)

Volume 6, Issue 5 2014: Journal of Chemical and Pharmaceutical Research

Pan Hua and Li Dan
Page No: 621-625

Economic development under the constraints of environmental policy: scenario analysis based on the endogenous growth model (http://www.jocpr.com/articles/economic-development-under-the-constraints-of-environmental-policy-scenario-analysis-based-on-the-endogenous-growth-mode.pdf)
Xin Ma*, Fu-xiao-xuan Liang, Wen-bin Wang and Chun-li Liu
Page No: 1806-1813

Estimation of reducing sugar by acid hydrolysis of black grape (Vitis vinifera L.) peels by standard methods. (http://www.jocpr.com/articles/estimation-of-reducing-sugar-by-acid-hydrolysis-of-black-grape-vitis-vinifera-l-peels-by-standard-methods.pdf)
S. Chandraju, C. S. Chidan Kumar and R. Venkatesh
Page No: 862-866

Research on the ultra-weak luminescence of maize seeds (http://www.jocpr.com/articles/research-on-the-ultraweak-luminescence-of-maize-seeds.pdf)
Feng Wang*1,2, Shanshan Duan2, YitaoLiang1,2 and Weiya Shi1,2
Page No: 42-46

Research of inhibition mechanism of morin on tumor cells aminopeptidase N (http://www.jocpr.com/articles/research-of-inhibition-mechanism-of-morin-on-tumor-cells-aminopeptidase-n.pdf)
Wenjuan Luo and Su Nan
Page No: 1510-1516

Comparison of sterilization reaction rate for Bacillus subtilis and Escherichia coli using UV-visible spectroscopy (http://www.jocpr.com/articles/comparison-of-sterilization-reaction-rate-for-bacillus-subtilis-and-escherichia-coli-using-uvvisible-spectroscopy.pdf)
Priyanka Verma, Shruti Chatterjee, Mayank Choudhary, Ramanathan K. and Shanthi V.
Page No: 1095-1100

Research and design on the remote internet video training system (http://www.jocpr.com/articles/research-and-design-on-the-remote-internet-video-training-system.pdf)
Yong Liu
Page No: 2064-2068

The design of E-commerce integrity evaluation system based on Logit analysis (http://www.jocpr.com/articles/the-design-of-ecommerce-integrity-evaluation-system-based-on-logit-analysis.pdf)
Zheng Dachuan* and Huang Zhen
Page No: 1814-1817

Practical Model for Predicting Compressive Strength of Aerated Concrete Based on Its Pore Structure Distribution (http://www.jocpr.com/articles/practical-model-for-predicting-compressive-strength-of-aerated-concretebased-on-its-pore-structure-distribution.pdf)
Chen, Tao Sun*, Kun Tian, Shengyin Zhou, Ge Yan
Page No: 1431-1436

The calculation method of small-scale water injection multiple in water drive reservoirs (http://www.jocpr.com/articles/the-calculation-method-of-smallscale-water-injection-multiple-in-water-drive-reservoirs.pdf)
Cui Chuanzhi, Li Kaikai, Guo Jincheng, Cao Gang and Zhang Chuanbao
Page No: 204-209

Study on empoasca vitis gothe egg incubation by non-thermal effect model (http://www.jocpr.com/articles/study-on-empoasca-vitis-gothe-egg-incubation-by-nonthermal-effect-model.pdf)
Chun-jiang Shuai and Rui-mei LI
Page No: 47-53

Investigation of CD4+ T cell numbers in HIV-infected patients among smokers and non-smokers in Thailand. (http://www.jocpr.com/articles/investigation-of-cd4-t-cell-numbers-in-hivinfected-patients-among-smokers-and-nonsmokers-in-thailand.pdf)
Sanguansittianan S, Martkamchan S., Nooroon N.,and Ammaranond P
Page No: 867-871

First-principles study of the (n, m) chiral ZnO nanotubes (http://www.jocpr.com/articles/firstprinciples-study-of-the-n-m-chiral-zno-nanotubes.pdf)
Fuchun Zhang, Hongwei Cui, Xingxiang Ruan and Weihu Zhang
Page No: 2069-2072

Conventional and greener approach for the synthesis of some pharmacologically active derivatives of thiazolidines substituted with indolo [2,3-b]quinoxalines (http://www.jocpr.com/articles/conventional-and-greener-approach-for-the-synthesis-of-some-pharmacologically-active-derivatives-of-thiazolidines-substi.pdf)
Krishnakant T. Waghmode
Page No: 1101-1105

Hydrophobic modification for nanoscale cross-linked polymer microspheres (http://www.jocpr.com/articles/hydrophobic-modification-for-nanoscale-crosslinked-polymer-microspheres.pdf)
Hailing Chen, Xiaoyu Zheng, Qinzhe Jiang, Qianqian Wei and Xianjie Li
Page No: 59-65

Numerical simulation of EVA in thermal power plants based on system dynamics (http://www.jocpr.com/articles/numerical-simulation-of-eva-in-thermal-power-plants-based-on-system-dynamics.pdf)
Shuliang Liu and Qianyun Liu
Page No: 368-373

Research on small and medium enterprises financing mode based on supply chain finance (http://www.jocpr.com/articles/research-on-small-and-medium-enterprises-financing-mode-based-on-supply-chain-finance.pdf)
[illegible]q, Hong Liu* and Xiaoxiao Qi
[illegible] 1818-1824

Dynamic analysis of cardiovascular drugs data (http://www.jocpr.com/articles/dynamic-analysis-of-cardiovascular-drugs-data.pdf)
Humei Yang and SunShi
Page No: 1517-1520

Identification of glucosamine hydrochloride diclofenac sodium interaction products by chromatography-mass spectrometry. (http://www.jocpr.com/articles/identification-of-glucosamine-hydrochloride-diclofenac-sodium-interaction-products-by-chromatographymass-spectrometry.pdf)
A. A. Sichkar
Page No: 761-767

Ore-controlling structure of gold deposit in southeast Guizhou (http://www.jocpr.com/articles/orecontrolling-structure-of-gold-deposit-in-southeast-guizhou.pdf)
Yi Chen
Page No: 2073-2077

The feasibility analysis and path selection of China-ASEAN regional monetary cooperation (http://www.jocpr.com/articles/the-feasibility-analysis-and-path-selection-of-chinaasean-regional-monetary-cooperation.pdf)
Wenli Ding, Shuai Yuan* and Donghui Yang
Page No: 1825-1829

Extraction of short-chain organic acids using imidazolium-based ionic liquids from aqueous media (http://www.jocpr.com/articles/extraction-of-shortchain-organic-acids-using-imidazoliumbased-ionic-liquids-from-aqueous-media.pdf)
Qianzhu Li, Xinglin Jiang, Huibin Zou, Zhifeng Cao, Haibo Zhang and Mo Xian
Page No: 374-381

Color license plate localization algorithm research based on moiré principle analysis (http://www.jocpr.com/articles/color-license-plate-localization-algorithm-research-based-on-moir-principle-analysis.pdf)
Shanhong Zhu and Pei Tang
Page No: 706-709

Synthesis of some new N-saccharin derivatives of possible biological activity. (http://www.jocpr.com/articles/synthesis-of-some-new-nsaccharin-derivatives-of-possible-biological-activity.pdf)
Ahmed Wahed Naser and Aseel Farhan Abdullah
Page No: 872-879

E-Learning system architecture based on Private Cloud for university (http://www.jocpr.com/articles/elearning-system-architecture-based-on-private-cloud-for-university.pdf)
Wang Shunye, Liu Dayong and Zhang Zijuan
Page No: 492-498

Research of computer electromagnetic information leakage based on three layers of protection model (http://www.jocpr.com/articles/research-of-computer-electromagnetic-information-leakage-based-on-three-layers-of-protection-model.pdf)
Yuanhui Yu and Ying Deng
Page No: 210-217

Analysis of effects of impeller inlet width on the performance of centrifugal pump (http://www.jocpr.com/articles/analysis-of-effects-of-impeller-inlet-width-on-the-performance-of-centrifugal-pump.pdf)
Qing Zhang, Hai Zhou*, Qingpeng Gao and Zhixiang Cui
Page No: 2078-2081

Physico-chemical and microbial analysis of certain water sources and industrial waste water samples in Chandrapur District, Maharastra, India (http://www.jocpr.com/articles/physicochemical-and-microbial-analysis-of-certain-water-sources-and-industrial-waste-water-samples-in-chandrapur-distric.pdf)
Pratiksha Tambekar, R. J. Batra and R. G. Weginwar
Page No: 710-714

Methods for transferring gene into silkworm and expression of hGM-CSF in silk glands of transgenic silkworm (http://www.jocpr.com/articles/methods-for-transferring-gene-into-silkworm-and-expression-of-hgmcsf-in-silk-glands-of-transgenic-silkworm.pdf)
Zhen Li, Guangli Cao, Renyu Xue and Chengliang Gong
Page No: 118-129

Empirical analysis for the impact of RMB real effective exchange rate on foreign direct investment in China (http://www.jocpr.com/articles/empirical-analysis-for-the-impact-of-rmb-real-effective-exchange-rate-onforeign-direct-investment-in-china.pdf)
Wenrong Pan* and Yu Song
Page No: 1830-1836

Analysis of the components of pumpkin seed oil in suppositories and the possibility of its use in pharmaceuticals (http://www.jocpr.com/articles/analysis-of-the-components-of-pumpkin-seed-oil-in-suppositories-and-the-possibility-of-its-use-in-pharmaceuticals.pdf)
Vorobyova O. A., Bolshakova A. E, Pegova R. A, Kol'chik O., Klabukova I. N., Krasilnikova E. V.c and Melnikova N. B.
Page No: 1106-1116

The evolutionary game study on the privacy and interest of users and websites in the social network environment (http://www.jocpr.com/articles/the-evolutionary-game-study-on-the-privacy-and-interest-of-users-and-websites-in-the-social-network-environment.pdf)
Xia Chen, Tingjie Lu, Longfei Guo and Shuyue Lou
Page No: 1521-1528

S on Morphogenesis of Creative Industry Based on ECHO Model--Example of Beijing 798
ww.jocpr.com/articles/study-on-morphogenesis-of-creative-industry-based-on-echo-modelexample-of-98.pdf)

Xiao Yun
Page No: 499-511

Wireless Sensor Network Factor Information Control Based on Genetic Algorithm (http://www.jocpr.com/articles/wireless-sensor-network-factor-information-control-based-on-geneticalgorithm.pdf)
Sun Zeyu
Page No: 1437-1445

Electrochemical determination and comparison of ascorbic acid in freshly prepared and bottled fruit juices: A cyclic voltammetric study. (http://www.jocpr.com/articles/electrochemical-determination-and-comparison-of-ascorbic-acid-in-freshly-prepared-and-bottled-fruit-juices-a-cyclic-volt.pdf)
Abraha Tadese, P. A. Subramanian, Amaha Woldu and Rishi Pal
Page No: 880-888

High speed micromouse servo controller based on DSP and FPGA (http://www.jocpr.com/articles/high-speed-micromouse-servo-controller-based-on-dsp-and-fpga.pdf)
Haoming Zhang, Yinghai Wang and Peh lian Soon
Page No: 218-225

Retailer's optimal decision model when marketing cost affected by demand (http://www.jocpr.com/articles/retailers-optimal-decision-model-when-marketing-cost-affected-by-demand.pdf)
Fu-chang Li
Page No: 1837-1841

A System Dynamics Based Simulation Model for Reliability Evaluation of Large Scale Information Systems (http://www.jocpr.com/articles/a-system-dynamics-based-simulation-model-for-reliability-evaluation-of-large-scale-information-systems.pdf)
Peng Min-jing and Li Bo
Page No: 626-631

The triple-parameters stochastic dynamics of ecological tourism system (http://www.jocpr.com/articles/the-tripleparameters-stochastic-dynamics-of-ecological-tourism-system.pdf)
Shouwen Wen and Dongping Wei
Page No: 715-720

Effect of different immobilization techniques on α- amylase (http://www.jocpr.com/articles/effect-of-different-immobilization-techniques-on--amylase.pdf)
N. Uma Maheswari* and S. Indra priyadharshini
Page No: 768-774

Hepatoprotective effects of Coleus forskohlii (wild.) Briq. against carbon tetrachloride-induced hepatotoxicity in (http://www.jocpr.com/articles/hepatoprotective-effects-of-coleus-forskohlii-wild-briq-against-carbon-ideinduced-hepatotoxicity-in-mice.pdf)
, Cong Li, Li-tao Yi, Lian-jin Weng and Yuan-yuan Han
Page No: 130-135

