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PROCEEDING

INTERNATIONAL MULTIDISCIPLINARY CONFERENCE AND CALL FOR PAPER REVITALIZATION OF PROFESSIONAL ASSOCIATION AND SCIENTIFIC KNOWLEDGE FOR HRD OF HIGHER EDUCATION

(Workshops as an Organization Profession, International Conference,
MoA/MoU Multy Kampus, OJS Training)

Pontianak, December 6-7, 2016

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PREFACE

Praise being said to Allah Almighty God for all the grace and guidance that has been given to us all, so the Proceedings of the ADRI 2016 International Multidisciplinary Conference and Call for Papers. Proceedings contains a number of articles and research papers from lecturers, teachers, students, researchers and / or observers of the development of science and technology.

This seminar is the series of the International Seminar organized by ADRI, the first was held in Lombok, Mataram; The second was held in Denpasar, Bali October 15 to 17, 2016; the third was held in Surabaya, East Java, on November 10, 2016 and the fourth was held in Pontianak, West Kalimantan, on 6 to 7 November 2016. The fourth International Seminar in Pontianak's speakers came from 5 countries; Indonesia, Taiwan, United Kingdom, Italy and Malaysia. Call papers Participated in an international conference in Pontianak as much as 103 paper came from 5 countries, with a number of writers were 156 persons, from Indonesia came from 15 provinces. Most writers of West Kalimantan: 67 person and East Java: 41 people.

The international conference has been made to be held as the realization of cooperation between ADRI, National University of Kaohsiung in Taiwan, Universiti Tun Hussein Onn Malaysia, STKIP Singkawang, and all the universities participating in the MoU / MoA multi campus.

On this occasion let us give awards and gratitude to:

Keynote speaker

1. Prof., dr. Ali Ghufon Mukti, M.Sc., Ph.D., Dirjen Sumber Daya Ilmu Pengetahuan, Teknologi dan Pendidikan Tinggi
2. Dr. Ir. Jumain Appe, M.Sc., Direktur Jenderal Penguatan Inovasi, Kemenristek Dikti.
3. Prof. Dr. Paulina Pannen, M.Ls., Staf Ahli Bidang Akademik, Menristek Dikti.
4. Prof. Dr. I-Hsien Ting (Associate Professor Department of Information Management, National University of Kaohsiung, Taiwan)
5. Prof. Dr. Wahid Bin Razzaly, Universiti Tun Hussein Onn Malaysia (UTHM)
6. International speakers; Tirthendu Bagchi (Nottingham University, UK) and Cristina Lanteri (Italy)

In special award and we thank to:

1. Drs. Cornelis, M.H., Gubernur Kalimantan Barat.
2. Dr. H. Achmad Fathoni Rodli, M.Pd., General Chairman DPP P-ADRI Board.
3. Dr. M. Zeet Hamdy, Sekretaris Daerah Propinsi Kalimantan Barat.
4. Board of DPP ADRI
5. The Board of Trustees and Governing ADRI DPD Kalbar
6. Rector and Leadership College participant MoU / MoA multi-campus
7. Board of Editor, executive Editors and the Executive Committee in ADRI International Multidisciplinary Conference and Call for Papers in Pontianak
8. The sending of paper and parallel scientific conference speaker

In addition to the international conference, at the same time as a multi-campus realization cooperation activities, as well as activities carried out:

1. Inauguration of ADRI DPD West Kalimantan.
2. Training Open Journal System, as we know that from 2017 Kemenristek Dikti already requires all scientific journals should be based online by implementing OJS and scientific work for the maintenance of mandatory functional academic journals published in the OJS.

Proceedings are published in book form only contains abstract, distributed to participants in the form of compact disks (full paper) and published online at:

www.p-adri.or.id/prosiding/prosiding4pontianak.

Hopefully, these proceedings may give benefit to us all, for the development of science, technology, arts, culture, and sports. In addition, it is also expected to be a reference for the nation and state-building efforts so that science and technology become a strong pillar in the face of the ASEAN Economic Community.

Lastly, we are sorry if there are things less pleasing.

