

DIKTAT METODOLOGI PENELITIAN



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**PROGRAM STUDI TEKNIK KIMIA
FAKULTAS TEKNOLOGI INDUSTRI
UNIVERSITAS AHMAD DAHLAN
YOGYAKARTA**

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KATA PENGANTAR



Puji syukur penulis ucapkan ke hadapan Allah SWT karena berkat rahmat, hidayah, dan karuniaNya penulis dapat menyelesaikan Diktat untuk Mata Kuliah **Metodologi Penelitian** untuk Program Studi Teknik Kimia edisi pertama ini. Diktat ini dapat digunakan untuk mahasiswa strata S1 dan S2.

Tujuan penulisan diktat kuliah ini adalah untuk memberikan pemahaman, pengetahuan tentang identifikasi permasalahan penelitian, penyusunan proposal penelitian, menyusun laporan penelitian dan menyusun paper ilmiah. Oleh karena itu, diharapkan setelah membaca dan mempelajari diktat ini mahasiswa dapat menentukan langkah-langkah melakukan penelitian, dapat menyusun proposal penelitian dan dapat menyusun paper publikasi ilmiah.

Ucapan terima kasih penulis sampaikan kepada semua pihak yang telah membantu penulis dalam menyelesaikan penulisan diktat ini. Akhirnya sumbang saran sangat penulis harapkan demi kesempurnaan diktat ini. Semoga diktat ini bermanfaat bagi seluruh pembaca, khususnya mahasiswa.

Yogyakarta, September 2021

Penulis

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BAB I. PENDAHULUAN

I.1. Skripsi S1, Tesis S2 dan Disertasi S3

1. Persamaan Skripsi, Tesis dan Disertasi

- a. Persamaan Skripsi, Tesis, dan Disertasi adalah bahwa ketiganya sama-sama merupakan Karya Tulis Ilmiah sehingga penyusunannya harus memenuhi metode ilmiah dan nilai-nilai kemanusiaan, baik redaksi maupun substansi.
- b. Ditulis oleh seorang mahasiswa, bukan kelompok.
- c. Semuanya harus dipublikasikan baik secara off-line maupun online sesuai level yang telah ditentukan.

2. Perbedaan Skripsi, Tesis dan Disertasi

- a. Hanyalah berupa pembuktian bahwa mahasiswa mampu menyelesaikan sebuah masalah dengan mengimplementasikan pengetahuan dan ketrampilan yang telah dipelajarinya selama kuliah S1.

Melalui Skripsi yang dibuatnya mahasiswa menunjukkan bahwa ia mampu berfikir logis, kritis, dan sistematis memanfaatkan akumulasi pengetahuan dan ketrampilan yang telah dipelajarinya selama kuliah S1 untuk mengidentifikasi opsi-opsi solusi sebuah masalah dan/atau peluang dan mengimplementasikannya atau mengkaji implikasi dari pengembangan/implementasi pengetahuan atau teknologi. Jadi kata kunci level Skripsi adalah Implementasi/Penerapan.

- b. Secara eksplisit dikatakan bahwa standar minimal Skripsi S1 Bukan Riset, namun cukup Implementasi suatu ilmu/metode/teknik/teori/ketrampilan tertentu secara benar untuk menyelesaikan suatu masalah.

Dengan demikian dalam Laporan Skripsi di bagian “Perumusan Masalah” seharusnya berisi pernyataan Masalah apa yang akan diselesaikan melalui Tugas Akhir (Skripsi) tersebut (Bukan Pertanyaan Penelitian atau Research Question).

Apakah tugas akhir mahasiswa s1 tidak boleh berupa riset/penelitian?.
KKNI hanyalah memberikan standar minimal sehingga apabila ada sebuah program studi mewajibkan standar skripsi mahasiswa S1 harus lebih sulit lagi yakni berupa riset/penelitian bahkan wajib melakukan publikasi internasional ya boleh-boleh saja, hanya saja layak difahami bahwa sistem tersebut melebihi standar KKNI level 6.

- c. Pendekatan Skripsi dapat berupa Mono Disiplin, yakni 1 disiplin ilmu saja, misal: Skripsi berjudul “Rancang Bangun Sistem Informasi Rumah Sakit” dapat saja dilakukan dengan hanya mengacu ke prinsip-prinsip disiplin ilmu Rekayasa Perangkat Lunak dengan informasi proses bisnis Rumah Sakit diperoleh hanya melalui proses wawancara (tidak ada kajian atau validasi dari teori atau referensi disiplin ilmu lain yakni Manajemen Kesehatan).

3. Tesis

Berupa Riset/Penelitian sehingga mahasiswa S2 wajib menguasai dan menerapkan berbagai aspek dan ketrampilan Metode Penelitian yang berlaku di disiplin ilmunya.

Mengembangkan pengetahuan/teknologi/seni menghasilkan Karya yang **Inovatif** dan **Teruji**. Terdapat 3 kata kunci di sini, yakni: “Mengembangkan” pengetahuan/teknologi/seni yang sudah ada sebelumnya sehingga menghasilkan luaran yang “Inovatif” yakni sesuatu yang baru hasil dari pengembangan yang sudah ada dan sudah melalui proses uji-coba atau validasi.

a) **Dari pengertian ini maka Laporan Tesis harus mampu:**

a. Menunjukkan **pengetahuan (teori/teknologi) apa saja yang telah ada?**

yang umumnya dilakukan melalui review banyak penelitian/teori/teknologi sebelumnya hingga terkini.

b. Dari review penelitian/teori/teknologi sebelumnya tersebut selanjutnya mahasiswa S2 dituntut mampu melakukan *Systematic Mapping Study (SMS)* sehingga dalam Tesis tersebut mahasiswa mampu:

- **memposisikan** penelitiannya dalam **suatu peta penelitian** yang dikembangkan melalui pendekatan interdisiplin atau multidisiplin, artinya mahasiswa mampu menunjukkan posisi penelitiannya dalam diagram peta disiplin ilmu, obyek dan/atau aspek penelitian.
- **mengidentifikasi *Knowledge Gap*** (*peluang-peluang aspek-aspek topik penelitian yang belum dikaji peneliti-peneliti sebelumnya*) apa yang ingin diisi/dibuat/ditambahkan.

Tesis menggunakan pendekatan Multidisiplin atau Interdisiplin. Berdasarkan Permendikbud No. 154 Tahun 2014 bahwa multidisiplin artinya penelitian dilakukan dengan melibatkan minimal 2 disiplin akademik untuk menyelesaikan suatu masalah secara bersama-sama. Dalam pendekatan multidisiplin ini, analisis

melibatkan (mengadopsi) perspektif teori/penelitian dari disiplin akademik lain namun masing-masing spesialis tetap berada di dalam disiplinnya.

Sementara Interdisiplin artinya penelitian dilakukan dengan transfer (mengadopsi dan mengadaptasi) suatu disiplin akademik ke dalam disiplin akademik lainnya untuk menyelesaikan masalah tertentu sehingga mampu memunculkan metode baru atau disiplin akademik baru.