The application of three-level filtration system and chlorine dioxide disinfection on hospital sewage treatment (http://www.jocpr.com/articles/the-application-of-threelevel-filtration-system-and-chlorine-dioxide-disinfection-on-hospital-sewage-treatment.pdf)
Yu Bo, Zhou Ying, Huang Zhengwen and Zeng Yonggang
Page No: 382-387

An evaluation index system of computer education quality based on analytic hierarchy process model (http://www.jocpr.com/articles/an-evaluation-index-system-of-computer-education-quality-based-on-analytic-hierarchy-process-model.pdf)
Dan Wei and Xin-qiang Zhao
Page No: 632-635

Synthesis of 3-alkylthio(sulfo)-1,2,4-triazoles, containing methoxyphenyl substituents at C5 atoms, their antipyretic activity, propensity to adsorption and acute toxicity (http://www.jocpr.com/articles/synthesis-of-3alkylthiosulfo124triazoles-containing-methoxyphenyl-substituents-at-c5-atoms-their-antipyretic-activity-pr.pdf)
Y. G. Samelyuk and A. G. Kaplaushenko
Page No: 1117-1121

Removal of ammonia from polluted waters using new bio-sorbents. (http://www.jocpr.com/articles/removal-of-ammonia-from-polluted-waters-using-new-biosorbents.pdf)
K. Prameela Rani and K. Ravindhranath
Page No: 889-900

An empirical analysis of the relation of U.S. dollar exchange rate and American trade balance (http://www.jocpr.com/articles/an-empirical-analysis-of-the-relation-of-us-dollar-exchange-rate-and-american-trade-balance.pdf)
Lin Wen
Page No: 1842-1847

Analysis and empirical study on influence factors of enterprise microblog marketing effect (http://www.jocpr.com/articles/analysis-and-empirical-study-on-influence-factors-of-enterprise-microblog-marketing-effect.pdf)
Xia Chen, Tingjie Lu, Longfei Guo and Xin Li
Page No: 1529-1535

Analyzing the Temperature Field of Combined Plasma Arc Based on Image Pre-processing and Spectral Diagnostics of Plasma Arc (http://www.jocpr.com/articles/analyzing-the-temperature-field-of-combined-plasma-arc-based-on-image-preprocessing-and-spectral-diagnostics-of-plasma-a.pdf)
Dong Xiaojuan, Wei Xiuting, Yin Zhanmin and Meng Jianbing
Page No: 512-519

Chemical constituents of the aerial parts of Cynanchum chinense R. Br. (http://www.jocpr.com/articles/chemical-[illegible]ents-of-the-aerial-parts-of-cynanchum-chinense-r-br.pdf)
[illegible]ng, Xiang Cheng, Zhang Mi, Li Peng and Li Bao-cai
[illegible]: 990-995

Water Environments Monitoring System Based on WSN (http://www.jocpr.com/articles/water-environments-monitoring-system-based-on-wsn.pdf)
Hong Juan Wei
Page No: 636-640

An improved fuzzy evaluation method for heavy metal pollution (http://www.jocpr.com/articles/an-improved-fuzzy-evaluation-method-for-heavy-metal-pollution.pdf)
Juli Deng and Guorong Chen
Page No: 1-11

The research on marine pharmaceutical innovation talent inZhejiang Province
Chen Hongxia , Yang Hongtao and Liu Hongzhen
Page No: 226-231

Research on the sampling methods of transportation price of highway general cargo (http://www.jocpr.com/articles/research-on-the-sampling-methods-of-transportation-price-of-highway-general-cargo.pdf)
Jin-wen Cao and Jing-yan Gu
Page No: 1848-1853

Screening of lactic acid bacteria with high activities malolactic enzyme and analysis of indigenous flora in red wine (http://www.jocpr.com/articles/screening-of-lactic-acid-bacteria-with-high-activities-malolactic-enzyme-and-analysis-of-indigenous-flora-in-red-wine.pdf)
Kai Liu , Jianhua Luo , Dawei Gao, Zhiwei Liu, Yanping Liu, Nan Li, Yanhong Bian, Ruiyu Zheng and Xuwu Zhang
Page No: 136-144

Effects of CBL mode to residency training doctor of pediatric dentistry for making diagnosis and treatment plan (http://www.jocpr.com/articles/effects-of-cbl-mode-to-residency-training-doctor-of-pediatric-dentistry-for-making-diagnosis-and-treatment-plan.pdf)
Limin Wei, Jianfeng Ma, Yihuai Pan and Jianfeng Wang
Page No: 393-397

Digital Electrical Resistance Tomography System and itsExperimental Research (http://www.jocpr.com/articles/digital-electrical-resistance-tomography-system-and-itsexperimental-research.pdf)
Lifeng Zhang
Page No: 520-526

Natural indicator as a eco-friendly in acid base titration. (http://www.jocpr.com/articles/natural-indicator-as-a-ecofriendly-in-acid-base-titration.pdf)
Shivaji H. Burungale and Ankush V. Mali
Page No: 901-903

Substantiation of the methods of quality control for a substance with the anticonvulsant action. (http://www.jocpr.com/articles/substantiation-of-the-methods-of-quality-control-for-a-substance-with-the-anticonvulsant-action.pdf)

Natalia Bevz, Victoriya Georgiyants and Vasyl Gryinenko
Page No: 775-779

An image classification algorithm using fuzzy support vector machine (http://www.jocpr.com/articles/an-image-classification-algorithm-using-fuzzy-support-vector-machine.pdf)
Cao Jianfang,Chen Junjie and Chen Lichao
Page No: 1854-1861

A study on the Identification of Factors Affecting the Safe Working State of Coal Miners (http://www.jocpr.com/articles/a-study-on-the-identification-of-factors-affecting-the-safe-working-state-of-coal-miners.pdf)
Weihua Zhang
Page No: 641-646

Rapid measurement of biodiesel conversion rate by near-infrared spectroscopy (http://www.jocpr.com/articles/rapid-measurement-of-biodiesel-conversion-rate-by-nearinfrared-spectroscopy.pdf)
Yucong Liu, Jiangang Lu, Long Xu, Gang Qu, Mingqiao Zhu, Xin Jiang, Jinshui Chen and Jiang Yang
Page No: 66-71

Study on the evaluation of strategic alliance partners among iron and steel enterprises in the perspective of energy-saving services (http://www.jocpr.com/articles/study-on-the-evaluation-of-strategic-alliance-partners-among-iron-and-steel-enterprises-in-the-perspective-of-energysavi.pdf)
Dajun Ye
Page No: 527-535

Corrosion inhibition of carbon steel in 1M HCl solution by Ruta graveolens extract. (http://www.jocpr.com/articles/corrosion-inhibition-of-carbon-steel-in-1m-hcl-solution-by-ruta-graveolens-extract.pdf)
Mahir H. Majeed, Abdul-Wahab A. Sultan and Hussein H. Al-Sahlanee
Page No: 996-1001

Two-sided software platform: Operating strategies under multi-homing users (http://www.jocpr.com/articles/twosided-software-platform-operating-strategies-under-multihoming-users.pdf)
Xia Chen, Yu Li, Tingjie Lu and Longfei Guo
Page No: 1536-1543

Micellar behavior of gemini surfactant butanediyl-1,4-bis(hexadecyl hydroxyethyl methyl ammonium bromide) and its monomeric counterpart on physicochemical properties. (http://www.jocpr.com/articles/micellar-behavior-of-gemini-surfactant-butanediyl14bishexadecyl-hydroxyethyl-methyl-ammonium-bromide-and-its-monomeric-c.pdf)
Borse S. S.and Patil T. J
Page No: 904-911

Analysis on demand factor of regional difference for reverse mortgage-based on survey data in Beijing & Hangzhou (http://www.jocpr.com/articles/analysis-on-demand-factor-of-regional-difference-for-reverse-mortgagebased-on-survey-data-in-beijing--hangzhou.pdf)

Zhou Haizhen
Page No: 232-238

Fast railway freight product structure based on transport efficiency
Yinying Tang and Zhongren Wang
Page No: 1862-1867

Comprehensive colliery safety monitoring system design based on wireless sensor network (http://www.jocpr.com/articles/comprehensive-colliery-safety-monitoring-system-design-based-on-wireless-sensor-network.pdf)
Weihua Zhang
Page No: 647-651

Development and validation of simultaneous estimation of diclofenac potassium, paracetamol and serratiopeptidase by first order derivative UV spectroscopy method in pharmaceutical formulation. (http://www.jocpr.com/articles/development-and-validation-of-simultaneous-estimation-of-diclofenac-potassium-paracetamol-and-serratiopeptidase-by-first.pdf)
Ekta J. Pandya*, Pankaj Kapupara and Ketan V. Shah
Page No: 912-924

The resources monitoring and optimization of scheduling research based on the cloud computing environment (http://www.jocpr.com/articles/the-resources-monitoring-and-optimization-of-scheduling-research-based-on-the-cloud-computing-environment.pdf)
Shengjun Xie
Page No: 536-539

Role of glycine as a '3D-structure' maker in aqueous mixture of protophilic dipolar aprotic dimethylsulfoxide. (http://www.jocpr.com/articles/role-of-glycine-as-a-3dstructure-maker-in-aqueous-mixture-of-protophilic-dipolar-aprotic-dimethylsulfoxide.pdf)
S. Roya, K. Mahalib, S. Mondalc and B. K. Dolui
Page No: 780-790

Coral Reef Lenticular Freshwater Assessment Based on Fuzzy Mathematical Method (http://www.jocpr.com/articles/coral-reef-lenticular-freshwater-assessment-based-on-fuzzy-mathematical-method.pdf)
Yuzheng Lv, Jiafu Yang, Kun Liu, Jie Shi
Page No: 1469-1472

How to define general purpose technologies in the cluster: Based on the view of industrial chain (http://www.jocpr.com/articles/how-to-define-general-purpose-technologies-in-the-cluster-based-on-the-view-of-industrial-chain.pdf)
Zheng Jianzhuang and Jin Yuhan
Page No: 1592-1596

Extraction of Public Library Service Function Transformation and Extension (http://www.jocpr.com/articles/extraction-of-public-library-service-function-transformation-and-extension.pdf)
Qingmin Shi
Page No: 652-654

Fast railway freight product structurebased on transport efficiency (http://www.jocpr.com/articles/fast-railway-freight-product-structurebased-on-transport-efficiency.pdf)
Yinying Tang and Zhongren Wang
Page No: 1862-1867

Study on correlation mechanism of motivation, conduct and performance of industrial alliance from the perspective of technology intermediary (http://www.jocpr.com/articles/study-on-correlation-mechanism-of-motivation-conduct-andperformance-of-industrial-alliance-from-the-perspective-oftechno.pdf)
Zhang Jian, Duan Shan and Huang Chun*
Page No: 1597-1600

Antimicrobial activity of newly synthesized hydroxamic acid of pyrimidine-5-carboxylic acid and its complexes with Cu(II), Ni(II),Co(II) and Zn(II) metal ions. (http://www.jocpr.com/articles/antimicrobial-activity-of-newly-synthesized-hydroxamic-acid-of-pyrimidine5carboxylic-acid-and-its-complexes-with-cuii-ni.pdf)
Bhawani Shankar, Rashmi Tomar, Rakesh Kumar, Madhu Godhara and Vijay Kumar Sharma
Page No: 925-930

The essential oil composition of Haplophyllum laeviusculum C. C.Towns. aerial parts.
Boshra Azadi and Sepideh Khaef
Page No: 1002-1005

A Statistical Study on How Migrant Workers Choose Migration Patterns ——A Case in Shandong Province of China (http://www.jocpr.com/articles/a-statistical-study-on-how-migrant-workers-choose-migration-patterns-a-case-in-shandong-province-of-china.pdf)
Gaojian and Chen Shunzhou
Page No: 540-546

The Application of AugmentedRealityTechnology in Teaching Education (http://www.jocpr.com/articles/the-application-of-augmentedrealitytechnology-in-teaching-education.pdf)
Fan Kang and Cunchen Tang
Page No: 1446-1449

A survey of the phytochemical and antioxidant potential of the fruit extracts of Sarcocephalus latifolius (Smith) bruce (Rubiaceae). (http://www.jocpr.com/articles/a-survey-of-the-phytochemical-and-antioxidant-potential-of-the-fruit-extracts-of-sarcocephalus-latifolius-smith-bruce-ru.pdf)
H. B. Yesufu, I. Z. Khan, F. I. Abdulrahman and Y. Z. Abatch
Page No: 791-795