Sincerely,

Pontianak, December 6, 2016.
Chief Executive,

Drs. Andi Mursidi, M.Si.
Chairman ADRI DPD Kalbar

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- 30. UNIVERSITAS NAHDLATUL ULAMA SUMATERA BARAT**

TABLE OF CONTENTS

INTERNATIONAL KEYNOTE SPEAKERS _____	1
KNOWLEDGE MANAGEMENT IN THE ERA OF SOCIAL MEDIA	
I-HSIEN TING _____	2
AN IDEAL CLASSROOM IN AN IDEAL SCHOOL: LEAPING ACROSS BOUNDARIES – CREATING INTERNATIONAL MINDEDNESS THROUGH HOLISTIC EDUCATION	
MEITHIANA INDRASARI & TIRTHENDU BAGCHI _____	3
GENDER EMPOWERMENT PROJECTS AND CREATIVITY: BETTER MANAGEMENT OF RESOURCES TO OPTIMIZE RESULTS	
CRISTINA LANTERI _____	5
EDUCATION SCIENCE CALL PAPER _____	8
AN INTRODUCTION: EVALUATION OF QUALITY ASSURANCE FOR HIGHER EDUCATIONAL INSTITUTIONS USING RASCH MODEL	
ANDI MURSIDI & SOEHARTO _____	9
ASSESSMENT SYSTEM IN CURRICULUM 2013 OF ELEMENTARY SCHOOL IN SUMENEP DISTRICT MADURA ISLAND	
DIAN EKA INDRIANI _____	15
CIVIL SOCIETY ORGANIZATION IN EMPOWERING SOCIETY	
DADA SUHAIDA & MOAD _____	19
COGNITIVE ERGONOMICS ASPECT BENEFIT IN THE LEARNING PROCESS	
PT. GDE ERY SUARDANA _____	26
CONSTRAINT OF PAUD TEACHER’S INNOVATIVENESS	
HENNY SUHARYATI _____	30
DEVELOPING LEARNING MEDIA BASED ON AUGMENTED REALITY (AR) TO IMPROVE LEARNING MOTIVATION	
RIDHO DEDY ARIEF BUDIMAN _____	33
DEVELOPMENT OF CHARACTER EDUCATION BASED ON LOCAL WISDOM IN INDEGENOUS PEOPLE TENGAHAN SEDANGAGUNG	
DINA ANIKA MARHAYANI _____	38
EFFECT OF MOTIVATON AND CREATIVITY ON STUDENTS’ PSYCHOMOTOR ABILITY	
MUHAMAD ARPAN, DEWI SULISTYARINI, & DANAR SANTOSO _____	42
EFFECT OF SELF EFFICACY AND PRIOR KNOWLEDGE ON STUDENTS’ SKILLS	
RYAN PERMANA, FEBRIANTO SABIRIN, & VINDO FELADI _____	47
BLENDED LEARNING METHOD BASED ON LOCAL WISDOM AS A SPIRITUAL GUIDANCE HOLY TRINITY COMMUNITY IN DISTRICT BENGKAYANG	
PRISKA VASANTAN _____	53

ENHANCING COLLEGE STUDENTS' CRITICAL THINKING ABILITY THROUGH VARIED DISCUSSION METHOD IN CIVIC EDUCATION SUBJECTS	57
ROHANIL & ERNA OCTAVIA _____	57
ENVIRONMENTAL LEARNING APPROACHES IN IMPROVING LEARNING OUTCOMES IN ACID-BASE SUBJECT	60
RACHMAT SAHPUTRA, DWI WIDIARTI, & RAHMAT RASMAWAN _____	60
IMPLEMENTATION OF TEXT TRANSFORMATION IN PHYSICS EDUCATION TO REDUCE STUDENTS' MISCONCEPTION	66
SOEHARTO _____	66
IMPLEMENTATION OF MODEL SAVI (SOMATIC, AUDIOTORY, VISUALIZATION, INTELLECTUAL) TO INCREASE CRITICAL THINKING ABILITY IN CLASS IV OF SOCIAL SCIENCE LEARNING ON SOCIAL ISSUES IN THE LOCAL ENVIRONMENT	70
DADANG ISKANDAR, ACEP RONI HAMDANI, & TETI SUHARTINI _____	70
IMPROVING STUDENTS' READING ABILITY BY USING LOCAL FOLKLORE COMICS FRANSISKA DWI MULYANI	76
WIJAYANTI _____	76
IMPROVING LECTURERS' PEDAGOGIC COMPETENCE THROUGH THE IMPLEMENTATION OF LESSON STUDY IN FACULTY OF TEACHER TRAINING AND EDUCATION OF PAKUAN UNIVERSITY, INDONESIA	81
ERI SARIMANAH _____	81
INFLUENCE BETWEEN USING THE MALAY SAMBAS LANGUAGE IN MOVIES TOWARD DEVELOPMENT AND VITALITY OF THE EDUCATIONAL CHILDHOOD LANGUAGE	86
SRI MULYANI _____	86
MEDIA LITERACY COMPETENCY-ORIENTED LIFE SKILLS FOR HIGH SCHOOL TEACHER IN THE CITY OF BANDUNG IN THE FACE OF MEA	89
IRMAN AZIZ _____	89
SMARTPHONE BASED LEARNING TO IMPROVE THE QUALITY OF VOCATIONAL HIGH SCHOOL GRADUATES	97
SULFKAR SALLU, OTTO FAJARIANTO, KISNO, MEGA ACHDISTY N., & KAPRAJA SANGAJI	97
LOCAL LITERATURE REVITALIZATION IN ORDER TO MALAY LANGUAGE ENDURANCE	101
HARIES PRIBADY, LILI YANTI _____	101
NATIONALISM APPLYING IN LEARNING CIVIC EDUCATION AS MORAL LEARNING MEDIA IN UNIVERSITY	104
RINI SETYOWATI _____	104
OPTIMIZATION APPROACH FOR USE SAVI TO LEARNING OUTCOMES CREATIVITY WRITING POETRY OF LEARNING TECHNIQUE THROUGH DIRECT OBJECT	107
ZULFAHITA _____	107