Dengan demikian level minimal kompleksitas Tesis adalah Mengembangkan Pengetahuan/Teknologi yang sudah ada sebelumnya dengan Metode Penelitian tertentu dan Pendekatan Multidisiplin atau Interdisiplin.

I.2. Penelitian Ilmiah

Penelitian adalah mencari pemecahan suatu masalah yang belum diketahui penanggulangannya. Penelitian yang dilakukan adalah:

- a. Hasil penelitian yang baru/asli, atau bahan baku yang baru atau alatnya yang baru
- b. Tindakan berdasarkan moral yang baik, bukan coba- coba, dan sistematis
- c. Masalah atau topik penelitian dapat berupa:
 - Pasif: permasalahan berupa pesanan (tinggal mengerjakan)
 - Aktif: dilakukan dengan identifikasi masalah (mencari masalah), kemudian dilakukan perumusan.

Metologi penelitian diperlukan karena kita masih memerlukan penelitian – penelitian ilmiah yang kreatif:

- a. Untuk pemanfaatan sumber daya alam

- b. Untuk memperbaiki proses – proses yang ada (walaupun teknologi aslinya “beli” dari negara lain), untuk mengurangi ketergantungan pada teknologi “beli”.

Contoh:

1. Kasus di pabrik kertas Leces

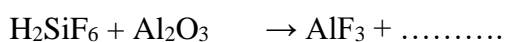
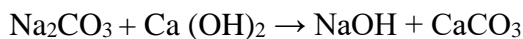
Cairan hitam mempunyai komponen:

- Na Lignat, biasanya dibakar
- NaOH
- H₂O
- $\text{Na}_2\text{SiO}_3 + \text{CO}_2 + \text{H}_2\text{O} \rightarrow \text{Na}_2\text{CO}_3 + \text{SiO}_2$

Cairan hitam ingin dimanfaatkan sebagai bahan bakar. Terbentuknya Na₂CO₃ direaksikan dengan Ca (OH)₂ menjadi NaOH sehingga bisa dipungut kembali NaOH nya (digunakan untuk hidrolisis CaCO₃ lagi).

Kendala yang timbul adalah terbentuknya SiO₂ yang menyumbat pipa – pipa. Rangkaian alat – alat untuk menghilangkan SiO₂ (dari Lisensi) tidak bekerja dengan baik. Dari penelitian diperoleh sistem yang baru, tanpa pipa berkelok – kelok, diusahakan terjadi pengolahan pengadukan cairan hitam dengan kolom berpenghalang dan hasilnya lebih baik.

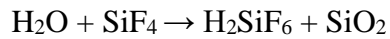
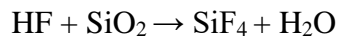
Lanjutan:



↓ untuk ekstraksi

Kasus di petrokimia Gresik (pupuk fosfat dari batuan fosfat dan asam sulfat).

Hasil samping: SiO_2



Masalah:

Pada pipa pengeluaran gas reaksi berlanjut dan terbentuk SiO_2 akan menyumbat.

Sebagai alternative penyelesaian dibuat penghalang dalam kolom.

2. Kasus di Pertamina Balongan (unit *catalytic cracking*)

Permasalahannya adalah pembuangan katalisator yang merupakan limbah B3.

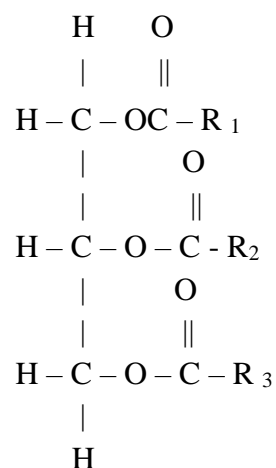
Kemungkinan pemecahan:

- Dijadikan campuran semen / batako
- Memperbaiki katalisator (mungkin fludisasi kurang merata).

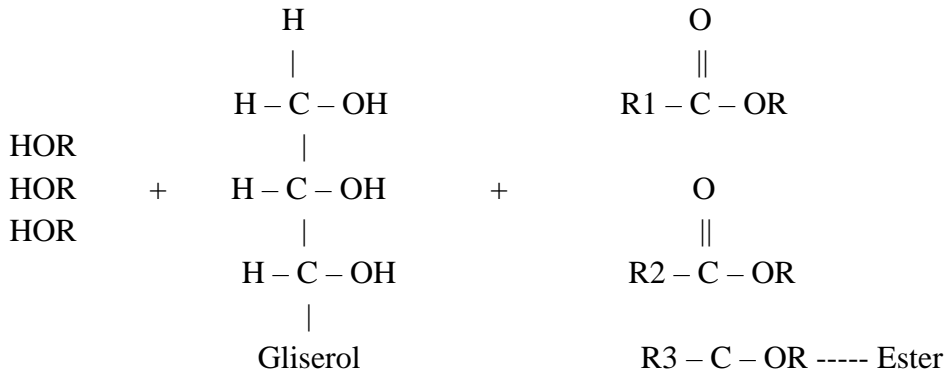
3. Biji karet, dikempa diambil minyaknya sebagai minyak diesel (bio disel).

Kekentalan minyak karet sangat tinggi sehingga pengkabutan menyulitkan pembakaran, perlu penurunan kekentalan.

Minyak lemak:



Dengan cara Alkoholis → ester



Ester mendekati sifat fisis minyak pelumas: $R > C17$

I.3. Penggolongan penelitian dalam teknik kimia berdasar tujuannya

1. Penelitian Eksploratif

Adalah penelitian mengenai mekanisme reaksi, reaktivitas bahan kimia, mengenal struktur bahan, usaha-usaha pembuatan sesuatu bahan kimia.

2. Penelitian Fundamental

Adalah penelitian untuk mencari data-data maupun persamaan yang berlaku luas yang bisa dipakai dalam perhitungan *Chemical Engeneering Tools* nomor 1 sampai 4.

Yakni:

1. Neraca massa
2. Neraca energi
3. Kesetimbangan
4. Proses-proses kecepatan

3. Penelitian Proses

Adalah penelitian yang bertujuan: mencari kondisi proses yang optimal secara komersil, mencari data untuk pra-evaluasi ekonomi dan untuk perancangan pilot plant/pabrik.