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Study on relationship between investment in science and technology and economics growth for petrochemical enterprise based grey relational degree analysis (http://www.jocpr.com/articles/study-on-relationship-between-investment-in-science-and-technology-and-economics-growth-for-petrochemical-enterprise-bas.pdf)
Yi Si fei
Page No: 721-726

The empirical research of the comprehensive benefit evaluation on liaoning Shihu wind power project (http://www.jocpr.com/articles/the-empirical-research-of-the-comprehensive-benefit-evaluation-on-liaoning-shihu-wind-power-project.pdf)
Chang-he Jiang and Jing-quan Liu
Page No: 1605-1609

Shot throwing technique biomechanical parameters sensitivity and optimization research (http://www.jocpr.com/articles/shot-throwing-technique-biomechanical-parameters-sensitivity-and-optimization-research.pdf)
Anlong Huang
Page No: 1873-1879

Big data learning resources integration and processing in cloud environments. (http://www.jocpr.com/articles/big-data-learning-resources-integration-and-processing-in-cloud-environments.pdf)
Sun Dapeng
Page No: 936-943

Analysis of the relationship between the ratio of man to livestock and malaria incidence in Shandong province, China (http://www.jocpr.com/articles/analysis-of-the-relationship-between-the-ratio-of-man-to-livestock-and-malaria-incidence-in-shandong-province-china.pdf)
Lijuan Liu, Benguang Zhang, Huaiwei Wang, Xiuxia Guo, Haifang Wang and Maoqing Gong
Page No: 145-149

Cost Control for Small and Medium-sized Enterprises (SMEs) (http://www.jocpr.com/articles/cost-control-for-small-and-mediumsized-enterprises-smes.pdf)
Lei Song
Page No: 409-412

Two-sided matching decision under incomplete score environment (http://www.jocpr.com/articles/twosided-matching-decision-under-incomplete-score-environment.pdf)
Qi Yue and Yuhua Li
Page No: 1610-1614

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Page No: 271-278

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Page No: 150-154

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Page No: 1450-1453

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Page No: 423-426

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Page No: 155-160

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Page No: 947-951

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Page No: 575-584

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Rongmei Chen
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Xiong Delan
Page No: 585-589

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Page No: 249-257

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Yuanyi Ding
Page No: 427-430

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Liuling Song and Songting Lu
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Xiuxia LI
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Page No: 796-800

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Page No: 431-436

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Zhaoxia Huang
Page No: 437-441

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Page No: 12-20

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Jiayong Dou, Yaya Wang and Wei Gao
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Page No: 1022-1028

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Page No: 801-807

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Yang Xiao
Page No: 266-270

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Page No: 1664-1670

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Xin Lei
Page No: 442-448

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Page No: 952-960

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Page No: 808-814

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Gui-lin Yang
Page No: 590-594

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Liu Wen-wei, Duan Wan-chun and Xu Yang-guang
Page No: 1574-1579

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Yu Shasha
Page No: 455-459

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Ying Wang
Page No: 1671-1675

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Page No: 1946-1951

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Page No: 815-822

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Anjali Anand and Meena Sharma
Page No: 961-967

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Wei Hui-Ping, Zhao Mu, Li Yong and Liu Weng-Quan
Page No: 161-165

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Jingpo Yang, Fei Lu and Yuanjie Zhao
Page No: 460-465

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Page No: 1952-1956

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Page No: 1676-1682

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Zhao Wanli, Huang Xin and Qiao Wen tao
Page No: 595-600

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Page No: 1580-1585

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Tang Yong-Zheng
Page No: 823-830

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Zhongjie Xiao
Page No: 665-668

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Jiejia Li, Xiaoyu Sun*, Peng Yang , Liang Zhou and Abdol Ghaffar Ebadi
Page No: 1683-1688

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Page No: 968-974

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Dongya Yang , Jun Gonga and Honggang Wangb
Page No: 83-90

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Page No: 1964-1967

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Page No: 1029-1034

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Xiaoqing Lu, Shuming Guan and Wenyi Zhang
Page No: 466-470

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Hongjuan Wei
Page No: 1477-1480

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Xiuge Zhang, Ye Ren and Qizhou Hu
Page No: 1689-1695

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Lu Zhao Gan and Li Junxia
Page No: 303-310

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Yi-Wei Wang, Jun Du, Xi Du, Yan-sen Cai, Hua Song and Jian-Min Guo
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Page No: 831-835

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Meng Zhang
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Baoyu Ma
Page No: 1416-1419

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Page No: 104-105

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Jitao Pu
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Jinfeng Lin
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Page No: 1059-1065

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Page No: 1769-1773

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Ruihui Mu* and Yulei Ma
Page No: 2024-2027

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Shaohua Tao, Zhanshen Feng and Zhili Zhang
Page No: 186-197

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Hua-li Jian, Guang-jun Song and Mei-de Liao
Page No: 27-33

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Page No: 1774-1777

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Jinhai Lu
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Page No: 617 - 620

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Page No: 340-346

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Page No: 1066-1071

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Li Liu
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Page No: 347-351

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Page No: 2037-2041

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Page No: 1072-1075

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Page No: 1504-1509

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Wang Qiaomin, Yan Zhiyu, Sun Bing, Zhu Xiaomei and Liu hui
Page No: 106-111

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Wang Xiaogang
Page No: 198-203

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Li Lin* and Li Fei
Page No: 1789-1794

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Page No: 2042-2047

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Jianhua Liu, Jianwei Wang and Wenjuan Bu
Page No: 352-359

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Page No: 1076-1088

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Li Xiang* and Yang Lina

Page No: 1795-1800

Polymer monolithic rods microextraction coupled with high performance liquid chromatography for the analysis of trimethoprim, sulfadiazine and sulfamethoxazole in honey samples (http://www.jocpr.com/articles/polymer-monolithic-rods-microextraction-coupled-with-high-performance-liquid-chromatography-for-the-analysis-of-trimetho.pdf)
Zhi-gang Xu*, Lian-guang Wang, Yun-li Chen and Zhi-min Liu
Page No: 54-58

Phytochemical screening and elemental analysis in different plant parts of Uraria picta Desv.: A Dashmul species. (http://www.jocpr.com/articles/phytochemical-screening-and-elemental-analysis-in-different-plant-parts-of-uraria-picta-desv-a-dashmul-species.pdf)
Hari Om Saxena*, Anjana Soni, Naseer Mohammad and Santosh Kumar Choubey
Page No: 756-760

Analysis and design of intelligent group decision support system of comprehensive transportation network layout planning (http://www.jocpr.com/articles/analysis-and-design-of-intelligent-group-decision-support-system-of-comprehensive-transportation-network-layout-planning.pdf)
Qiao Xiong* and Ting Ren
Page No: 2048-2056

Intrapreneurship, human resource management and their interfacial interaction: A study on multi-cases in the mature stage (http://www.jocpr.com/articles/intrapreneurship-human-resource-management-and-their-interfacial-interaction-a-study-on-multicases-in-the-mature-stage.pdf)
Huibin Guo
Page No: 1801-1805

Primary Analysis on Computer Basis Course Reformation (http://www.jocpr.com/articles/primary-analysis-on-computer-basis-course-reformation.pdf)
Dan Liu, Xuejun Wang and Lijuan Zheng
Page No: 1390-1393

Art Design Specialty Evaluation Based on Analytic Hierarchy Process Theory (http://www.jocpr.com/articles/art-design-specialty-evaluation-based-on-analytic-hierarchy-process-theory.pdf)
Xiao Zhao
Page No: 1394-1398

Autonomous Learning Model for College Students under Multimedia Environment (http://www.jocpr.com/articles/autonomous-learning-model-for-college-students-under-multimedia-environment.pdf)
Shanhong Zhu, Cunchen Tang, Hong Pan
Page No: 1399-1402

of shareholding structure on Chinese bio-pharmaceutical listing companies' R & D investment. (www.jocpr.com/articles/impact-of-shareholding-structure-on-chinese-biopharmaceutical-listing-companies-r--d-investment.pdf)

Liu Bin and Wei Changsheng
Page No: 858-861

Anti-proliferative effects of C-phycocyanin on a human leukemic cell line and induction of apoptosis via the PI3K/AKT pathway (http://www.jocpr.com/articles/antiproliferative-effects-of-cphycocyanin-on-a-human-leukemic-cell-line-and-induction-of-apoptosis-via-the-pi3kakt-pathw.pdf)
Phakorn Tantirapan1 and Yaneenart Suwanwong
Page No: 1295-1301

Solitary pulmonary nodules detection based on dot-filter and region growing (http://www.jocpr.com/articles/solitary-pulmonary-nodules-detection-based-on-dotfilter-and-region-growing.pdf)
Liwei Liu1, Xin Wang, Yang Li and Liping Wang
Page No: 1176-1181

Primary processing technology on gluten soy sauce with low-salt and solid-state fermentation (http://www.jocpr.com/articles/primary-processing-technology-on-gluten-soy-sauce-with-lowsalt-and-solidstate-fermentation.pdf)
Changlu Ma, Jianfeng Sun, Hongxia Luo, Shurong Li and Wentong Xue
Page No: 1182-1186

Study of separation and flocculation characteristics of a bioflocculant MBF B31 (http://www.jocpr.com/articles/study-of-separation-and-flocculation-characteristics-of-a-bioflocculant-mbf-b31.pdf)
Hongxia Cui and Sha Zhang
Page No: 1187-1192

In vitro antioxidant activity and phytochemical analysis of ethanolic extract of Lentinus connatus (http://www.jocpr.com/articles/in-vitro-antioxidant-activity-and-phytochemical-analysis-of-ethanolic-extract-of-lentinus-connatus.pdf)
L. Sushila Devi, Mayukh Chakraborty, Chandrima Chakraborty, S. K. Borthakur, and N. Irabanta Singh
Page No: 1302-1309

Simultaneous quantification of levosulpiride and rabeprazole in tablet dosage form by validated normal phase high performance thin layer chromatographic method (http://www.jocpr.com/articles/simultaneous-quantification-of-levosulpiride-and-rabeprazole-in-tablet-dosage-form-by-validated-normal-phase-high-perfor.pdf)
Pravin Devidas Pawar, Sachin Eknath Potawale, Kakasaheb Ramoo Mahadik and Satish Yashwant Gabhe*
Page No: 1193-1199

Nano α-Al2O3 for removal of Hg(II) from water: Adsorption and desorption studies (http://www.jocpr.com/articles/nano-al2o3-for-removal-of-hgii-from-water-adsorption-and-desorption-studies.pdf)
Qing-Zhou Zhai
Page No: 1310-1317

performance evaluation of digital resources in college library based on fuzzy analytic hierarchy process (http://www.jocpr.com/articles/study-on-performance-evaluation-of-digital-resources-in-college-library-based-on-fuzzy-analytic-hierarchy-process.pdf)

Chen Shi Qiang
Page No: 1122-1127

Chemical constituents of the bark of Aleurites moluccana L. Willd. (http://www.jocpr.com/articles/chemical-constituents-of-the-bark-of-aleurites-moluccana-l-willd.pdf)
Agnes B. Alimboyoguen, Kathlia A. De Castro-Cruz, Chien-Chang Shen,Wen-Tai Li, and Consolacion Y. Ragasa
Page No: 1318-1320

Evaluation study on training of 100m sprint athletes based on improved D-S evidence theory (http://www.jocpr.com/articles/evaluation-study-on-training-of-100m-sprint-athletes-based-on-improved-ds-evidence-theory.pdf)
Agnes B. Alimboyoguen, Kathlia A. De Castro-Cruz, Chien-Chang Shen,Wen-Tai Li, and Consolacion Y. Ragasa
Page No: :1321-1326

Determination of vitamin C and nicotinic acid content in black bean by capillary zone electrophoresis (http://www.jocpr.com/articles/determination-of-vitamin-c-and-nicotinic-acid-content-in-black-bean-by-capillary-zone-electrophoresis.pdf)
Hai-Xing Liu Yang Xu, Hong Li, Yong-Mei Yan, Kai-Qi-Ye, Lin--Tong Wang, and Hui Cao
Page No: 1327-1330

Psychological stress leads to hepatic iron accumulation and disturbs iron homeostasis (http://www.jocpr.com/articles/psychological-stress-leads-to-hepatic-iron-accumulation-and-disturbs-iron-homeostasis.pdf)
Min Zhaoa,b, Li-Juan Liuc, Xin-Guo Lid,Wan-Yin Wang, Lei Wangb, Long Mab, Hui, Shenb Zhi-Ming Zhua and Min Lib
Page No: 1128-1134