PROBLEM SOLVING ABILITY IN PROBABILITY THEORY THROUGH PROBLEM SOLVING BASED LEARNING	
JAMILAH _____	110
A DESCRIPTION VOCABULARY MASTERY OF THE FIFTH GRADE STUDENTS OF SDN 3 SUNGAI PINYUH ACADEMIC YEAR 2016/2017	
IRMA MANDA NEGARA _____	113
THE STUDY OF LOCAL WISDOM VALUES IN NAIK DANGO CEREMONY AS CIVIC CULTURE IN KANAYATN DAYAKNESE SOCCIETY IN SAHAM VILLAGE	
PITALIS MAWARDI BAGING _____	117
THE APPLIANCE OF GENDER ANALYSIS MODEL SARA H. LONGWEE STUDY ON THE PROBLEM OF FEMALE LECTURERIN FUNCTIONAL POSITION IN HIGH EDUCATION	
WIDYATMIKE GEDE, ABDULLAH KARIM, & ENDANG DWI SULISTYAWATI _____	121
THE DEVELOPMENT OF BIOLOGY PRACTICUM LEARNING BASED ON VEE DIAGRAM FOR REDUCING STUDENT COGNITIVE LOAD	
ANNA FITRI HINDRIANA _____	125
THE IMPORTANCE OF SERVICE PLACEMENT AND CHANNELLINGTO PREPARE HUMAN RESOURCES FOR STUDENTS TO MEET THE CAREERS OF THE FUTURE	
EWI MARIANA _____	130
THE EFFECT OF ARIAS LEARNING MODEL AND STUDENT'S CREATIVITIES TO THE LEARNING OUTCOMES ON CONTINENTAL FOOD PROCESSING AND PRESENTING SUBJECT AT STATE VOCATIONAL SENIOR HIGH SCHOOL 3 BOGOR	
SUPARI MUSLIM, NISA RAHMANIYAH UTAMI, & RITA ISMAWATI _____	133
THE EFFECTIVENESS OF THE COLLABORATIVE LEARNING MODEL ON TRIGONOMETRY TOPIC OF SENIOR HIGH SCHOOL STUDENT GRADUATE X USING OPEN-ENDED APPROACH	
NURHAYATI _____	141
THE IMPLEMENTATION OF ICLOUD SYSTEM BASED ON KNOWLEDGE SHARING AT THE UNIVERSITY OF MAARIF HASYIM LATIH SIDOARJO	
ACHMAD FATHONI RODLI _____	146
THE INFLUENCE OF IMPLEMENTATION OF COOPERATION LEARNING MODEL TYPE NUMBER HEADS TOGETHER AND THINK-PAIR-SHARE TO THE CONCEPT COMPREHENSION OF ECONOMY	
PUPU SAEFUL RAHMAT _____	152
THE INFLUENCE OF ADVERTISING LANGUAGE TOWARD THE USE OF BAHASA INDONESIA	
FITRI _____	158
THE CORRELATION BETWEEN CARIOGENIC FOOD CONSUMPTION AND TOOTH BRUSHING HABITS WITH CARIES INCIDENCE AND DENTAL HYGIENE OF STUDENTS AT ELEMENTARY SCHOOL OF KAMPUNG OLO PADANG	
DEWI ELIANORA, ABU BAKAR, & RATIA SENGGANI _____	161
VALIDITY TEACHING MATERIALS OF INDONESIAN EDUCATION IN BEGINNING CLASS OF ELEMENTARY SCHOOL COURSE BASED INTEGRATED SCIENCE AND SOCIAL STUDIES	

WAHYU SUKARTININGSIH	166
VALUES OF CHARACTER IN TRADITIONAL CHILDREN GAMES IN WEST JAVA (STUDY OF ORAL TRADITION)	
YUSIDA GLORIANI	173
WRITTEN CORRECTIVE FEEDBACK: ENHANCING WRITING ABILITY THROUGH DIRECT CORRECTION	
DAYAT	176
DEVELOPING AN ADAPTIVE AND ENGAGING E-LEARNING MEDIA FOR E-LEARNING COURSE IN HIGHER EDUCATION	
UNUNG VERAWARDINA	183
LANGUAGE ATTITUDE AND THE SELECTION OF THE LANGUAGE OF URBAN STUDENTS IN IKIP PGRI PONTIANAK	
ELVA SULASTRIANA	188
ANALYSIS OF COMPETENCE EXAM MASTER (UKG) TEACHER IN ECONOMIC SMA IN JAKARTA	
SITI NURJANAH	193
ANALYSIS OF STUDENT'S ABILITY ON MATHEMATICAL REPRESENTATION IN JUNIOR HIGH SCHOOL	
SYARIFAH FADILLAH	201
 SCIENCE AND TECHNOLOGY	 209
ANALYSIS OF QUANTUM MECHANICS PARAMETERS TO HARMONIC OSCILLATOR BY USING SPREADSHEETS AS WELL AS ITS APPLICATION IN PHYSICS EDUCATION TECHNOLOGY	
ANDIKA KUSUMA WIJAYA, & ARIEF HERMANTO	210
CONCRETE TECHNOLOGY TO SUPPORT SUSTAINABLE TOURISM INFRASTRUCTURE	
SRI WIWOHO MUDJANARKO, & M. IKHSAN SETIAWAN, & KOESPIADI, & FREDY KURNIAWAN	215
DOMESTICATION OF LAIS (OMPOK HYPOTHALMUS) IN THE FISHPOND AS A SUSTAINABLE CONSERVATION EFFORT	
INFA MINGGAWATI, & LUKAS	217
REDESIGN OF ENVIRONMENTAL WORK WITH ERGONOMIC INTERVENTION TO REDUCE FATIGUE AND INCREASE OUTPUT PRODUCTION	
SAJIYO, & M. ADHI PRASNOWO	220
SMART CITY : E-SERVICE HOSPITAL IN PONTIANAK	
SYF.PUTRI AGUSTINI ALKADRI, MENUR WAHYU PANGESTIKA , & ALDA CENDEKIA SIREGAR	223
STUDY AND ANALYSIS OF AGRICULTURE SPATIAL PLANNING IN PENAJAM PASER UTARA, EAST KALIMANTAN, INDONESIA	
TUKIMUN, WAHYU MAHENDRA, & M IKHSAN SETIAWAN	226