I.4. Faktor – faktor pendukung

1. Manusia:

- jumlah
- kemampuan (kecerdasan)
- pendidikan
- pengalaman
- keuletan

2. Sarana / Prasarana

Prasarana: laboratorium, listrik, air, gas, (infrastruktur)

Sarana: alat - percobaan

- analisis

bahan - bahan baku

- analisa

pustaka - majalah

- PDII (LIPI)

- Pusat Dokumentasi dan Informasi Indonesia

3. Dana

Sumber: - sendiri

- swasta

- pemerintah

Jumlah - PUNAS RISTEK



SUB SEKTOR



PROGRAM

- a. Teknik Reproduksi
- b. Teknologi
- c. Ilmu Pengetahuan Terapan
- d. Ilmu Pengetahuan Dasar
- e. Pembinaan Kelembagaan IPTEK
- f. Pengembangan Prasarana & sarana ilmu Pengetahuan.
- g. Pengembangan Sistem Informasi
- h. Penyempurnaan & Pengembangan Strategi

4. Waktu

Yang tersedia: berusaha tepat waktu, harus mengigit Tri Dharma PT (alokasi waktu), ruang lingkup disesuaikan

5. Etika

Berpegangan pada "*program paying*" Perguruan Tinggi, mencakup banyak bidang dan bidang ilmu.

Contoh: seseorang dari bidang proses tidak akan melakukan penelitian bidang OTK, sedapat mungkin mengembangkan bidang sendiri.

I.5. Langkah-langkah Penelitian

1. Identifikasi dan perumusan masalah serta menemukan masalah
2. Keterangan penunjang: - pengumpulan data
 - pengolahan
3. Menyusun landasan teori dan dijabarkan sendiri.
4. Hipotesis (berdasarkan landasan teori).
5. Menyusun rencana (usul) penelitian: diseminarkan (sebaiknya setelah perencanaan dahulu).
6. Pelaksanaan:
 - penyiapan bahan
 - pembuatan alat
 - jalan penelitian: a. percobaan pendahuluan.
 - b. pengumpulan data.
 - analisis hasil
7. Pengolahan data: - daftar (table)
 - grafik
 - persamaan
8. Pembuatan laporan.
9. Pembuatan makalah untuk: - majalah
 - seminar

BAB II. PERUMUSAN MASALAH

II.1. Identifikasi masalah

Sumber:

1. Ilmu Pengetahuan: katalisator dan enzim.
2. Masyarakat:
 - a. Pangan : jumlah dan pengolahan hasil
 - b. Sandang : bahan (kapas, sutera, wol, sintetik), alat, cara.
 - c. Papan : bentuk (indah, efisien, hemat energi, kokoh), bahan (alam, awet, buatan), alat dan cara.
 - d. Energi : - efisien
- baru
- sumber terbarukan (air, angin, surya, biomassa)
 1. padat: kayu, grajen, arang
 2. cair: alkohol dan minyak nabati.
 3. gas: biogas, produser.

Contoh kasus:

1. Katalisator di Pertamina Balongan

- Setiap hari membuang berton-ton katalisator yang mengandung vanadium dan nikel.
- Termasuk limbah B3 dan pemborosan.
- Dikirim untuk diolah lebih lanjut di semen Cibinong (mahal).
- Perlu dicari kemungkinan penggunaan katalisator lain.

Gambar macam-macam katalis yang dipakai di industri pengolahan minyak bumi dapat dilihat berikut ini.



Gambar 1. Macam-macam katalis padat

2. Masalah kekurangan gizi Tahun 60-an

1. Kadar protein dalam makanan kurang sehingga banyak penyakit busung lapar.
2. Penelitian untuk meningkatkan kadar protein dalam makanan:
 - a. Masalah pangan secara ekstensif: masalah pengadaan lahan.
 - b. Masalah pangan secara intensif: pengolahan tanah
3. Pemupukan sebaiknya menggunakan pupuk organik untuk mempertahankan struktur tanah (tetap berongga-rongga) dengan pupuk sintesis maka akan terjadi pemadatan tanah.

3. Pupuk SP 36 di Petrokimia Gresik

Dulu TSP dimodifikasi menjadi SP 36 (kadar P_2O_5 senyawa yang mudah larut hanya 36 %, agar tidak banyak pupuk yang hilang).

Proses sama:

- TSP: umpan as fosfat + bahan fosfat.
- SP 36: umpan as fosfat + bahan fosfat + asam sulfat.

Masalah: Setelah switch ke SP 36, banyak debu terbawa gas keluar.

Penyebab: Adanya H_2SO_4 menyebabkan kecepatan pembentukan gas lebih cepat padahal alat yang ada dirancang untuk flow rate gas < (reaksi H_3PO_4 maka as lemah dengan batuan fosfat lebih lambat).

4. Protein Nabati

- 1) Kara benguk: kadar protein cukup tinggi tapi kandungan HCN tinggi. Dari kara benguk ini bisa dibuat protein konsentrat. Protein konsentrat sebaiknya dibuat dari bahan-bahan yang tidak semuanya bisa dikonsumsi.

Contoh:

- a. Ampas biji karet → kadar protein >> tapi mengandung alkaloid.
- b. Membuat protein konsentrat dari kedelai tidak menguntungkan karena seluruh kedelai bisa dikonsumsi (tidak perlu mengambil proteinnya saja).



Gambar 2. Korobenguk dan produk olahan

- 2) Tempe: protein rantai panjang dipecah dengan proses fermentasi sehingga mudah dicerna.

5. Pengawet

- 1) Pengeringan: mengurangi kadar air agar mikro organisme tidak bisa hidup.
- 2) Penambahan N_2 atau CO_2 biasanya dipakai untuk ekspor sayuran.



Gambar 3. Berbagai Macam Cara Pengawetan makanan

6. Bahan Sandang

- 1) Kapas: kadar selulosa sudah tinggi, tinggal dipintal saja.
- 2) Serat Linen: dari kulit tumbuhan dan perlu “dilepaskan” dari pengikatnya dengan cara hidrolisis.
- 3) Sutera: masalah sutera Indonesia, kaku dan sulit diwarnai.
- 4) Sintetik: serat rayon, selulosa dilarutkan dalam CS_2 (larut viscose) lalu disemburkan melalui lubang-lubang kecil (spinnertte) kelarutan yang mengandung asam → berubah menjadi selulosa padat lagi → ditarik sehingga menjadi serat.



Gambar 4. Berbagai macam serat untuk sandang

7. Bahan Bangunan

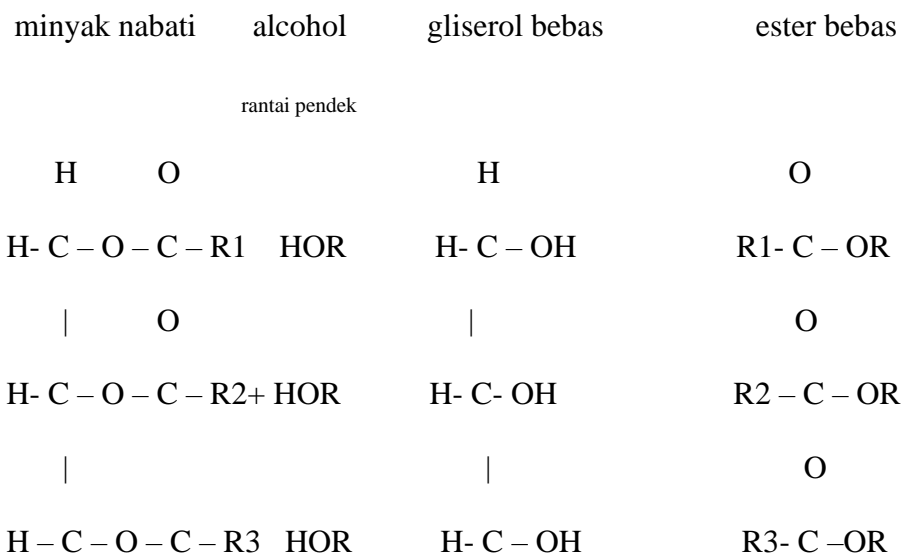
- Kayu: perlu diberi zat pengawet.
- Semen: - kapur, tanah liat, dan silika.