Study on a structure of 2,6-diaminopyridine vanadate hydrate, C20H38N12O15V4 (http://www.jocpr.com/articles/study-on-a-structure-of-26diaminopyridine-vanadate-hydrate-c20h38n12o15v4.pdf)
Hai-Xing Liu Yang Xu, Hong Li, Yong-Mei Yan, Kai-Qi-Ye, Lin--Tong Wang, and Hui Cao
Page No: 1331-1335

Transformation of green fluorescent protein gene in Valsa mali var.mali using Agrobacterium tumefaciens
Qingming Zhang and Caixia Wang
Page No: 1200-1203

Insecticidal activity of substituted 1,3-diynes (http://www.jocpr.com/articles/insecticidal-activity-of-substituted-13diynes.pdf)
D. Munirajasekhar
Page No: 1204-1207

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bi-diagonal scheme for computation of discontinuous flow problems
ww.jocpr.com/articles/implicit-bidiagonal-scheme-for-computation-of-discontinuous-flow-problems.pdf)
Yuling Liu, K. Yang, W. L. Wei and X. F. Yang

Page No: 1336-1341

Study on influence factor of braking torque in liquid-cooled eddy current retarder with a structure of two salient poles (http://www.jocpr.com/articles/study-on-influence-factor-of-braking-torque-in-liquidcooled-eddy-current-retarder-with-a-structure-of-two-salient-poles.pdf)
Zhang Long Xi, Li De Sheng and Yin Wang Lei
Page No: 1135-1141

The study of thione-thiol tautomerism of 4-amino-5-(4-nitrophenyl)- 2,4-dihydro-3H-1,2,4-triazole-3-thione by HPLC-MS method (http://www.jocpr.com/articles/the-study-of-thionethiol-tautomerism-of-4amino54nitrophenyl-24dihydro3h124triazole3thione-by-hplcms-method.pdf)
B. A. Varynskyi, M. A. Scherback, A. G. Kaplaushenko and I. A. Yurchenko
Page No: 1342-1350

Fermentative production of ribavirin by overexpressing purine nucleoside phosphorylase in Bacillus (http://www.jocpr.com/articles/fermentative-production-of-ribavirin-by-overexpressing-purine-nucleoside-phosphorylase-in-bacillus.pdf)
Yuechao Ma, Shuyao Yang, Li Liu, Qingyang Xu, Chenglin Zhang, Ning Chen and Xixian Xie
Page No: 1377-1384

A novel pricing model to new financial products based on the game theory (http://www.jocpr.com/articles/a-novel-pricing-model-to-new-financial-products-based-on-the-game-theory.pdf)
Jie Pan
Page No: 1142-1147

Antibacterial and mosquito larvicidal activity of Ficus casiguranensis and Ficus camarinensis leaves (http://www.jocpr.com/articles/antibacterial-and-mosquito-larvicidal-activity-of-ficus-casiguranensis-and-ficus-camarinensis-leaves.pdf)
John Benson D. Choa, Aleth L. Dacanay, and Mario A. Tan
Page No: 1351-1354

A new and rapid analytical method development & validation for simultaneous estimation of hydrochlorothiazide, amlodipine & olmesartan in tablet dosage form by using RP-HPLC (http://www.jocpr.com/articles/a-new-and-rapid-analytical-method-development--validation-for-simultaneous-estimation-of-hydrochlorothiazide-amlodipine.pdf)
S. Ashutosh Kumar, Manidipa Debnath, J. V. L. N. Seshagiri Rao and D. Gowri Sankar
Page No: 1208-1213

Effect of complexation conditions on microcapsulation of B. bifidum BB01 in xanthan–chitosan polyelectrolyte complex gels (http://www.jocpr.com/articles/effect-of-complexation-conditions-on-microcapsulation-of-b-bifidum-bb01-in-xanthanchitosan-polyelectrolyte-complex-gels.pdf)
He Chen, Yajuan Song, Nina Liu, Hongchang Wan, Na Liao and Guowei Shu
Page No: 1355-1360

Synthesis and properties of new metal complexes involving unsymmetrical tetrathiafulvalenes (http://www.jocpr.com/articles/synthesis-and-properties-of-new-metal-complexes-involving-unsymmetrical-tetrathiafulvalenes.pdf)
Tahar Abbaz,Amel Bendjeddou, Nadia Nait Said, Rachida Khammar, Djamel Bouchouk, Souheila Bouacherine, Noura Sedira, Soulef Maache, Abdelkrim Gouasmia, Rabah Rehamnia, Mabrouka Dekhici and Didier Villemin
Page No: 1385-1389

Phytoremediation of aluminium and lead using Raphanus sativus, Vigna radiata and Cicer arietinum (http://www.jocpr.com/articles/phytoremediation-of-aluminium-and-lead-using-raphanus-sativus-vigna-radiata-and-cicer-arietinum.pdf)
James Raj and L. Jeyanthi Rebecca
Page No: 1148-1152

RP-HPLC method development and validation for the determination of ciprofloxacin from marketed tablet dosage forms (http://www.jocpr.com/articles/rphplc-method-development-and-validation-for-the-determination-of-ciprofloxacin-from-marketed-tablet-dosage-forms.pdf)
Ashok Kumar Bera, Amit Kumar De and Biswajit Pal
Page No: 1214-1218

Development of virtual instrument motor experiment teaching system based on LabVIEW (http://www.jocpr.com/articles/development-of-virtual-instrument-motor-experiment-teaching-system-based-on-labview.pdf)
Zongjian Huang and Xingju Wang
Page No: 1361-1368

Synthesis, biological activity evaluation and QSAR studies of novel 3-(aminooxalyl-amino)-and 3-(carbamoyl-propionylamino)-2 phenylamino-benzoic acid derivatives (http://www.jocpr.com/articles/synthesis-biological-activity-evaluation-and-qsar-studies-of-novel-3aminooxalylaminoand-3carbamoylpropionylamino2-phenyl.pdf)
M. M. Suleiman, S. G. Isaev, O.V. Klenina and V. V. Ogurtsov
Page No: 1219-1235

Isolation and partial purification of lipase and protease from marine algae (http://www.jocpr.com/articles/isolation-and-partial-purification-of-lipase-and-protease-from-marine-algae.pdf)
Shilpak Dilip Bele, Swapnil Suresh Patil, S. Sharmila and L. Jeyanthi Rebecca
Page No: 1153-1156

Isolation and screening of protease producing bacteria from marine waste (http://www.jocpr.com/articles/isolation-and-screening-of-protease-producing-bacteria-from-marine-waste.pdf)
S. Sneha, Merina Paul Das* and L. Jeyanthi Rebecca
Page No: 1157-1159

Research on the novel network pattern of enterprises under WIFI environment (http://www.jocpr.com/articles/research-on-the-novel-network-pattern-of-enterprises-under-wifi-environment.pdf)
P
1160-1163

The research on methods of storing events detected in memory and external memory (http://www.jocpr.com/articles/the-research-on-methods-of-storing-events-detected-in-memory-and-external-memory.pdf)
Ya Wang and Yange Chen
Page No: 1169-1175

Screening of a newly marine bacteria producing alkaline protease from Qinhuangdao sea area and its characterization of alkaline protease (http://www.jocpr.com/articles/screening-of-a-newly-marine-bacteria-producing-alkaline-protease-from-qinhuangdao-sea-area-and-its-characterization-of-a.pdf)
Hongxia Cui1, and Liping Wang
Page No: 1236-1242

Evaluation of anti-cholesteremic and anti-lipidemic activity of seed extract of Achyranthes aspera in diet induced hyperlipidemia model in rats (http://www.jocpr.com/articles/evaluation-of-anticholesteremic-and-antilipidemic-activity-of-seed-extract-of-achyranthes-aspera-in-diet-induced-hyperli.pdf)
Kanakam Vijayabhaskar, Vurugonda Ramadevi, Kalakota Chaitanyaprasad, Sadhiram Rajeshkumar, Divya Sripada and G. Himabindu
Page No: 1247-1250

Chinese universities sports teachers' cultural consciousness in perspective of globalization (http://www.jocpr.com/articles/chinese-universities-sports-teachers-cultural-consciousness-in-perspective-of-globalization.pdf)
Zhanqiang Teng
Page No: 1251-1254

Energy efficiency and investments in low-carbon economy: The impact of carbon finance on sustainability development (http://www.jocpr.com/articles/energy-efficiency-and-investments-in-lowcarbon-economy-the-impact-of-carbon-finance-on-sustainability-development.pdf)
Li Zhou
Page No: 1255-1261

Empirical analysis of PE in college physical education based on student satisfactions (http://www.jocpr.com/articles/empirical-analysis-of-pe-in-college-physical-education-based-on-student-satisfactions.pdf)
Luo Y, Zhang W. B. and Zheng J. Y.
Page No: 1262-1265

Gap element method and its application on force analysis of tubing strings (http://www.jocpr.com/articles/gap-element-method-and-its-application-on-force-analysis-of-tubing-strings.pdf)
Liu Weikai, Song Mingxing, XU Ziyi and Zhang Xuehong
Page No: 1266-1273

The application of hollow fiber-liquid phase microextraction based on green chemistry to analyze carcinogenic nitrosamines in food samples (http://www.jocpr.com/articles/the-application-of-hollow-fiberliquid-phase-microextraction-based-on-green-chemistry-to-analyze-carcinogenic-nitrosamine.pdf)
Raharjo*, Usreg Sri Handajani and Miratul Khasanah
Page No: 1274-1280

A new chemistry of 2,3-dimethyl-6,7,8,9-tetrahydrobenzocyclohepten-5-one (http://www.jocpr.com/articles/a-new-chemistry-of-23dimethyl6789tetrahydrobenzocyclohepten5one.pdf)
Srinivasa Reddy Konda, Buchi Reddy Reguri and Mukkanti Kagg
Page No: 1281-1285

Biological activities of extract prepared from Sorbus sibirica fruit (http://www.jocpr.com/articles/biological-activities-of-extract-prepared-from-sorbus-sibirica-fruit.pdf)
Jie Wei, Jia Shi, Jun Gao, Zhiquan Zhou and Jungang Fan
Page No: 1369-1372

Preparation, characterization, and antibacterial properties of mixed ligand complexes of L-aspargine and sulfamethoxazole(antibiotic) with Mn(II), Co(II), Ni(II), Cu(II), Zn(II), Cd(II) and Hg(II) ions (http://www.jocpr.com/articles/preparation-characterization-and-antibacterial-properties-of-mixed-ligand-complexes-of-laspargine-and-sulfamethoxazolean.pdf)
Taghreed H. Al-Noor, RaheemTaher Mahdi and Ahmed H. Ismael
Page No: 1286-1294

Study on a structure of Tris-(4,4'-dimethyl-2,2'-dipyridyl)-Ni iodine hydrate, Ni(C12H12N2)3 ● 2I ● H2O (http://www.jocpr.com/articles/study-on-a-structure-of-tris44dimethyl22dipyridylni-iodine-hydrate-nic12h12n232ih2o.pdf)
Hai-Xing Liu1, Yang Xu, Feng Li, Huan-Mei Guo, Kai-Qi Ye, Lin-Tong Wang1 and Hui Cao
Page No: 1373-1376

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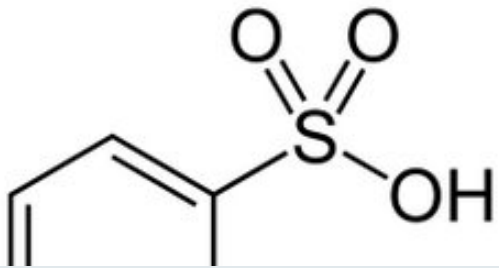

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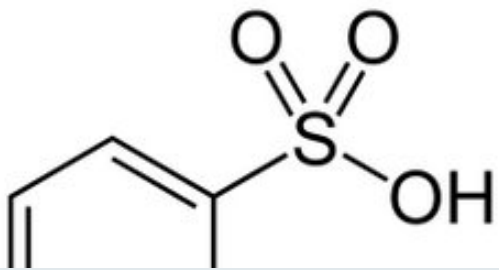