THE ASSESSMENT OF ECOLOGY DIMENSION SUSTAINIBITY OF RICE PRODUCTION IN WEST KALIMANTAN	
EKAWATI, DARSONO, KUSNANDAR, & NOVIRA KUSRINI _____	229
GEOGRAPHIC INFORMATION SYSTEM (GIS) APPLICATION TO DETECT THE POTENTIAL FOR TOURISM GEOLOGY AND FOREST IN THE DISTRICT BERAU, EAST KALIMANTAN	
VEGA VITIANINGSIH ANIK, M. IKHSAN SETIAWAN, SRI WIWOHO MUDJANARKO, AGUS SUKOCO, TRI ADHI WIJAYA, & KHOLIDA NENGRUM _____	235
TIDE FORECAST USING RADIAL BASIS FUNCTION NEURAL NETWROK	
NERFITA NIKENTARI _____	237
SOCIAL SCIENCE CALL PAPER _____	240
URBAN AGRICULTURE TECHNOLOGY TO SUPPORT URBAN TOURISM	
YENI IKA PRATIWI, MAHRUS ALI, M. IKHSAN SETIAWAN, HERY BUDIYANTO, & BAMBANG SIGIT SUCAHYO _____	241
A STUDY OF ESSENTIAL BASIC VALUES WHICH SUPPORT SOCIAL HARMONY IN CONFLICT-PRONE AREAS (A PROFOUND STUDY AT PRIMARY SCHOOLS IN SAMBAS REGENCY, WEST KALIMANTAN)	
AUNURRAHMAN _____	244
ACCELERATING THE IMPROVEMENT OF INFRASTRUCTURE AND HUMAN RESOURCES TO SUPPORT NATIONAL ECONOMY GROWTH	
CHOLIL HASYIM, M. IKHSAN SETIAWAN, & VERONIKA NUGRAHENI SRI LESTARI _____	249
ADVERTISING JARGON FOR LOCAL PRODUCTS AS CREATIVE INDUSTRY	
ROSIDA TIURMA MANURUNG _____	252
THE CONTRIBUTION OF CONSCIOUSNESS THE TAXPAYER AGAINST TAX REVENUES IN THE KPP PRATAMA SURAKARTA	
ARIEF BUDHI DHARMA, ANDJARWANI PUTRI W, & ESKASARI PUTRI _____	257
COMPARATIVE ANALYSIS OF DETERMINATION AND MANAGEMENT HAJJ FEES IN INDONESIA AND MALAYSIA (STUDY OF COMPARATIVE ANALYSIS)	
DIAN NURMASTUTI, ARIEF BUDHI DHARMA, & YUNIATIN DKW _____	264
ANALYSIS OF THE FACTORS OF SERVICE, PRODUCT, PROMOTION TO THE DECISION OF THE COSTUMER DEMAND SERVICES PRODUCTS MANDIRI SHARIA BANK BATAM BRANCH	
LUKMANUL HAKIM _____	269
ANALYSIS OF THE USE OF APPLICATIONS ‘MOBILE BANKING’ BRI BUSINESS TRANSACTION OF TRADERS IN THE MARKET BENGKAYANG	
AGUSTINUS RAHANWARATI _____	275
ANALYSIS ON LEVEL OF SACCHARIN AND CYCLAMATE ADDITIVES INSIDE UP-PACKAGING-DRINK ATSD KARANG TENGAH, TANGERANG CITY	
YUSNIDAR YUSUF, M.RAMDHAN, & YUNI ROCHMAWATI _____	279

PERFORMANCE OF EQUITY MUTUAL FUNDS ACCORDING TO SHARPE, TREYNOR AND JENSEN METHODS PERIODE 2013-2015	
CATUR FATCHU UKHRIYAWATI _____	283
CHIEF ELECTION LAW OF REGIONAL AND PREVENTION OF CORRUPTION	
M. ZAMRONI, & AANG KUNAIFI _____	289
PETENCE DEVELOPMENT STRATEGY CORE INDUSTRIAL AREA DISTRICT OF NORTH KAYONG	
SYARIF AGUSSAID ALKADRIE _____	295
CREDIT UNION ROLE IN SUPPORTING CAPITAL SMALL AND MEDIUM ENTERPRISES (CASE STUDY LANTANG TIPO CREDIT UNION BRANCH OFFICE BENGKAYANG)	
SABINUS BENI, & BLASIUS MANGGU _____	301
DEVELOPMENT OF CREATIVE ECONOMY THROUGH THE BLUE OCEAN STRATEGY FOR A SMALL BUSINESS (STUDY ON BIDAI HANDICRAFT INDUSTRY IN THE DI IN THE DISTRICT OF JAGOI BABANG, BENGKAYANG)	
RISSA AYUSTIA _____	306
EFFECT OF EMOTIONAL INTELLEGENGE AND OCCUPATIONAL HELATH ON EMPLOYEE PERFORMANCE	
CHAMDAN PURNAMA, DINDA FATMAH _____	310
EFFECT OF WORK MOTIVATION AND JOB SATISFICATION OF MARKETING RESEARCH PART ERFORMANCE OF EMPLOYESS IN PT.DECKA MARKETING	
ASMARA INDAHINGWATI _____	317
FINANCIAL RATIO ANALYSIS FOR EVALUATION THE HEALTH AND DEVELOPMENT OF THE BUSINESS OF PEGADAIAN SYARI'AH (PERSERO) BRANCH SEI PANAS BATAM	
AZNEDRA _____	322
FISCAL DEPENDENCE ANALYSIS JOMBANG DISTRICT GOVERMENT REGIONAL AUTONOMY ERA (JOMBANG DISTRICT LOCAL REVENUE AGENCY)	
RACHYU PURBOWATI _____	333
IMPACT ANALYSIS SERVICE PERFORMANCE IN ESTABLISHING CUSTOMER SATISFACTION AND LOYALTY	
ISTININGSIH _____	341
ASYMMETRY INFORMATION: INVESTORS TRUST REFLECTION TOWARD QUALITY OF EARNINGS	
RATNA WIJAYA DANAR PARAMITA, & NOVIANSYAH RIZAL _____	344
ENVIRONMENTAL MANAGEMENT STRATEGIES IN FISH PROCESSING BY IMPLEMENTING CLEANER PRODUCTION	
ERINA RAHMADYANTI & ANDRE DWIJANTO WITJAKSONO _____	349
JOINT ECONOMIC LOT SIZE IN THREE LEVEL SUPPLY CHAIN WITH PROBABILISTIC DEMAND	
MOCH. ANSHORI _____	354
LINEAR TREND ANALYSIS IMPACT OF INCREASING INVESTMENT IN AREA OF DEVELOPING COMMERCIAL PROPERTIES	