Semua tidak terbarukan, perlu dipikirkan alternative lain, masalahnya semen daur ulang dari gempuran bangunan lama).

- Padas: buat padas sintesis dari campuran abu bebas kalium dengan semen.

8. Energi:

1. Efisiensi: hemat, pakai seperlunya.
2. Sumber baru: minyak nabati (tidak berjelaga tapi kekentalannya masih terlalu tinggi)



R₁, R₂, R₃ adalah rantai-rantai panjang maka diikat oleh gliserol, jadi kekentalannya sangat tinggi, setelah dipecah menjadi gliserol dan ester maka

cabang-cabang berkurang sehingga viskositas turun dan bisa dikabutkan dengan baik. Tapi nilai kalor ester masih rendah karena masih banyak C-O nya.

Kemungkinan:

CO₂ dikeluarkan sehingga diperoleh hidrokarbon. Dalam hasil mungkin masih ada ikatan rangkap, maka perlu dijernihkan lagi.

- Panas bumi: bisa dimanfaatkan untuk membangkitkan kukus
- Air : dengan kincir.
- Angin : untuk membantu petani garam.
- Surya : untuk pengeringan, membuat air panas, membuat air tawar dari air payau/air asin.

9. Sel Surya: menghasilkan arus listrik searah, ada penelitian (usul) untuk membuat silicon yang akan digunakan dalam surya dari tanah bentonit. Tidak layak karena pengotor dalam tanah terlalu banyak sehingga pemurniaannya sulit, lebih baik pakai salah satu limbah petrokimia Gresik dari pabrik SP- 36 (SiO₂)

10. Bahan bakar dari Biomassa

Arang: Menghasilkan gas CO dioksidasi menjadi CO₂ (gasifikasi) menghasilkan kalor cukup besar

Grajen (sekam padi): Bila langsung dibakar nyalanya hanya sebentar dan transfer panas sangat lambat (hanya bara saja) karena udara tidak bisa bersinggungan dengan grajen dibagian dalam.

Pemecahan:

1. Pakai kisi-kisi.

2. Serbuk dicetak menjadi batangan-batangan tidak dengan perekat, sebagian dipirolisis sehingga terikat oleh tar yang dihasilkan, menghasilkan batangan-batangan lalu dipirolisis dengan penampang.
3. Plastik bungkus, jika didaur ulang kurang baik karena pengotornya sudah terlalu banyak. Dipirolisis, menghasilkan minyak hidrokarbon yang kualitasnya cukup baik.
4. Alkohol, di Brazil sudah dicoba menggunakan alkohol sebagai bahan bakar.

Keuntungan: tanpa jelaga, nyala biru.

Kelemahan:

- kerosif, karena dalam alkohol kandungan air masih $\pm 4,5$ %.
 - nilai kalor/berat masih lebih rendah (karena ada O yang tidak bisa terbakar) untuk mengeluarkan oksigen maka digunakan zeolit khusus untuk mengeluarkan air.
5. Biogas, untuk keperluan rumah tangga biasanya diperoleh dari kotoran ternak

Problem: PH turun sehingga bakteri mati, umpan dicampur dengan abu.

Gas prodeser (campuran CO, CO₂, H₂O, metan) dari kayu yang dioksidasi parsial



Gambar 5. Berbagai macam biomassa

11. Lingkungan

Untuk: a. Penyaman: penyejuk tv, radio, alat rumah tangga.

b. Limbah:

1. Padat: sampah, grajen.

2. Cair:

- organik (cairan hitam, cairan tapioka, panyamakan, tekstil)
- anorganik (penyepuhan, cat, cuci + cetak foto).

3. Gas: cerobong asap, debu, kendaraan.

12. Sampah Kota

Di Singapura, dipakai untuk membangkitkan tenaga listrik untuk menghindari bau busuk, udara diruang penimbunan sampah ditarik dan digunakan untuk udara pembakaran (membangkitkan kukus dan pembangkit listrik)

Abu, logam diambil dan sisanya untuk menimbun tanah.



Gambar 6. Timbunan Sampah

Di Indonesia, sampah difermentasi menjadi pupuk (kompos) dan dibakar dalam incenarator.

13. Limbah cair

1. Cairan hitam dari pabrik kertas, didaur ulang, lignin untuk bahan bakar.
2. Tapioka: cairan keruh putih (white water), karbohidrat dimakan oleh mikro organisme akan menghabiskan O_2 dalam air (ikan akan mati), ketela mukibat: kadar HCN tinggi (racun).

Penyelesaian: digumpalkan dengan tawas+Ca (OH)₂ dan digelembungkan udara agar CN terikat sebagai Ca (CN)₂

- cairan difermentasi menghasilkan CO dan CO₂.

3. Penyamakan kulit dengan penyamak khrom. Dalam limbah terdapat protein, zat organik lain, dan khrom kemungkinan penyelesaian: reaksi dengan Ca (OH)₂ menjadi endapan CaCr digelembungkan udara untuk menurunkan BOD dan COD

4. Limbah pemurnian emas, larutkan HNO_3 yang mengandung Cu dielektrolisis
5. Limbah cetak Foto: $\text{AgBr} \rightarrow \text{Ag}$ cukup banyak, diambil dengan elektrolisis memakai elektroda Cu.
6. Gas pembakaran dari kendaraan bermotor CO, CO_2 dipakai katalitor untuk $\text{CO} \rightarrow \text{CO}_2$
7. Transportasi
 - a. Darat: mobil dan sepeda motor, kereta api, sepeda, suku cadang, bahan baker dan pelumas.
 - b. Laut: kapal, perahu, suku cadang.
 - c. Udara: jenis kendaraan, penunjang.