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29

H Index

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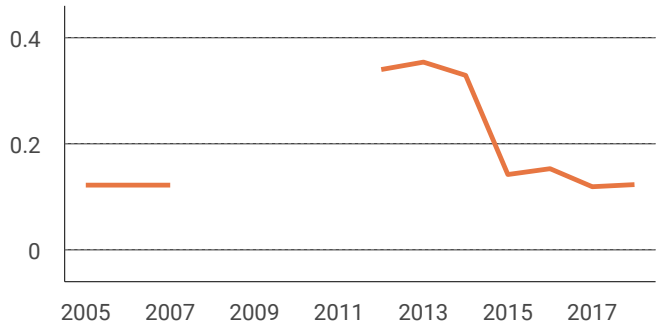

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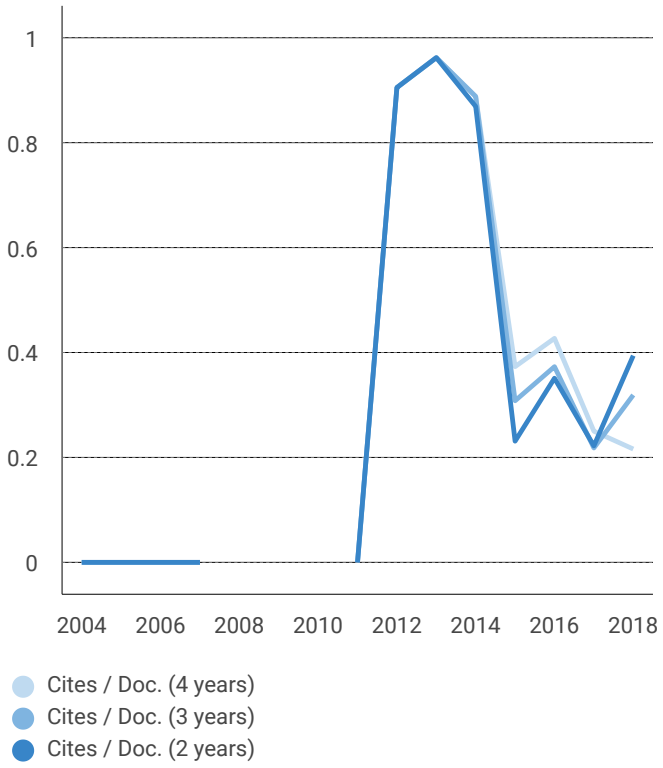

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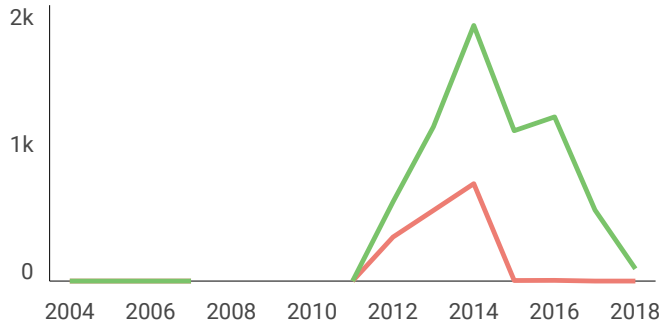

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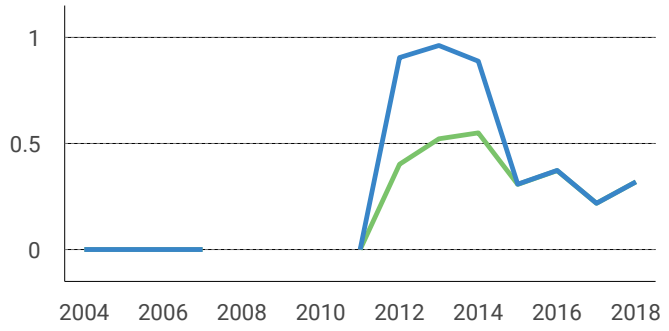

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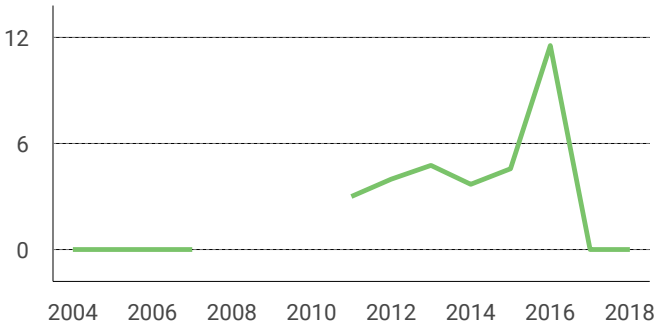

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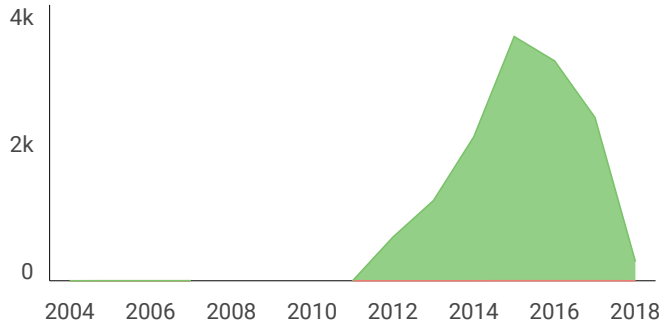

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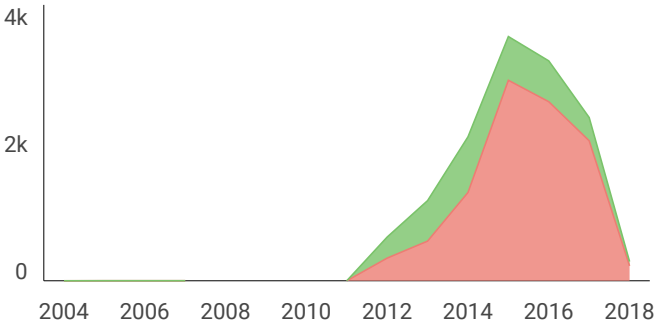

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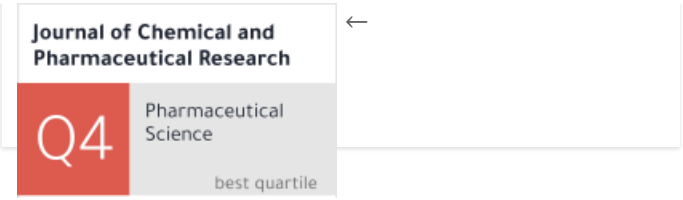

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Chalcones: Synthesis, structure diversity and pharmacological aspects

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ABSTRACT

The diverse pharmacological activities of chalcones, such as antioxidant, antimicrobial, anticancer, and antihepatotoxic, attract many researchers to isolate and elucidate them from nature and to develop efficient synthetic method. In the development, chalcones do not only comprise derivatives of trans-1,3-diaryl-2-propen-1-ones, but also their analog. Chalcones isolated from nature show exotic structure, which is sometime unrecognizable directly. The structure diversity of chalcone whether from nature or synthetic origin and various synthetic method of chalcone are discussed. Furthermore the bi-electrophile character of chalcone makes them more attractive to be used as synthon in the synthesis of heterocyclic compounds, such as pyrazoline, pyrimidinone, and benzazepine, through cyclo-condensation reaction with a bi-nucleophilic species.

Key words: chalcone, pyrazoline, pyrimidinone, and benzazepine

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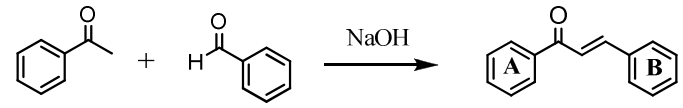
INTRODUCTION

Chalcones (*trans*-1,3-diaryl-2-propen-1-ones) are natural products belong to flavonoid, are considered as intermediate in the flavonoids biosynthesis, and are widespread in plants. The existence of the α,β-unsaturated ketone moiety in chalcones is a common part found in a large number of biological active compounds. Therefore, chalcone derivatives from nature or synthetic origin exhibit diverse pharmacological activities, such as antimicrobial [1], antitumor [2], anticancer [3,4], radical scavenger [5], and inhibitor of topoisomerase I [6]. However, isolation of chalcone derivatives from nature requires a long and usually complicated procedure which does not comparable to the yield obtained. Due to time consuming and intensive process in the isolation procedure, and to their diverse pharmacological activities, the development of an efficient synthetic protocol of chalcone derivatives attracts many researchers. A good synthetic method gives us advantages to obtain chalcone derivatives attaching various substituents in excellent yield which possibly do not exist in nature. Furthermore, chalcones are known as the key intermediate in the synthesis of various biologically important heterocyclic compounds. In this article, the preparation methods of chalcones, structure diversity, role of chalcone as synthon for the synthesis of diverse heterocyclic compounds, and their biological activity are reviewed.

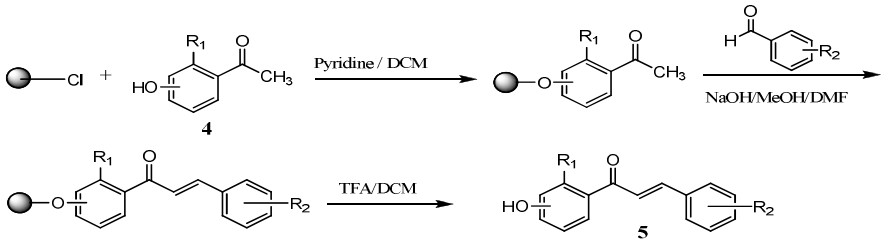
SYNTHESIZE OF CHALCONES

Chalcone (**1**) and its derivatives are primarily synthesized in the laboratory using Claisen-Schmidt reaction, in which acetophenone (**2**) or its derivative is reacted with benzaldehyde (**3**) or its derivative using strong base, such as NaOH, KOH, or NaH as catalyst in a polar solvent as shown in the following reaction [7]. Other catalysts are also used, such as sodium phosphate doped sodium nitrite [8] and aluminum-magnesium hydroxide hydrate [9].



2 **3** **1**

Solid phase Claisen-Schmidt reaction employing various solid catalysts was applied to synthesize chalcones by different scientific group. Cross aldol condensation catalyzed by complex of Co(II)-pyridine polymer was applied to synthesize chalcone. The complex of Co(II)-cross linking 4-vinyl pyridine-styrene showed the best performance, in which no side product was observed [10]. Solid phase synthesis of chalcones using 2-chlorotrytilchloride as supporting resin has also been performed [11]. First, the hydroxy-acetophenone (**4**) derivatives was bounded to the resin, and then treated with derivatives of benzaldehydes using NaOH as catalyst in methanol. The formed hydroxychalcones (**5**) were then released by the addition of trichloro acetic acid.



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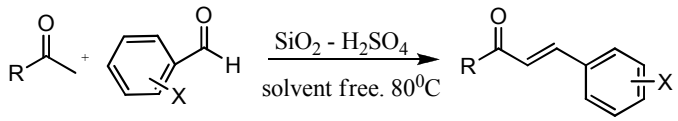
●-Cl = tritylchloride resin

Solid phase cross aldol condensation employing magnesium hydrogen sulphate was used to synthesize chalcone in good yield and self condensation products were not observed [12]. In attempt to accelerate the development of green chemistry, solvent free Claisen-Schmidt reaction was conducted using polymer as supporting material and TBD (1,5,7-trisazabicyclo[4,4,0]dec-en) as catalyst [13]. Solid phase synthesis of chalcones was also conducted using silica-sulfuric acid as catalyst. The conversion of reactants into product proceeded completely by heating the reaction mixture at 80^0C for 2 until 3 hours. The catalyst was made by the reaction of silica gel with chlorosulfonic acid [14].

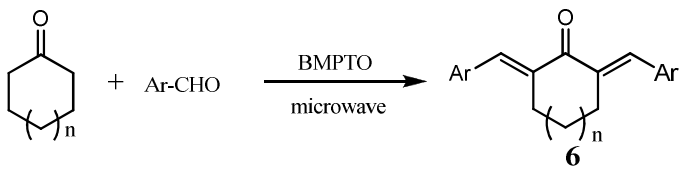


R = 4-biphenyl or 9H-2-fluorenyl

X = H, *m*-NH_2, *p*-NH_2, *m*-Br, *m*-Cl, *p*-Cl, *p*-NMe_2, *p*-OH, *p*-OMe, *p*-Me, *o*-NO_2, *m*-NO_2, *p*-NO_2

Pursuing an environmentally benign reaction condition, the use of Zn (L-proline)$_2$ as catalyst in the synthesis of chromonyl-chalcones in water was successfully conducted. The eminence of this protocol was the use of water as solvent which is nontoxic, cheap, and non-flammable. Moreover, Zn (L-proline)$_2$ can be easily recovered and reused several time without significant loss of its performance [15].