JOKO SUYONO, M. IKHSAN SETIAWAN, AGUS SUKOCO, SRI WIWOHO MUDJANARKO, & SANTIRIANINGRUM S _____	358
LOCAL WISDOM AS AN IMPORTANT ASPECT IN THE SPIRITUAL CAPITAL OF TRADITIONAL KINSHIP	
HELENA ANGGRAENI (RENI) TJONDRO SUGIANTO _____	363
MANAGING HUMAN RESOURCE MANAGEMENT IN BUSINESS ENVIRONMENT IN THE ERA OF THE ASEAN ECONOMIC COMMUNITY IN WEST NUSA TENGGARA REGION	
BAIQ SALKIAH, & DIDIN HADI SAPUTRA _____	367
OUTSOURCING MODEL IN PERSPECTIVE LEGAL PROTECTION AND LABOR RIGHTS	
YUNIATIN TRISNAWATI DWI K, ANDJARWANI PUTRI, & DIAN NURMASTUTI _____	370
POTENTIALS OF SUBAK TO DEVELOP AGRO-TOURISM IN BALI PROVINCE	
MADE SUMITRA CHANDRA JAYA, PUTU DYATMIKAWATI, & GEDE SEDANA _____	378
IMPLEMENTATION OF ARTICIAL INSEMINATION SNAPPING LUST (GBIB) IN CATTLE IN THE DISTRICT CITY WEST K. CENTRAL KALIMANTAN PROVINCE	
TRESIA KRISTIANA _____	381
RAISING PUBLIC AWARENESS OF RUBBISH RECYCLING; THE EXPERIENCE OF PONTIANAK CITY, WEST KALIMANTAN	
DONNA YOULLA, & SOEMARNO _____	387
RECONSTRUCTION OF LEGAL PROTECTION THE TENURE RIGHTS OF LAND AND BUILDING ON THE RIPARIAN ZONE BASED ON VALUES OF	
SETYO UTOMO _____	392
SME'S CENTER: PUBLIC-PRIVATE PARTNERSHIP FOR ACCELERATING REGIONAL ECONOMIC	
M. IKHSAN SETIAWAN, AGUS SUKOCO, SRI WIWOHO MUDJANARKO, & ISWACHYU DHANIARTI _____	396
STRATEGIC MARKETING AND COMPETITIVE STRATEGY OF SMES IN THE ERA OF ASEAN ECONOMIC COMMUNITY	
FAHRUDDIN SALIM _____	400
STRATEGY OF THE TRADERS IN PASAR RAYA PADANG AGAINST ABOUT REHABILITATION AND RECONSTRUCTION POLICY AFTER EARTHQUAKE	
RINEL FITLAYENI _____	403
STUDY ISLAMIC CONSUMPTION THEORY: REVIEW OF PUBLIC CONSUMPTION PATTERNS IN SURABAYA	
ROHMASARI, SLAMET RIYADI, & TRI RATNAWATI _____	408
SUPPORTING COOPERATIVE THROUGH IMPROVEMENT OF ORGANIZATIONAL CAPACITIES LESSON FROM FARMERS' COOPERATIVE IN BALI	
PUTU DYATMIKAWATI, MADE SUMITRA CHANDRA JAYA, & GEDE SEDANA _____	412
A STUDY OF FISH NURSERY AROUND PEOPLE'S HOME IN THE SUBDISTRICT OF BENGKAYANG	
SHANTI VERONICA BR SIAHAAN _____	416

THE IMPACT OF HUMAN RESOURCES COMPETENCE, INFORMATION TECHNOLOGY UTILIZATION,
FINANCIAL SUPERVISION AND ACCOUNTING INTERNAL CONTROL TO TIMELINESS OF FINANCIAL
REPORTING IN JOMBANG PUBLIC HOSPITAL

DWI ERMAYANTI S. _____ **420**

THE NAROTAMA FUND MANAGEMENT BASED ON THE ACCREDITATION OF BAN-PT

AGUS SUKOCO, M. IKHSAN SETIAWAN, & ISWACHYU DHANIARTI _____ **427**

THE POWER OF ARBITARIAN CLAUSE IN AN AGREEMENT AS A CHOICE OF LAW TO RESOLVE THE
BUSINESS DISPUTE

ANNURDI _____ **430**

THE SUCCESS OF HATCHING DUCK'S EGG IN PETIK MAS PROGRAM AT PADANG

RUDY KUSUMA _____ **433**

UTILIZATION OF RUPIAHS CURRENCY IN JAGOI BABANG BORDER

YOSUA DAMAS SADEWO, & PIETER RADIANTUS _____ **436**

WEAVING CULTURE OF THE DAYAK KENINJAL (CASE STUDY IN RIBANG SEMALAN VILLAGE,
TANAH PINOH DISTRICT -MELAWI REGENCY)