Unit penyerapan H_2S dari gas alam (di Pusri)

- a) Dilakukan dengan oksidasi besi yang diserap dalam sisa pasahan kayu \rightarrow tekanan pemakaian ringan dan luas permukaannya
- b) Agar oksid bisa bisa masuk kecelah-celah kayu
 - serpihan kayu direndam dalam larutan teri khlorid.
 - Direaksikan dengan $\text{NH}_4 \text{OH} \rightarrow$ terbentuk ferihidroksid.
 - Dipanaskan sehingga terbentuk Fe_2O_3

II.2. Penghasil dana, bagaimana menghasilkan dana

Pemilihan:

1. Sumber dana
2. Panas Ristek
3. Kemampuan pelaksana

4. Prasarana dan sarana
5. Waktu
6. Pustaka
7. Pembimbing

BAB III. KETERANGAN PENUNJANG

III.1. Sumber Keterangan

1. Umum dan relative tua (± 2 thn sebelum terbit)

Dapat berupa:

- a. Ensiklopedia karena umur, susunan menurut abjad, jadi mudah ditelusuri dan ada acuan sumber pustakanya.
- b. Buku teks teori umum
- c. Monografi
- d. Majalah tua mengembangkan informasi yang telah diperoleh.

Beda buku Ensiklopedia dan buku teks adalah bab-bab dibuku teks terbatas dan tidak disusun menurut abjad sehingga melihat lihat indeks atau daftar isi saja.

Monografi adalah buku yang khusus membicarakan satu topik. Majalah adalah menyajikan hasil penelitian di laboratorium dan keterangan jauh lebih terinci serta teori lebih mendalam dan spesifik.

2. Baru

Abstrak: menyingkat waktu penelusuran karena disusun menurut bidangnya, penyediaan keterangan sumber pustaka (majalah / paten).

chemical abstrak

Engineering Index

↓

↓

rujukan paten

rujukan majalah

↓

↓

photo kopi

majalah

(bisa melalui PDII-LIPI) atau meminta ke Biro Paten Negara yang bersangkutan.

3. Tambahan Sumber Keterangan

- Hand book
- *Interen Critical table*
- Brosur
- iklan

Contoh:

Ensiklopedia.

1. Kirk, RE and Other, DF, 1953," Encylopedia of Chemical Tecnologi
2. Heilbron, SI, 1950, "Thope's dictionary Of Applied Chemistry.
3. Ullman, R, 1980, "Enzyklopadie der Tecnischen chemie"

Buku Teks: Groggins

Monografi: Hou, TP, 1966," Manufakturing of Soda "

Majalah:

1. Ind. Eng. Prog: a. Fundom; Ind. Eng. Chem. Fundom.
 - b. Proses Des. Dev.: Ind. Chem. Proses Des. Dev.
 - c. Prod. Res. Dev: Ind. Eng. Prod. Res. Dev.
2. AICHE Journal
3. Chem. Eng. Prog.: Chemical Eng. Progres
4. Chem. Eng
5. Chem. Eng. Sci (Inggris)
6. Can. J. Chem. Eng: The Canadian Journal of Chem. Eng.
7. Chem. Eng. Data: Journal of Chem. & Eng. Data
8. J. Sci. Industri. Res. (India)

add 1 dan 3: Diterbitkan oleh AICHE tapi Chem. Eng. Prog. disajikan lebih populer dan tidak banyak memuat hasil penelitian.

add 4: Diterbitkan oleh Mc. Graw. Hill

ditujukan pada para praktisi industri kimia.

III.2. Tempat Informasi

1. PDII (Pusat Dokumentasi & Informasi Ilmiah) LIPI
2. Perpustakaan Universitas / Lembaga Penelitian
3. Luar Negeri

Keterangan yang diperlukan:

1. Bahan baku
2. Alat
3. Cara
4. Variabel
5. Hasil

III.3. Pencatatan Keterangan

Catatan singkat:

Tempat: Lembar dengan snelechter (1 lembar untuk 1 judul).

Yang dicatat:

1. Penulis, Tahun, Judul, Nama majalah/buku, dll untuk keperluan menyusun daftar pustaka
2. Cara penelitian, hasil, dan pembahasan (bahan baku, alat, cara, variable, hasil, dll)

III.4. Pengolahan Keterangan

Pengelompokan menurut jenis:

1. bahan
2. alat
3. cara
4. variable
5. hasil

III.5. Pembuatan Usul Penelitian

Usul penelitian berisi: judul, latar belakang masalah, tinjauan pustaka, landasan teori jika ada, hipotesis, cara penelitian, jadwal penelitian dan daftar pustaka

1. Judul

-Singkat dan Tepat

-Peneliti dan identitas (no Mahasiswa)

contoh: "Pemanfaatan sampah Kota "tidak mengarah pada suatu cara tertentu.

Lebih tepat: "Pirolisis sampah kota secara sinambung ", tidak perlu menambahkan "dengan udara dan aliran lawan arah.

Untuk Program S2: Penekanan pada sisi ilmiah dan pembangunan Negara.

2. Latar Belakang

- 1) Permasalahan yang mendorong perlunya penelitian dilakukan, pengantar indentifikasi dan perumusan masalah

Contoh: Jumlah sampah kota makin meningkat (dilengkapi dengan data kuantitatif). Asalnya dari mana susunanya, penangananya, bagaimana kemampuan armada, pembusukan, pengolahan dibiarkan jadi kompos.

Masalah yang mungkin timbul: bau, binatang, bakteri, air, tanah.

Kemungkinan Pemecahannya:

Pirolisis dalam waktu singkat, sampah dapat dijadikan arang (30% vol semula) yang tidak membusuk.

- 2) Keaslian perlu ditunjukkan bahwa sepanjang penelusuran pustaka belum pernah ada penelitian sejenis
- 3) Faedah untuk membangun dan untuk ilmu pengetahuan dalam bidang teknik mencakup keduanya.

Pembuatan kalium Sulfat dari Gips dan abu.

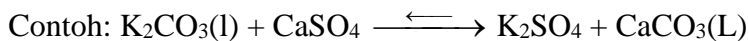
Permasalahannya: Pupuk kalium hampir seluruhnya diimpor dalam bentuk KCl dan K_2CO_3 tidak dipakai karena reaksinya dalam air bersifat basa (KCl dan K_2SO_4) netral. Sumber Kalium di alam:

- 1) Batuan atau mineral tidak ada di Indonesia
- 2) Tumbuhan di Indonesia banyak limbah pertanian yang belum dimanfaatkan.
- 3) Sumber Sulfat yang murah: Gips

Gips + Eksak abu: kalium Sulfat + kalium Karbonat

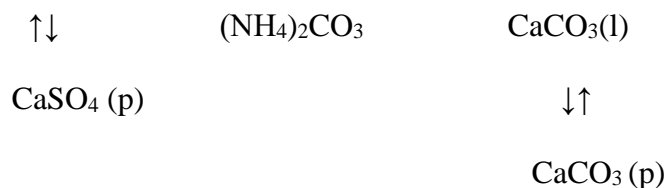
2. Tinjauan Pustaka

1. Keterangan penunjang dari pustaka atau majalah

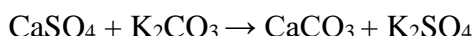


Keterangan Penunjang yang diperlukan:

Reaksi sejenis: $\text{CaSO}_4(\text{l}) + \text{NH}_3 + \text{CO}_2 + \text{H}_2\text{O} \rightleftharpoons (\text{NH}_4)_2\text{SO}_4 +$



Berhasil baik sehingga bisa dikatakan bahwa reaksi $\text{CaSO}_4 + \text{K}_2\text{CO}_3$ punya prospek baik.