Microwave irradiation induced reaction in the chalcones synthesis is another alternative procedure to synthesize chalcones. This reaction method can shorten the reaction time and simplify the purification procedure. Cross aldol condensation by using microwave was used for the synthesis of chalcone analog namely 2,6-bis(benzyliden)-cyclohexanone (**6**) employing BMPTO (bis-(4-methoxyphenyl)-telluroxide) as catalyst [16].



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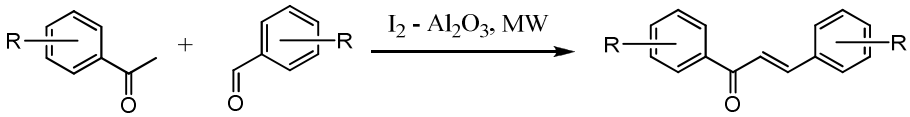
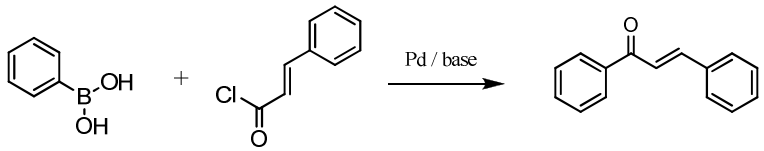
An attractive single step synthesis protocol of chalcone was reported, in which chalcones were synthesized using molecular iodine impregnated over neutral alumina as catalyst, and employing microwave irradiation as source of energy without any solvent [17]. Under this reaction condition, preparation of polyhydroxychalcones can be conducted by reacting of hydroxylated acetophenone and hydroxylated benzaldehyd without using any protecting group, which is impossible to be conducted by alkaline catalyzed reaction. The molecular iodine acts as Lewis acid, which facilitates the enolisation of the hydroxyl aryl ketone as well as activates the carbonyl group of hydroxyl benzaldehyde towards nucleophilic attack. The neutral alumina powder serves to enlarge the effective catalytic surface area.

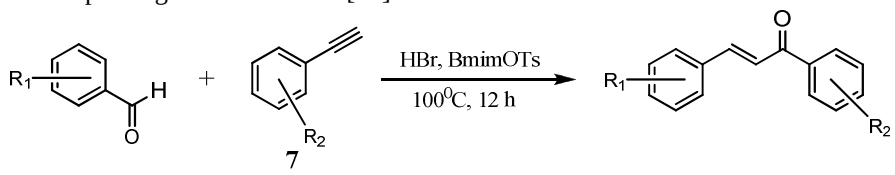


Acid catalysed of hydroxylated chalcones synthesis was successfully conducted by Jayapal and co-worker without any protecting step. The acid catalyst was prepared in situ from $SOCl_2$ and ethanol [18]. The combination of continuous flow processes and microwave technology is a relatively new method applied in organic synthesis. This technique offers many advantages, both in technical and economical aspects. Applying this technique, synthesis of chalcone derivatives were successfully conducted using mix-solution of phenyl acetylenes and benzaldehydes in 1,2-dichloroethane. Initially, the solution mixture was flowed continuously by a pump through the reaction vessel charged with amberlyst-15 as a solid acid catalyst, which was inserted in a reactor equipped with microwave generator (50 W) in a defined time. Solution of the crude product was subsequently collected from the outlet tube, and then purified chromatographically. The electron-rich or electron-poor substrates are tolerable in this reaction condition and the corresponding products are obtained in good yield [18].

More exotic synthetic protocols have also been developed to pursue high reaction yield and to minimize the side reaction. Chalcone could be synthesized using Suzuki reaction, employing cynnamoyl-chloride and phenyl boronic acids as reagents and Pd as catalyst in base reaction condition [20].



Coupling reaction between aromatic alkynes (**7)** and aldehyde in ionic solution was applied to synthesize of chalcones with high conversion. It was found that aromatic aldehydes whether attach electron withdrawing or electron donating group are able to proceed coupling reaction. However, the use of aliphatic alkynes does not give any products, even in a prolonged reaction time [21].



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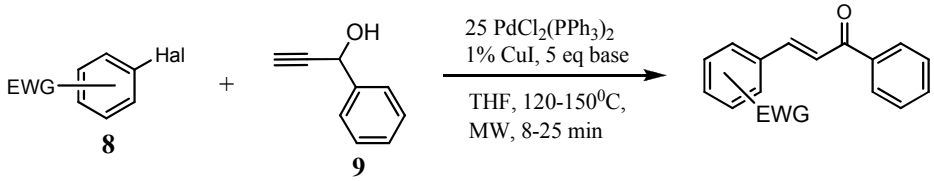
The application of Sonogashira isomerisation coupling reaction under microwave-assisted condition was used to synthesize chalcones through a reaction between aryl-halide attaching electron withdrawing group (**8**) and propargyl alcohol (**9)** [22]. Using this method, high yield was obtained in short reaction time (8 – 25 minutes).



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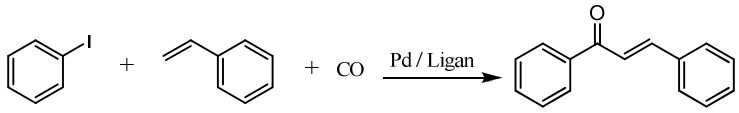
Carbonylative Heck coupling reaction employing Pd as catalyst is another protocol to produce chalcone by the reaction between aryl halide and styrene in the presence of carbon monoxide [23].

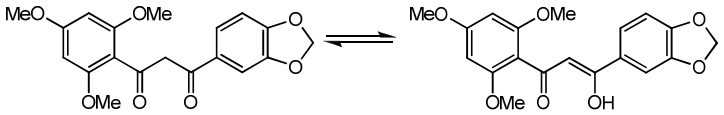


STRUCTURE DIVERSITY

Classical structure of chalcones is the derivatives of 1,3-diphenyl-2-propen-1-one and they are usually from natural products. However in the development, chalcones do not only comprise derivatives of 1,3-diphenyl-2-propene-1-one, but also derivatives of 2-propene-1-one, in which at the position 1 and 3 attached other aromatic ring or even non-aromatic ring. The structurally modified chalcones are usually obtained from the synthetic origin.

Natural origin

The classical substitution pattern of hydroxyl group in ring A of natural chalcone is 2,4,6-trihydroxy or 2,4-dihydroxy, whereas in ring B could be 4-hydroxy, 3,4-dihydroxy, 3,4,5-trihydroxy, or no substituent attached in it [23,24]. An interesting small group of natural chalcones is β-hydroxy-chalcones, which occur as the enol tautomers of dibenzoylmethane derivatives (**10**). Galliposin (**11**) isolated from the stem bark of *Galipea granulose* (Rutaceae) possessing β-hydroxy and dioxymethylene substituens is an interesting example of this group [26]



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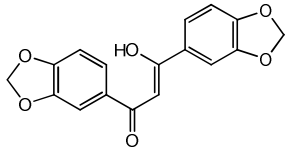
Tautomerization of dibenzoylmethane



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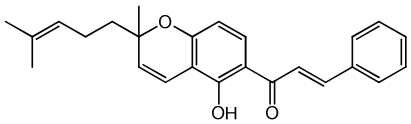
During period 1992 until 2003, more than 80 new isoprenylated chalcones have been reported, mostly from the Leguminosae. The isoprenyl moiety exists in an open chain or in a cyclic form [25]. Spinochalcone-B (**12**) isolated from *Tephrosia* is a representative of cyclic farnesylated chalcones [27], whereas Xanthohumol-D (**13**) from *Humulus lupulus* is a representative of an open chain isoprenylated chalcones [28].



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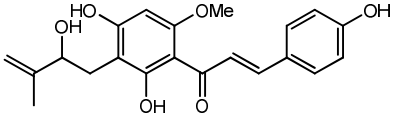

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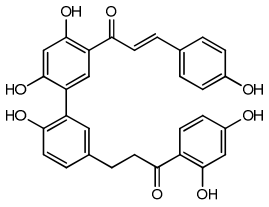
Chalcone dimers or oligomers are also found in plant, especially in the family of Ochnaceae and Anachardiaceae. To form a dimer, two chalcones are linked whether by a single C-C bond or by C-O-C bond. Rushchalcone VI (**14**) obtained from the twigs and stem bark of *Rhus pyroides* (Anacardiaceae) is an example of chalcone dimer formed from two molecules of isoliquiritigenin (2',4',4-trihydroxychalcone) linked by a C-C single bond between C-3 and C-5'. On the other hand Rushchalcone I (**15**) and II (**16**) represent unsymmetrical chalcone dimmers linked by C-O-C single bond, which is composed from isoliquiritigenin and its 4'methyl ether derivative [29,30].



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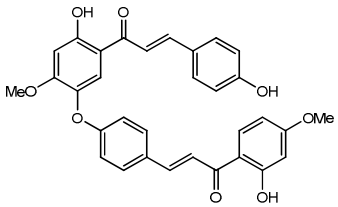

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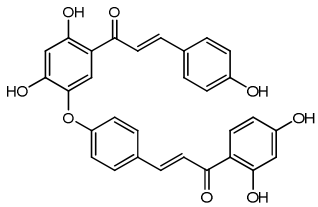

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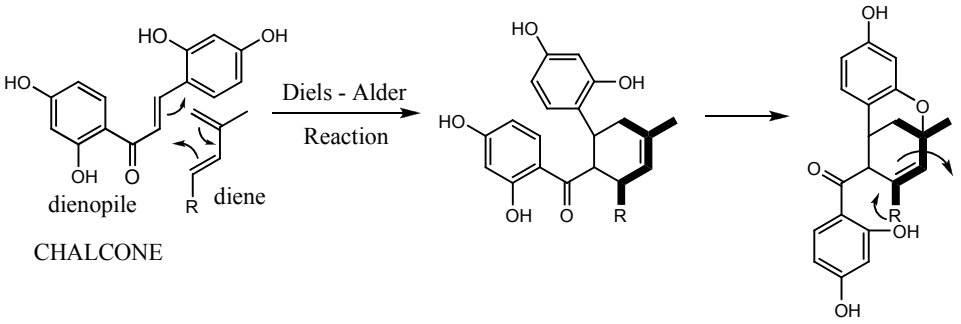
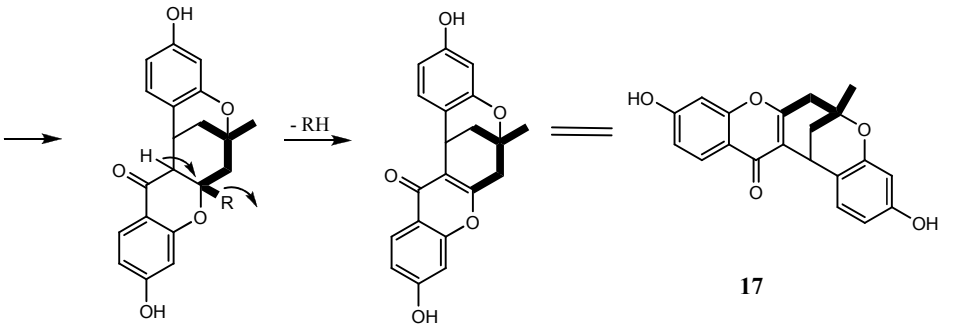
Based on their chemical structure, a characteristic feature of chalcones is their ability to act as dienophiles in an enzymatic Diels-Alder reaction. Serving as dienes in this reaction are range from isoprene, monoterpene, coumarine, and other class of flavonoid. The Diels-Alder adducts of chalcone are mostly found in family Moraceae. However, they can also be found in Annonaceae and Zingiberaceae [24]. The biosynthetic pathway of Sanggenon R (**17**) proposed by Hano et al [31] is an example of the enzymatic Diels-Alder reaction between a chalcone as dienophile and an isoprene as diene. The product of this reaction proceeds subsequently rearrangement and oxidation reaction to form sanggenon R.

The following compounds fissistin (**18**), Palodesangren C (**19**), Mulberrofuran U (**20**), Dorstenone (**21**), and Sanggenol M (**22**) are some representatives of chalcones formed through the Diels-Alder reaction between chalcone derivatives as dienophile and various kinds of diene.