KRISTIANUS, & MAGDALENA _____ **440**

GENDER INEQUALITIES IN THE NOVEL ISINGA AND EMANCIPATION STRUGGLE

PRIMA GUSTI YANTI, UMMUL QURA _____ **450**

UNIVERSALITY OF "ADIL KA' TALINO, BACURAMIN KA' SARUGA, BASENGAT KA' JUBATA",
SYNCHRONISTIC APPROACH ON THE FAMOUS BYWORD OF DAYAKi KANAYATNiï TRIBE

FERRY HARTONO _____ **455**

MICROBIAL CONTAMINATION ON SLAUGHTERHOUSE IN KENDARI

HARAPIN HAFID, NURAINI, ANDI MURLINA TASSE, INDERAWATI, & MUH. HASDAR _____ **459**

ANALYSIS ON LEVEL OF SACCHARIN AND CYCLAMATE ADDITIVES INSIDE CUP-PACKAGING-DRINK ATSD KARANG TENGAH, TANGERANG CITY

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Abstract. The use of food additives today were extremely diverse, from preservatives to flavor concentrates and sweeteners, the circulation needs to attention. This study aims at analyzing the levels of saccharin and cyclamate at children elementary school's beverage at Karang tengah, Tangerang City. This study uses HPLC with a C18 column with mobile phase KH₂PO₄ and methanol (70:30), with UV-Vis detector for cyclamate at 200 nm wavelength and for saccharin at 227 nm wavelength. The results of this study showed that five samples containing saccharin (sample A = 73.059 mg/L, the sample B = 56.624 mg/L, the sample C = 0.73 mg/L, the sample D = 0.82 mg/L, the sample E = 61,52 mg/L), this figure was still below the recommended threshold BPOM RI. Whereas four of the sample including, containing cyclamate (sample A = 1184.42 mg/L, the sample B = 1409.95 mg/L, the sample D = 1418.31 mg/L, the sample E = 444.38 mg/L), it's levels exceeding BPOM RI's threshold recommended and one sample containing cyclamate whose levels do not exceed the threshold (sample C = 117.095 mg/L).

Keywords: Saccharin and Cyclamate, Sweeteners, Beverage Packaging

I. INTRODUCTION

Food additives, such as preservative, additive, and food coloring, were easily found in various food and beverage. Factory-based-industries used those additives for their products as well as home-based-industries. The safety of school's snacks had to be paid attention because it related to students' physical-growth. Food that was often become source of intoxication were snacks. This was due to lack of quality control from the manufacturer (that was often come from home-based-industry) (Adriani M, Wirjatmadi B, 2012).

Additive that was often used was artificial sweetener. It was processed chemically, and its compound was not found naturally (Anonym, 2012). It was purposely added to increase flavor, aroma and physical appearance of food or drink. Sweetener was divided into 2 (two): synthetic sweetener (cyclamate, aspartame, saccharin) and natural sweetener (sucrose, fructose, glucose, sorbitol).

Saccharin was artificial sweetener in form of salt, consisted of calcium, potassium and sodium saccharin. In general, saccharin salt was crystal white, odorless, soluble in water and very sweet (Head of National Agency of Drug and Food Control, 2014). Saccharin was 200-700 times sweeter than sucrose. Unfortunately, it left bitter or metal after taste in middle until high concentration. This was due to low purity as result from synthetic process. To remove the after taste, saccharin could be mixed with cyclamate by comparison of 1:10 for cyclamate (Putri Intan Eriska, 2013).

In 1971, a research conducted by Wisconsin Alumni Research Foundation (WARF) proved that saccharin classified as carcinogenic substance. From 15 rats which were given saccharin, 5% (or 7 rats among them) had cancer on gall bladder after consuming saccharin for 2 years (Djojosoebagio, Soewando dan G, Wiranda, 1996). In 1977, Canada's Health Protection Branch reported that saccharin was responsible as the cause of urine bladder cancer. Afterwards, the use of saccharin was banned in Canada, except as sweetener (Winarno F.G, 2008).

Initially, cyclamate was only use in drug industry. The purpose was to cover bitter taste from active drug substance, like in antibiotic and pentobarbital. After it was proclaimed safe on 1958, cyclamate was more popular as low-calorie artificial sweetener. However, research in 1969 showed that it could cause urine bladder cancer on mice that was given cyclamate regularly (Anonym, 1998). As result, United States withdrew its distribution from food industry totally on 1969 and continued by United Kingdom on 1970.

Cyclamate (C₆H₁₂NNaO₃S) was often found in form of calcium salt, potassium, and sodium cyclamate. Cyclamate was quite cheap additive, 30 to 50 times sweeter than sucrose. Unlike saccharine, cyclamate seldom leave bitter after taste like saccharin (Anonym, 2009). Cyclamate had 0 kJ/gor equal to 0 kJ/g and ADI: 0 mg/kg - 11 mg/kg per body weight. Based on regulation from Head of National Agency of Drug and Food Control (NADFC) No. 4 year 2014 regarding maximum limit of sweetener additives allowed in flavored drink, including

sport drink or electrolyte and particle drink, was 350 mg/kg (Head of National Agency of Drug and Food Control, 2014).