2. Uraian disusun secara sistematis, mulai dari yang umum sampai yang dekat sekali.
3. Dalam uraian, sumber pustaka disebutkan: Dimunculkan bila kita mencantumkan jumlah atau metode. Sumber pustaka langsung disebutkan setelah informasi tersebut tidak perlu menunggu selesai 1 paragraf

Jika penulisanya 2 paragraf: (Othmer and fernsstrom,1943)

Penulis > 2: (Othmer dkk,1943)

Nama penulis pertama dalam daftar pengarang buku tersebut. Atau Othmer *et al*, 1943 garis bawah, tanpa titik antara et dan al dengan titik setelah al. Bila tidak ada pengarang, sebutkan nama penerbitnya jangan pakai “anonym”

Contoh: (BPS, 1943)

Kalau keluar berkali-kali: (Kirk&Othmer,1943, pp.179-182), lalu didaftar pustaka tak perlu lagi mencantumkan no halaman lagi.

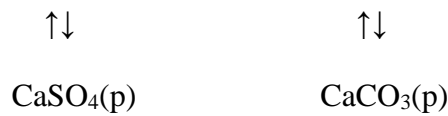
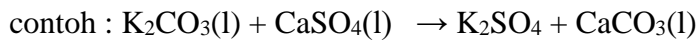
Tujuan Pencantuman Sumber:

- 1) petunjuk bagi orang yang ingin mendalami, nomer halaman disebutkan pada daftar pustaka

- 2) Penghormatan kepada penulis yang diakui
- 3) Pengecekan apakah interprestasinya benar

4. Landasan Teori

“Dijabarkan oleh peneliti berdasarkan tinjauan pustaka secara kualitatif.



$$S-1 : r = k [K_2CO_3][CaSO_4]$$

$$[CaSO_4] \text{ dianggap tetap, sehingga } r = k_1 [K_2CO_3]$$

untuk S-2: harus membuktikan meknisme reaksi bahwa reaksi itu benar –benar

order 1 semua ditinjau reaksi permukaan tahanan difusi dan tahanan kimia.

pemungutan senyawa Al dari Lumpur penjernihan air

Butir Lumpur ($Al_2(SO_4)_3 + Ca(OH)_2$)

↓

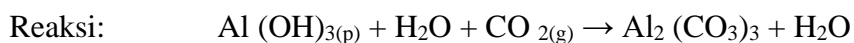
Butiran-butiran Lumpur diselubungi oleh $Al(OH)_3$

↓

Menggumpal

↓

Mengendap → Al akan dipunggut lagi karena mahal dan mencemari lingkungan



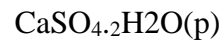
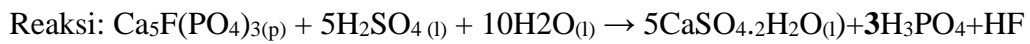
Mekanisme Reaksi langkah-langkah:

1. CO₂ mendifusikan ke cairan
2. Difusi CO₂ dalam cairan kepermukaan padatan
3. Reaksi permukaan

Reaksi permukaan kemungkinan mengikuti “*Skrinking Core Model*”

Contoh:

Reaksi pembentukan asam fosfat dari batu fosfat dan asam sulfat secara sinambung dalam kolom berpenghalang.



Kecepatan reaksi tergantung:

1. Perpindahan massa melalui film cair – padat
2. Reaksi kimia pada permukaan baku fosfat

Persamaan yang digunakan:

$$-\frac{dx_A}{d\tau_B} = \frac{B(1 - X_A)}{(Ro(1 - XB) / Das + 1 / (Kras(Bs))} \dots\dots\dots(17)$$

$$-\frac{dXA}{d\tau B} = \frac{R(1 - XA)}{\frac{Ro}{Das} (1 - XB)^{1/3} + \frac{1}{Kr.as.CBs}} \dots\dots\dots(18)$$

Reaksi pembentukan NaHCO₃ cukup cepat sehingga kemungkinanya:

1. reaksi kimia saja
2. reaksi kimia dan difusi

5. Hipotesis:

“Perkiraan hasil /pernyataan singkat serta kesimpulan sementara dan dibuktikan kebenarannya dengan mengadakan penelitian.

6. Rencana Penelitian

1. Bahan
 - a. jenis
 - b. spesifikasi yang harus diketahui kalau dilaporan penelitian, spesifikasi lebih lengkap dalam lampiran disebutkan cara analisisnya
2. Alat: Gambar dan keterangan ukurannya dll
3. Cara penelitian
 - a. uraian terinci tentang cara kerja
 - b. Variabel yang akan diteliti rancangan percobaan
4. Analisis Hasil: kimia, fisis, mekanis, dan statistik

7. Jadwal Penelitian:

Tahap – Tahap:

1. Persiapan
 - a. Pembuatan Alat
 - b. Pengambilan Lumpur aktif
 - c. Analisis bahan baku
 - d. Penerapan Alat
2. Pelaksanaan
 - a. Percobaan pendahuluan
 - b. Pengumpulan data
 - c. Pengolahan Hasil

d. Percobaan penjernihan

e. Pengolahan Data

Pada percobaan pendahuluan:

- mengenal Alat
- mencari kisaran variable

Pengolahan hasil:

Jangan hanya angka tapi juga bisa menunjukkan wujud produknya, beserta spesifikasinya dan kualitasnya.

Pengolahan Data:

Dalam bentuk grafik dll.

Tahap- tahap seminar:

- I. Tahap persiapan
- II. Setelah pengumpulan data
- III. Setelah selesai semuanya

Tahap – tahap Penelitian:

1. Persiapan
 2. Pelaksanan
 3. Penyelesaian
- pembuatan laporan
 - pembuatan makalah
 - pengetikan
 - penggandaan
 - penjilidan

8. Daftar Pustaka

Kebawah, menurut abjad nama akhir penulis pertama/yang bertanggung jawab

Kekanan

- a. **Majalah**: penerbit, tahun, judul tulisan, nama makalah, jilid, dan nomer halaman.
- b. **Buku**: penerbit, tahun buku, terbitanya

Dalam daftar Pustaka:

- tidak ada nomer urut untuk mempermudah penyisipan
- kalau ada 2 pustaka dengan nama penulis II
- kalau tahunnya sama bahkan dari majalah yang sama no halamannya lebih kecil didahulukan, tahun terbitnya.

BAB IV. PELAKSANAAN PENELITIAN

IV.1. Penyiapan

Penyiapan untuk melakukan penelitian adalah mempersiapkan:

- 1) Bahan: dilakukan pencarian bahan, penghalusan dan pengeringan
- 2) Alat: dilakukan pembuatan atau merangkai alat sesuai yang direncanakan, kemudian dilakukan pengujian.