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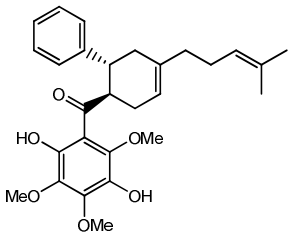

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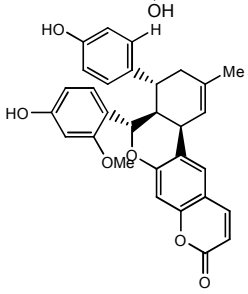

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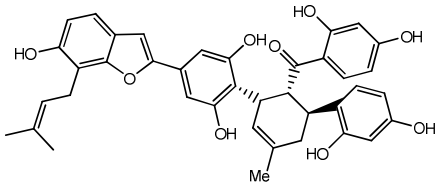

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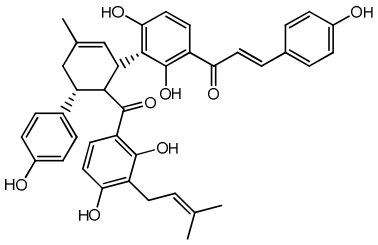

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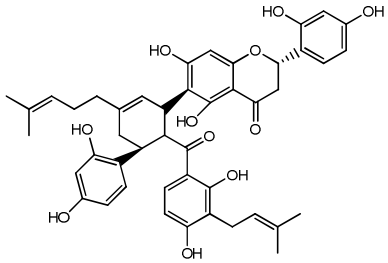

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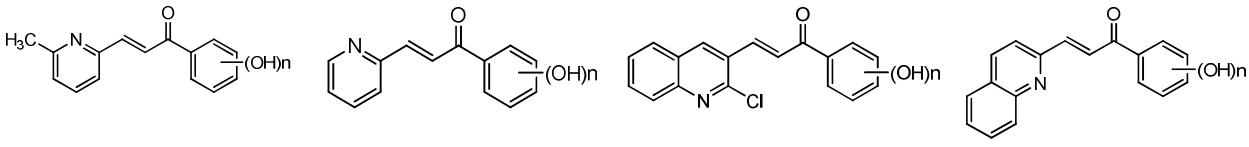
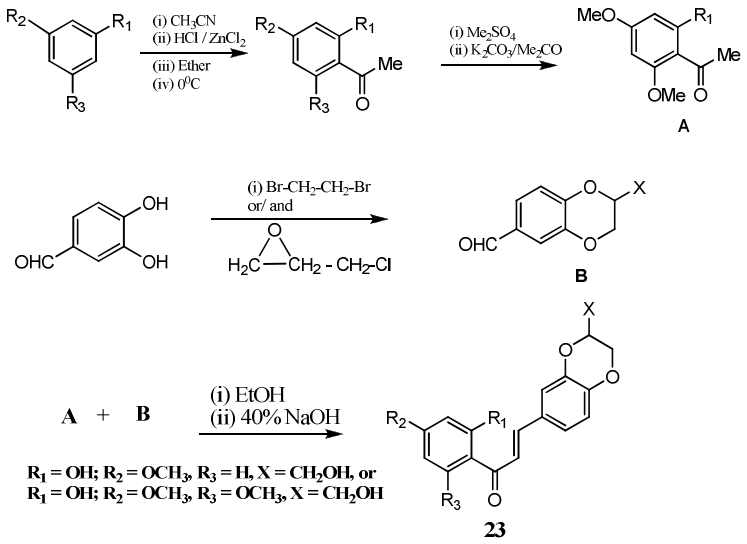
Synthetic origin

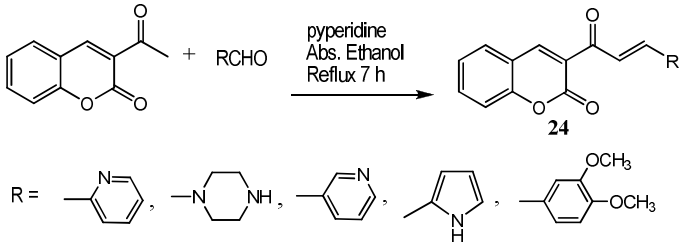
The molecular structure of chalcones of synthetic origin comprises both of derivatives of 1,3-diphenyl-2-propen-1-one and derivatives of 2-propen-1-one in which at the position 1 and 3 other aromatic or non aromatic ring attached . The classical structures of chalcones are not discussed in this part of the article. Cheng and coworkers [11] have synthesized chalcones composed of pyridine, quinoline ring, and polyhydroxy aromatic.



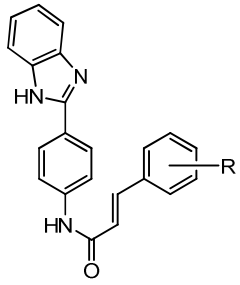
Chalcones possessing benzo-1,4-dioxane (**23**) ring have also been synthesized. The benzo-1,4-dioxane ring was made from the reaction between 1,2-dihydroxybenzaldehyde with a derivative of an epoxide. The aldehyde obtained was then treated with the derivatives of acetophenone to produce the desired products [32].



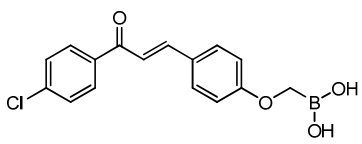
The synthesis of coumarine-based chalcones (**24**) was successfully conducted by Claisen-Schmidt condensation applying piperidine as catalyst in ethanol [1].



Baviskar and co-workers [33] have synthesized some novel benzimidazolyl chalcones (**25**) in attempt to gain antimicrobial agents by condensation of N-(4-(1*H*-benzo[*d*]imidazol-2-yl) phenyl)acetamide with aromatic aldehydes in presence of aqueous potassium hydroxide at room temperature.



AM 58 (**26**) and AM 114 (**27**) are examples of chalcone-analogues bearing of boronic group which showed anticancer activity [34], whereas two others boronic chalcones were synthesized as fluorescent probes for saccharides signaling analysis [35].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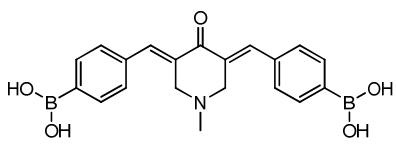
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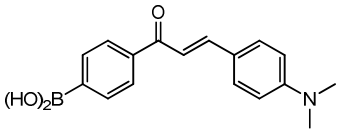
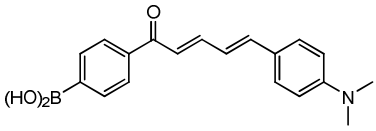
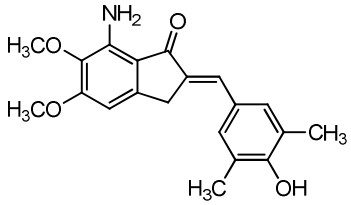



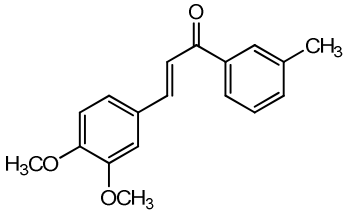
Indanocine (**28**) is an example of another chalcone analog which is classified as annulated chalcone. It is a chalcone, where the C-3 part is in a cyclic form [36].



28

FUNCTIONAL GROUP TRANSFORMATION OF CHALCONE

Enone moiety of chalcone is an important part in functional group or structure transformation, due to its reactivity to be transformed into other functional group. The transformation can take place whether on the carbonyl or on alkenes group. The carbon-carbon double bond can be reduced into carbon-carbon single bond under hydrogen gas atmosphere using various catalyst such as Raney nickel, Adam catalyst (PtO_2), Pd/C, Rh-Al_2O_3. Although these heterogeneous catalysts are useful in the hydrogenation process, they show bad selectivity if the reduced compound possesses more than one functional group. Furthermore, the application of homogeneous catalyst in the hydrogenation of carbon double bond is also reported employing rhodium or ruthenium complex, such as Wilkinson catalyst [$(Ph_3P)_3RhCl$] and [$(Ph_3P)_3RuClH$]. It is an efficient catalyst for hydrogenation of unconjugated homogeneous alkene at standard temperature and pressure [36]. An interesting result was obtained by Alptuzun & Gozler [38] when they hydrogenated chalcone 3-(3,4-dimethoxyphenyl)-1-(3-tolyl)-2-propenone (**29**) using Zn/acetic acid in attempting to get a saturated ketone (**30)** . Indeed they got the desired product, but only as side product. The major product obtained was the derivative of cyclopentanol (**31**) as the result of cyclodimerization.



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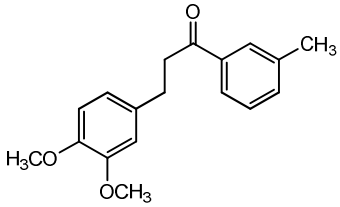

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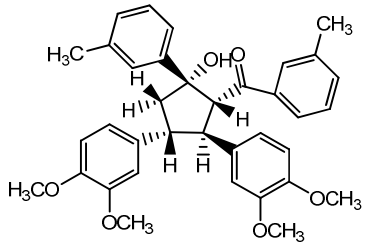
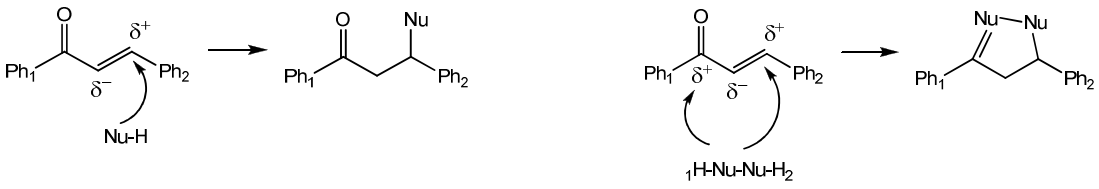


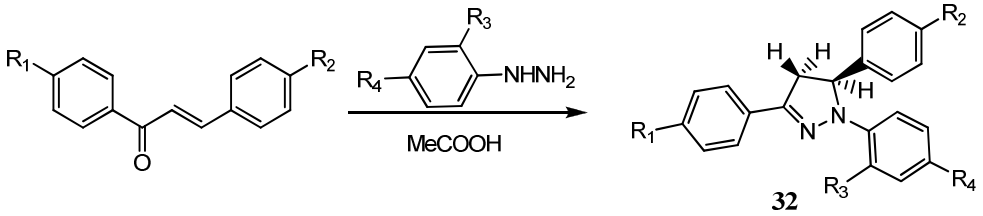
31

CHALCONE AS SYNTHON IN HETEROCYCLES SYNTHESIS

Chalcones are versatile precursors in the synthesis of heterocyclic compounds. From the organic synthesis point of view, enone moiety is important for the structural transformation of chalcones. Acting as an electrophile, chalcones can react with a nucleophile in Michael addition. In a cyclocondensation reaction, chalcones can act as a bi-electrophile which reacts with a bi-nucleophile, and this is an attractive route for the synthesis of heterocyclic compounds [22], such as derivative of pyrazoline, oxiran, pyran, oxopyrimidine, isoxazoline [39], derivatives of pyridine [40], derivatives of benzheteroazepine [41], and other heterocycles. Furthermore, substituted cyclohexenone can also be made from substituted chalcones [60].



Pyrazoline (**32**) is a five-ring heterocycle which is composed of two nitrogen atoms and three carbon atoms, and several procedures of their synthesis have been developed. The most popular preparation procedure is the reaction of α,β-unsaturated carbonyl compound with hydrazine. Chalcone can be used as source of α,β-unsaturated carbonyl compound, and the reaction is conducted in weak acidic condition [42].



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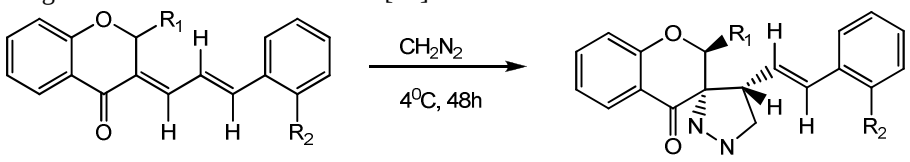
This synthesis route of pyrazoline derivatives was also reported by Katritzky and coworkers [43], Patel and coworkers [44], and Shah et al [45]. An interesting synthesis method of spiro-pyrazoline scaffold (**33**) using exocyclic α,β,γ,δ-unsaturated ketone as synthon was proposed by allowed it to react with diazomethane, and the reaction proceed regioselective and stereoselective [46].