High Performance Liquid Chromatography (HPLC) was a chromatography method which could separate macro molecule, ionic compound, unstable natural products, polymeric compound and poly-functional groups with high mass molecule. It worked by fractional filtering, absorption or ion substitution that used interactive active phase and active solid/liquid passive phase (Ibrahim Slamet S, 1995). Its instruments consisted of: active phase's container, pump, tool to insert sample (injection container), column, detector, container to collect emission from active phase and a computer or integrator or recorder. Advantages of using this method were fast, well-separation power, sensitive to unique detector, column could be reused, ideal for big molecule and ion, and easy to reclaimed.

II. RESEARCH METHOD

A. Material and Instrument

Materials used in this research were plastic-cup-drink with P.I.R.T label (Product of Home-Industry), saccharin, cyclamate, potassium dehydrogen phosphate (KH₂PO₄), methanol, aquades.

Instruments used in this research included: Shimadzu SPD-20A as HPLC with C 18 (150 mm x 4.6 mm) of nucleosil column, analytic scale, whatman filter paper no. 42, volumetric flask, beakerglass, pipet volume.

B. Analysis Method

Drink sampling

Sampling method used in this research was purposive sampling; sample was taken intentionally depended on sample requirements needed. Researcher decided himself which sample taken due to particular consideration; sample must have P.I.R.T label (label for home-made industry product) and potentially contained saccharin or cyclamate. Drink with cup-package was chosen and five samples were taken.

1. Making of standard solution (British International Standard, 1997).

a. Making of saccharin standard solution 1000mg/l

Measure 0.0103 gram of saccharin. After that, pour it into 100 ml volumetric flask. Then dissolved it with KH₂PO₄solution – methanol until the border and homogeneous. 10 ml was taken from the 100 mg/l source solution. It was then dissolved and diluted into 100 ml volumetric flask with KH₂PO₄solution – methanol. Series of standard solution was then made; 10, 50, 100, 300, and 500 mg/l by pipetting 0.1 ml, 0.5 ml, 1.0 ml, 3.0 ml, and 5.0 ml solution. These standard solution series was then poured into 10 ml volumetric flask. Afterwards, it was dissolved with KH₂PO₄solution – methanol.

b. Making of cyclamate standard solution

Measure 0.0508 gram of cyclamate. After that, pour it into 50 ml volumetric flask. Then dissolved it with KH₂PO₄solution – methanol until the border and homogeneous. Series of standard solution was then

made; 10, 50, 100, 300, and 500 mg/l by pipetting 0.1 ml, 0.5 ml, 1.0 ml, 3.0 ml, and 5.0 ml solution. These standard solution series was then poured into 10 ml volumetric flask. Afterwards, it was dissolved with KH₂PO₄solution – methanol until the border and homogeneous.

2. Sample Preparation

Measure 2 gram of sample. After that KH₂PO₄solution and methanol were added until the border. It was then shaken until dissolved and homogeneous. After that, it was filtered by whatman filter paper no. 42. The solution was ready to be injected into HPLC (British International Standard, 1997).

3. Conditioning Instrument

a. Conditioning Instrument for cyclamate

HPLC's optimal condition for this experience was as follow : C-18 (150 mm x 4.6 mm) of nucleosil column, the flow speed was 1 ml/minute, 200 nm Ultraviolet detector, composition of active phase from potassium dehydrogen phosphate (KH₂PO₄) - methanol was (70:30) with 20 µl injection volume.

b. Conditioning Instrument for saccharin

HPLC's optimal condition for this experience was as follow : C-18 (150 mm x 4.6 mm) of nucleosil column, the flow speed was 1 ml/minute, 220 nm Ultraviolet detector, composition of active phase from potassium dehydrogen phosphate (KH₂PO₄) - methanol was (70:30) with 20 µl injection volume.

c. Determining level of synthetic additives (saccharin and cyclamate)

Sample, which had been diluted by KH₂PO₄solution – methanol, was taken for the amount of 5 µl. It was then injected into HPLC. The area obtained from the result was recorded, and calculated using calibration curve from each element. Concentration of saccharin and cyclamate synthetic additives was calculated using standard curve by linear regression equation as follow (Winarno F.G, 1997):

$$y = a + bx$$

whereas: y = area's width

x = sample's concentration

a = intercept

b = slope

While level of synthetic additives could be calculated using formula as follow (Winarno F.G, 1997) :

$$\text{Synthetic_Additives_Level} = \frac{C_{\text{sample}} \times \text{Dilution_factor}}{W_s} = \text{ppm}$$

Note: C_{sample} = concentration of sample

W_s = weight of sample

III. RESULT AND DISCUSSION

A. Conditioning HPLC Instrument

Conditioning instrument in this research was conducted with the objective to find optimal condition for HPLC

method. Cyclamate optimal conditioned obtained from this research was C-18 (150 mm x 4.6 mm) column, flow speed 1 ml/minute, 200 nm Ultraviolet detector, and 70:30 composition of active phase from KH₂PO₄-methanol. Saccharin optimal conditioned obtained from this research was C-18 (150 mm x 4.6 mm) column, flow speed 1 ml/minute, 220 nm Ultraviolet detector, and 70:30 composition of active phase from KH₂PO₄-methanol.

We could differentiate between saccharin and cyclamate with instrument's condition stated above. Separation between saccharin and cyclamate could be seen from its retention period. Retention period was time needed after sample was injected to bring out an analit peak by detector (IbrahimSlamet S, 1995).