IV.2. Peneraan alat ukur

- suhu
- kecepatan alir

Perancangan Alat:

Perhatikan prosesnya, misal: kalau dalam proses dihasilkan zat padat maka jangan pakai kolom berisi sehingga akan cepat tersumbar. Agar pangadukan cukup sempurna, bisa dipakai kolom berpenghalang. Penghalang $\frac{1}{2}$ ellips bergerigi.

Peneraan Alat:

- 1) Peneraan Termokopel

Keterangan:

1. pemanas listrik
2. panci
3. media pemanas: minyak, pasir dll
4. thermometer standar
5. termokopel
6. pirometer

catatan:

- Termokopel dan thermometer untuk peneraan tanpa bungkus, walaupun kelak dalam prakteknya termokopel dalam selubung (misal pada autoklaf).
- kenaikan suhu diusahakan sedikit demi sedikit dan diulangi pada penurunan suhu.

Suhu termometer terlalu sedikit lebih tinggi dari termokopel, pemuaian air raksa lebih cepat dari pada perubahan tegangan. Suhu maksimal 350°C, kalau diteruskan air raksa mendidih sehingga thermometer pecah walaupun dalam percobaan termokopel akan dipakai sampai 600°C, peneraan boleh dilakukan pada suhu <360°C dan selanjutnya diekstrapolasi.

2) Penaraaan Flow Meter

- 1) Botol pengaman
- 2) Termometer
- 3) Manometer
- 4) Flowmeter
- 5) Botol dan air
- 6) Peluapan
- 7) gelas ukur
- 8) ember

IV.3. Percobaan Pendahuluan

Menentukan kisaran variable, missal untuk mempelajari pengaruh waktu.

Prinsip Golden Section:

1. Tentukan waktu tersingkat dan terlama (1a dan 1b) dari pustaka coba hasil langsung dianalisis.

2. Coba tengah-tengahnya.
3. coba lagi tengah-tengah antara 1a dan 1b.
4. Coba lebih banyak data pada daerah kritis.

Kadang-kadang data 2b sampai dengan 1c tidak perlu lagi karena nilainya justru menurun misal untuk mencari kecepatan reaksi maka kita hanya ingin mempelajari kisaran waktu yang memberikan kenaikan konversi yang cukup berarti.

IV.4. Lembar Pengamatan

Yang perlu diperhatikan saat membuat atau menjalankan penelitian:

1. Pencatatan

- a. Sifat catatan - lengkap
 - jelas
 - tahan lama, bisa dilanjutin orang lain
- b. Yang dicatat - bagan alat
 - grafik
 - hasil pengamatan
 - perhitungan
 - jenis dan ukuran
 - penurunan

c. Cara Pencatatan

Langsung dalam buku dengan karton supaya ada duplikat, tapi jangan disalin, untuk menghindari salah tulis. Penulisan dengan tinta jangan pensil

d. Cara Penulisan: rapi, tidak berjejal yang salah dicoret dan pembentukan di atasnya tidak boleh ditumpuki.

e. Lembaran Pengamatan:

Nomer percobaan, tanggal, bahan, jenis, dan jumlah, kolom pengamatan paraf peneliti.

2. Keseksamaan

1. jenis – jenis alat: ketelitian tertinggi
2. penempatan alat ukur harus tepat
3. pengambilan cuplikan: pakai pipet volum jangan gelas ukur karena tampang lintang terlalu besar.
4. kebersihan Alat: untuk percobaan, untuk analisis

3. Keselamatan

- a. zat-zat yang berbahaya diletakkan di tempat yang khusus
- b. kebakaran, perlengkapan, Langkah penanganan
- c. pertolongan sebelum pada kecelakaan laboratorium
- d. pemeriksaan sebelum meninggalkan laboratorium

Keselamatan kerja di laboratorium

Alat pemadam kebakaran: pasir, selimut, goni, penyemprot api.

Langkah penanganan kebakaran:

1. Sumber api diisolasi agar api tidak menjalar kemana-mana.
2. Hindari kontak dengan O₂ ditimbun pasir, dll.

3. Jangan dipadamkan dengan air karena air harus banyak sekali agar api padam kalau air cuma sedikit ada kemungkinan air pecah menjadi H_2 dan O_2 maka akan memperbesar nyala api.
4. Kalau memakai goni maka goni basah agar tertutup sehingga O_2 tidak bisa masuk dan air diuapkan oleh kalor sehingga suhu turun.

Pertolongan pada korban kebakaran:

1. Matikan sumber bahan bakar.
2. Korban diselimuti.
3. Jangan buka baju korban karena bisa membuat kulit yang terbakar terkelupas.
4. Luka bakar diberi vaselin/margarine karena merupakan bahan yang mudah dan menyerap, banyak kalor dan melindungi tubuh dari penguapan selanjutnya jangan memakai es karena suhu akan merusak jaringan tubuh dan air tidak bisa melindungi tubuh.
5. Bawa ke Dokter.

Terkena asam sulfat pekat:

1. Disembur dengan air.
2. Kulit yang terlanjur kena diberi pasta $NaHCO_3$, jangan diberi $NaOH$ karena panas reaksi besar sekali.

Terkena Penol:

1. Jangan dicuci dengan air karena kulit akan memutih dan mengelupas.
2. Cuci dengan etanol.

4. Cara Pengambilan Cuplikan:

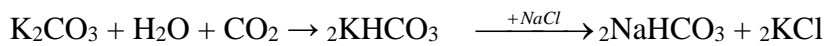
Duplikat: diambil 2 kali pada ukuran yang sama, tidak boleh ambil 1x10ml diencerkan jadi 100 ml , ambil 2x10 m pengenceran harus menggunakan labu takar, tidak boleh dengan gelas ukur.

5. Penempatan alat

Contoh:

Thermometer harus ditempatkan pada tempat terjadinya reaksi.

Pembuatan KCl dari ekstrak abu



Untuk mengukur jumlah CO₂ masuk:

Alat ukur suhu, tekanan, flowmeter

$$\text{Jumlah mol: } n = \frac{VP}{RT}$$

Pengambilan Cuplikan

- a. harus mewakili cairan reksi dalam reactor
- b. pengadukan tetap baik saat pengambilan cuplikan pengaduk tidak dihentikan.

Kebersihan Alat

- a. alat untuk percobaan
- b. alat untuk analisis
- c. untuk mencuci buret

buret diisi asam pencuci (K₂Cr₂O₇ + H₂SO₄ pekat) selama minimal 24 jam agar senyawa organic teroksidasi lalu dibilas denagn air.