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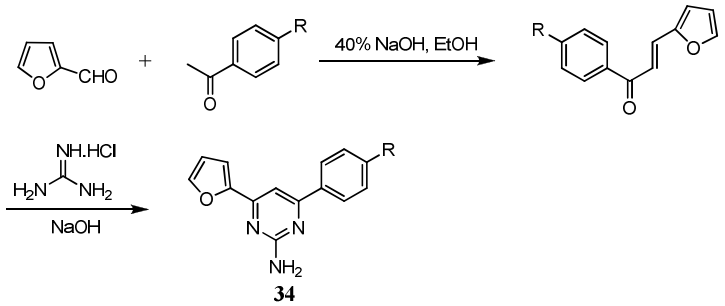
One of the most important six-member heterocyclic is pyrimidine. Pyrimidine scaffold exists in many natural and synthetic biologically active materials and also found as constituents of DNA or RNA base. Various synthesis route of pyrimidinone-nucleus has been reported by many authors, such as through one pot three-component Biginelli reaction [47], ring annulation of 2-amino-2-oxazoline acting as heteroamidine synthon by using two nucleophilic sites of the amidine moiety [48], and by tandem reaction of aza-Wittig/heterocumulene-mediated annulations [49]. However the most applied synthesis route of substituted pyrimidinones is the reaction of enone moiety of chalcone derivatives with urea or thiourea [50 - 53]. As reaction example of the transformation of a chalcone derivative into substituted pyrimidine (**34**) is displayed in the following reaction [54].



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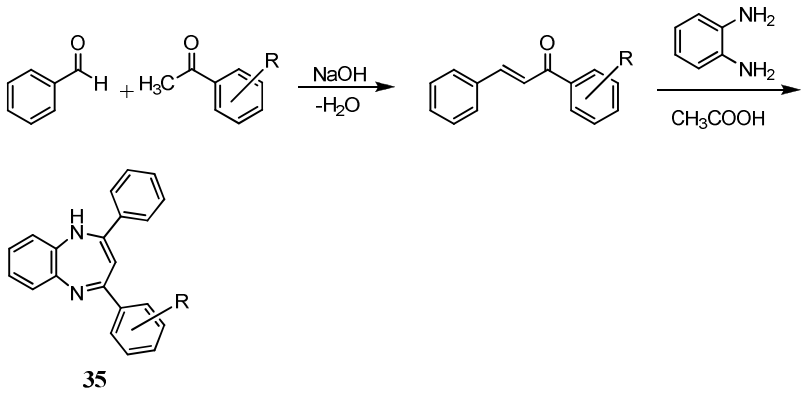
The classical method to synthesize seven-membered ring heterocyclic compound is ring enlargement reaction using Beckmann rearrangement [55]. However, due to its bi-electrophilic character, chalcone give an alternative route to build seven-membered ring through reaction with a bi-nucleophile to form derivatives of azepines, oxepines, or thiepins. Using chalcone derivative as intermediate and 1,2-diamino benzene, Bhatia et al [56] have successfully synthesized 2,4-disubstituted 1,5-benzodiazepine (**35**) as antibacterial agent. This synthesis route was also used to synthesize benzodiazepine [57].



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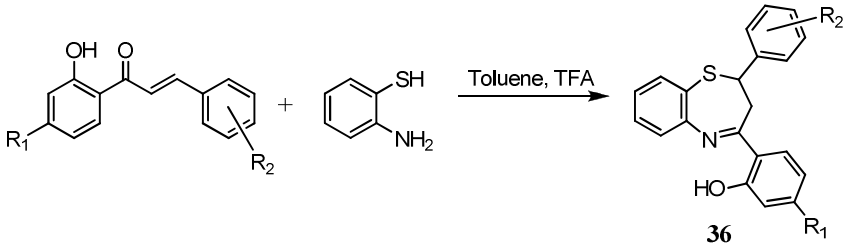
Applying the same synthesize route, many authors [58-61] have successfully synthesized benzothiazepine derivatives by the reaction of chalcone or its analog with 2-amino thiophenol,. The reaction equation of the synthesis of benzothiazepine (**36**) from chalcone derivative and 2-amino thiophenol is displayed below [62].



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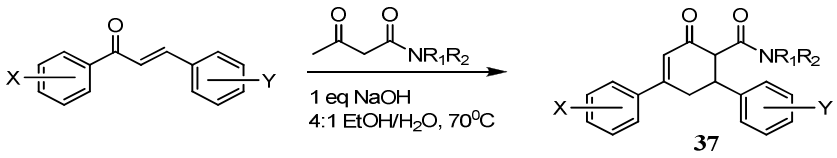
Beside used in the synthesis of heterocyclic compounds, chalcone is also applied in the synthesis of cyclohexenone derivatives (**37**). Preparation of cyclohexenone derivatives by the reaction between chalcone and 1,3-dicarbonyl compound was performed successfully and proceeded through Robinson annulations [63].



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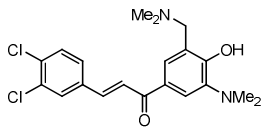
BIOACTIVITY OF CHALCONES

Chalcones are valuable chemicals because of their well known diverse pharmacological activity. A number of chalcones have demonstrated cytotoxic properties which is an implication of anticancer activity. Dimnock et al [64] have studied cytotoxic property of a number of chalcones and their related Mannich base toward murine P388 and L1210 leukemia cell lines, as well as human tumor cell lines, and they found that compound **21** exhibited the highest activity toward L1210 and human tumor cells. Compound **38** is other compound of interest due to its huge differential in cytotoxicity between P388 and L1210 cells, whereas compound **42** exhibited a high therapeutic index by comparison of the toxicity of P388 cells toward Molt 4/C8 T-lymphocytes. The study showed that in general the Mannich bases were more toxic than the corresponding chalcones.



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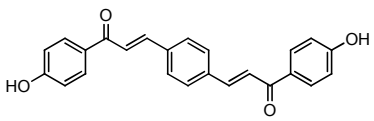

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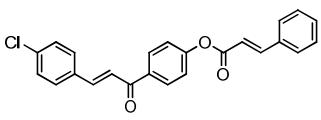

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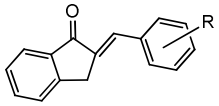
Attempting to determine the influence of relative orientation of the two phenyl rings toward cytotoxicity effect, Dimnock et al [65] studied the cytotoxicity properties of 2-arylideneindanones **41**, 2-arylidenetetralones **42**, and 2-arylidenesuberones **43** derivatives against murine P388, L1210, and Molt 4/C8 cancer cell lines, and found out that in general the order of cytotoxicity was **43** > **42** > **41**.



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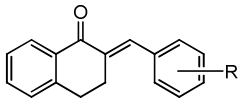

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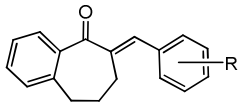
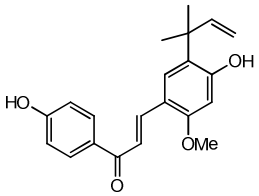


43

Substituted 6,7-dimethoxy-1-tetralones and 5,6-dimethoxy-1-indanones have also been synthesized and evaluated of their antitumor activity [36] and it was found that position of the lipophilic substituent affected the cytotoxic activity. 2'-amino chalcone derivatives displayed potential antitumor activity and also demonstrated significantly increased antitumor properties compared with the corresponding chalcones. Furthermore, position and the size of substituents are important for the activity of 2'amino chalcone [66].

Explorations of the mechanism of action an anticancer agent bring us to a better understanding of cancer and can lead us to design better anticancer drug. Licochalcones A (**44**) and E (**45**) are retrochalcone isolated from the root of *Glycyrrhiza inflata* exhibited the DNA topoisomerase I inhibitory activity in dose dependent manners and this property might explain the cytotoxic activity of these compounds against some human cancer cells line [6].



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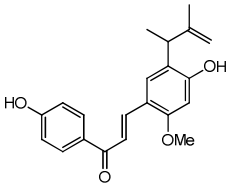

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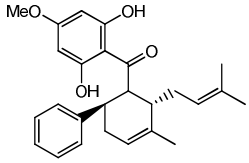
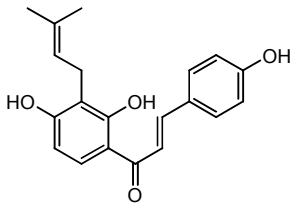


46

The mechanism of action of cytotoxic property toward human prostate cancer cell lines of Panduratin A (**46**) isolated form *Kaempferia pandurata* has already been explored [4]. Panduratin A was able to induce apoptosis through inhibition of procaspases 9, 8, 6, and 3 with significant increase in the ratio of Bax:Bcl-2. It indicates that the process involving a mitochondrial-dependent apoptotic pathway. Similar cytotoxic mechanism of action was shown by isobavachalcone (**47**), a constituent of *Angelica keiskei*. It reduced significantly pro-caspase-3 and pro-caspase-9 in neuroblastoma cell lines. Moreover, it can also activate Bax. These results suggested that mitochondrial-dependent pathway responsible for the apoptotic cell death in neuroblastoma by isobavachalcone application [67]. However, although the cytotoxic activity of xanthoangelol (**48**) - a major component of *Angelica keiskei* - is through apoptotic pathway, but it does not involve Bax/Bcl-2 signal transduction [68]. A boronic-chalcone derivative AM114 (**49**) also exhibited antimitotic activity through inhibition of proteasome and did not significantly disrupt the interaction of protein p53-MDM2 [34]



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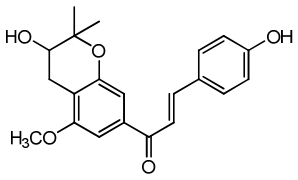

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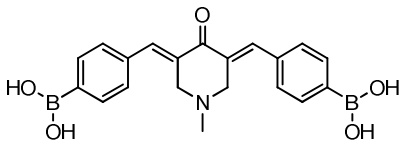
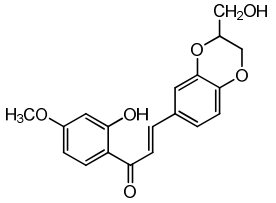


49

Due to the important role of 1,4-dioxane ring in displaying antihepatotoxic activity, chalcones bearing 1,4-dioxane ring system have been successfully designed, synthesized and tested to their antihepatotoxic activity. Compounds (**50**) and (**51**) exhibited potent activity compared to standard drug sylybon-70 [69]



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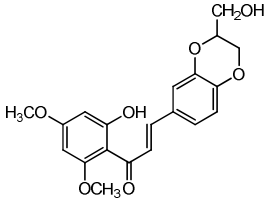
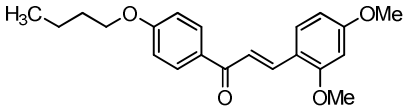


51

Derivatives of chalcones are also known for their antimalaria activity. Oxygenated chalcone such as 2,4-dimethoxy-4'butoxychalcone (**52**) exhibited potent activity against human malaria parasite *Plasmodium falciparum* in vitro

[70]. Ferrocenyl chalcone derivatives (**53**) showed antiplasmodial activity in vitro. Parameters influencing antiplasmodial activity were location of ferrocene and the polarity of the carbonyl linkage. Chalcones with ferrocene adjacent directly to the carbonyl linkage were displayed more selective and potent antiplasmodial activity [71].



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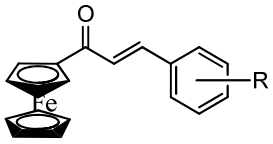

53

Another well known property of chalcones is their antimicrobial activity, and many authors have been reported about it. The chalcones of 2-hydroxy-1-acetonaphtone and 3-acetyl coumarin had been synthesized and tested of their antimicrobial activity. These compounds showed moderate to considerable of antibacterial and antifungal activity compared to the standard drugs chloramphenicol and fluconazole as positive control at dose of 1000 µg/mL [72]. Furthermore it was found out that chalcones attaching electron releasing group such as methoxy and hydroxyl group displayed better antibacterial activity than the others not having such functional groups [73], whereas chalcones having chloro, dichloro, or fluoro groups exhibited more antifungal activity. Chalcones bearing benzimidazolyl moiety exhibited active antibacterial and fungicidal properties [33], whereas the existence of phenyloxy moiety in chalcones is important for their antibacterial activity [74].

The existence of phenolic moiety in a compound indicates its antioxidant property. Synthesis of dihydroxylated chalcone derivatives and their radical-scavenging ability toward DPPH free radicals have been reported [5]. Furthermore study of the structure-activity relationship was also conducted. It was indicated that the very important structural factors to increase radical scavenging activity is the substitution pattern of two hydroxyl groups on ring B.

CONCLUSION

Chalcones are intermediate in the biosynthesis of flavonoid. They are valuable compounds whether from bioactivity aspects or from organic synthesis aspects. Chalcones exhibit diverse pharmacological activities and can serve as intermediate for synthesis of heterocyclic compounds. Due to these reasons, various preparation procedures were developed by many working groups, including ecofriendly protocol.

Acknowledgement
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