Sample Preparation

Sample preparation was conducted by dissolving sample with the fit solvent and conducting filtration (Ibrahim Slamet S, 1995). The solvent used was KH₂PO₄ - methanol (70:30). Meanwhile the filtration process was using whatman filter paper no. 42 so that sample would still be in clear condition.

Determining Calibration Curve

Correlation coefficient value (r) was a quality indicator from linier parameter which described proportionality of analytic respond (area width) towards the measured concentration. Data of area width generated from saccharin standard series (with concentration of 0.101; 0.505; 1.009; 3.028; 5.047 µg/ml) showed that it had linier relation with correlation coefficient value (r = 0.9999987) by using linier regression equation; $y = 69020.9 x - 112.852$. The coefficient obtained showed good result (close to 1 score). This informed that there was proportional relation between area width and concentration measured. Result from linier test of saccharin calibration curve could be found in Figure 1:

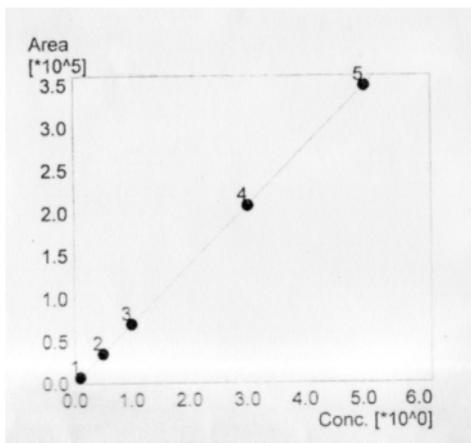


Fig. 1 Saccharin calibration curve

From series of saccharin concentration (9.957; 49.784; 99.568; 298.704; 497.840 µg/ml), correlation coefficient value (r) was obtained (r = 0.9999088)

using linier regression equation; $y = 929.899 x - 1479.25$. The coefficient value obtained revealed good relation between concentration and area width (because the score was close to 1). The linier test result from cyclamate calibration curve was shown in Figure 2:

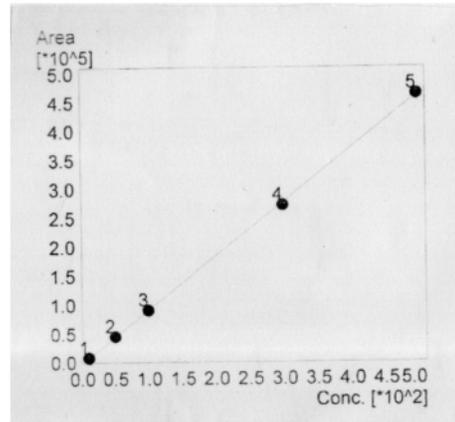


Fig. 2 Cyclamate calibration curve

B. Analysis Result of Saccharin and Cyclamate Content

Based on linier regression calibration curve obtained from saccharin and cyclamate on Picture 1 and 2 above, saccharin and cyclamate level content was found in various amounts within all five samples. All five samples tested positively contained cyclamate. One sample showed safe level of cyclamate as determined by NADFC, while the rest four samples had cross it. Those five samples also positively contained saccharin in safe level.

The analysis result was shown on table below:

Table 1 :Analysis result towards saccharin and cyclamate content in drink sample

Sample	Saccharin		Cyclamate	
	Curve's width (ppm)	Level (mg/L)	Curve's width (ppm)	Level (mg/L)
A	0.735	73.059	47.704	1184.422
B	0.575	56.624	57.447	1409.954
C	0.030	0.73	4.754	117.095
D	0.0083	0.82	59.289	1418.306
E	0.615	61.522	17.854	444.385

This research was conducted to test whether cup-package-drink with P.I.R.T (Home-Made Industry Product) label contained synthetic additives, such as saccharin and cyclamate. Based on result from the research, the five samples contained saccharin with concentration between 0 – 73.059 ppm, while cyclamate's concentration ranged between 0 – 1418.306 ppm. After calculating the content level, saccharin found in all five samples didn't exceed safe

level of 120 mg/L as determined by NAFDC's regulation No. 4 Year 2014. The highest found only contained 73.059 ppm saccharin. However, the cyclamate found in all five samples range between < 2.00 mg/L – 1418.306 mg/L, whereas safe level of cyclamate determined by NAFDC must not exceed 350 mg/L.

The sweet taste of every synthetic additive was always evaluated towards its relativity with the sweet taste from sucrose. Cyclamate was 30 – 50 time sweeter than sucrose, while saccharin was 200 – 700 times sweeter. Due to its strong sweet taste, the use of synthetic additive was thought to be more efficient than natural additive (Wisnu C, 2006).

The use of cyclamate had been banned in United States. However in Indonesia, it wasn't forbid and could be used with certain daily intake which had been determined for each food and drink substance. Considering the danger that might cause by this synthetic additive, supervision and regulation towards the use of synthetic additives for commercial food and drink needed to be stricter.

Cyclamate from the analysis result had exceeded safe level determined by NADFC. This could cause negative effect for children in the future. This issue needed to be anticipated. Government in general and NADFC in particular, as authorized institution needed to warn manufacturer which already P.I.R.T label but still used cyclamate.

CONCLUSION

Based on research towards saccharin and cyclamate inside commercial cup-drink with P.I.R.T (Home-made industry product) label using HPLC method, it can be concluded that :

1. All five samples positively contained saccharin and cyclamate
2. Saccharin levels in all five samples were still in safe level as determined by NADFC. However, cyclamate level in four samples had exceeded limit set by NADFC, while one sample was still in safe level.

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