BAB V. PENGOLAHAN DATA

V.1. Pengumpulan Data

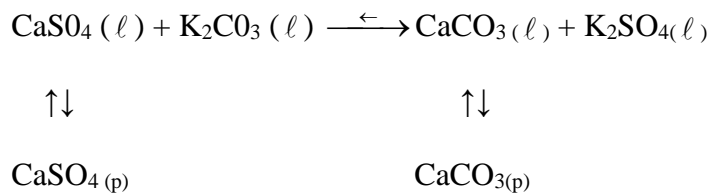
Data yang didapatkan dari hasil penelitian dikumpulkan dan disusun diurutkan menurut urutan terkecil, kemudian dilakukan langkah-langkah pengolahan data:

1. Perhitungan hasil, dilakukan sejak awal setiap selesai 1 run.
2. Pembuatan daftar:
 - a. Daftar lengkap seluruh nomor percobaan, disusun dengan nomor percobaan.
 - b. Hasil setiap variable diurutkan berdasarkan besarnya variabel (nomor percobaan tidak urut) dan dicantumkan dalam pembahasan.
3. Pembuatan Grafik

Agar lebih mudah dilihat kecenderungannya maka dibagi dua yaitu grafik hasil dan grafik penolong.

V.2. Contoh pada pembuatan kalium sulfat dari ekstrak abu

1. Penjelasan kualitatif menjelaskan kecenderungan yang tampak dalam grafik
2. Penjelasan kuantitatif:



Karena CaSO_4 dalam larutan berkeselimbangan dengan CaSO_4 padat, konsentrasinya boleh dianggap tetap.

$$r = k C_{\text{CaSO}_4} C_{\text{K}_2\text{CO}_3} = k' C_{\text{K}_2\text{CO}_3}$$

$$\Rightarrow -\ln(1-x) = k't - k't_0$$

Linearisasi \rightarrow hati-hati:

Cek apakah betul-betul linear untuk kisaran variable yang dipelajari (jangan asal masukan program computer), gambar dulu titik-titiknya.

Kembali ke contoh kalium sulfat

Konversi perlu ditinjau dari dua sisi pereaksi

a. Pereaksi cair (K_2CO_3)

$$r = -\frac{dC_{\text{K}_2\text{CO}_3}}{dt} = \frac{C_{\text{K}_2\text{CO}_3}}{\frac{1}{k_s \cdot a_s} + \frac{1}{k_r \cdot a_s \cdot C_{B_s}}}$$

b. Pereaksi Padat (CaSO_4)

$$r = \frac{dcp}{dt} =$$

ditinjau berdasarkan teori shrinking core model (mengamati perubahan/pengurangan jari-jari).

Persamaan a dan b diselesaikan secara simultan dengan metode numeris (misalnya runge kutta) untuk memperoleh nilai k_r dan k_s . Misalnya diperoleh $k_s \gg k_r$, sehingga persamaan a bisa disederhanakan menjadi:

$$-\frac{dC_{\text{K}_2\text{CO}_3}}{dt} = k_r a_s C_{B_s} C_{\text{K}_2\text{CO}_3} \text{ merupakan konstanta}$$

$$= k' C_{\text{K}_2\text{CO}_3} \text{ terbukti bahwa reaksi order 1 semu (untuk S-2)}$$

harus ada pembuktian seperti ini).

Variabel yang harus dipelajari (dalam kasus K_2SO_4 diatas)

1. Yang erat hubungannya dengan difusi: pencampuran

- a. pengaduk, penggelembungan gas (ukuran butir zat padat).
- b. Kaitannya dengan bilangan Reynold.

$ks = a (RE)^b \rightarrow$ jika b nilainya $>$ artinya difusi menentukan

- E/RT

$$k = A e$$

A = erat kaitannya dengan difusi

2. Yang berkaitan dengan reaksi kimia:

Suhu: jika suhu naik $10^\circ C$, k naik ± 2 kali lipat maka reaksi kimia menentukan.

Seandainya belum ada teori yang bisa mendukung, penelitian dilakukan sedemikian rupa sehingga bisa didekati dengan teori-teori yang sudah dipelajari.

Kalau teorinya memang belum berkembang maka:

1. Tetep harus dibahas secara kualitatif dan kuantitatif.
2. Pembahasan kuantitatifnya boleh merupakan persamaan EMPIRIS.
3. Untuk pegangan penyusunan persamaan empiris lihat bagan akhir bab matematika di Perry edisi 2.

Persamaan-persamaan yang diperoleh dari pembahasan kuantitatif adalah kecenderungan Umum sesuai teori yang ada. Kemungkinan ada titik-titik yang “Salah” dan dianggap kesalahan pada percobaan, persamaan 100 % betul.

$$\text{Kesalahan relative} = \left| \frac{x_{perc.} - x_{hitung}}{x_{hitung}} \right| \cdot 100 \% \text{ (untuk setiap titik)}$$

$$\text{Kesalahan rata-rata} = \frac{\text{jumlahkesdahanrelatif}}{\text{jumlahdata}} = \pm \text{tak terhingga } \%$$

Cara statistik:

1. Untuk penelitian yang variabelnya sulit dikendalikan.
2. Untuk penelitian yang mempelajari perbandingan.

Penelitian yang mempelajari beberapa macam variabel misal diperoleh:

Konversi : $x = a e^{bt}$

Suhu : $K = A e^{-E/RT}$

Pengadukan: $k = a P^b$

Katalisator: $k = \alpha \cdot e^{-\beta/N}$

Gabungan variabel $k = m \cdot e^{n/T} \cdot P^s \cdot e^{q/n}$

$$\ln k = \ln m + \frac{n}{T} + s \ln P + \frac{q}{N}$$

$$\ln k = Z + \frac{n}{T} + S \ln P + \frac{q}{N} \rightarrow \text{dengan 4 persamaan simultan, bisa dihitung } z, n,$$

s, dan q.

Cara lain: $k = \alpha e^{-E/RT} \cdot P^b e^{\beta/N}$

$$\ln k = a \left[-\frac{E}{RT} + b \cdot \ln p + \frac{\beta}{N} \right] + \ln \alpha$$

BAB VI. LAPORAN PENELITIAN

Laporan penelitian terdiri dari, bagian depan, bagian utama dan bagian akhir.

VI. Bagian depan

1. Sampul: - judul dan keterangan
 - Lambang UAD
 - Nama penelitian
 - Yang dilapori
 - TahunDitulis pada kertas manila biru
2. Halaman Judul sama dengan sampul (tapi pada lembar kertas putih biasa).
3. Prakata: - tujuan
 - penjelasan
 - ucapan terima kasih.
4. Daftar Isi: nomor halaman, bab, sub judul, anak sub judul.
5. Intisari : - Permasalahan atau latar belakang
 - Cara penelitian atau jalan penelitian
 - Hasil atau kesimpulan.

Jika ada banyak table, gambar, dan lambang dalam daftar isi ditambahkan: daftar table, daftar gambar, daftar dan arti lambang.

Tujuan daftar isi: memudahkan pembaca untuk mencari topik yang ingin dibaca.

Dalam buku, daftar isi dibantu dengan indeks (ada indeks penulis dan indeks subjek).

Contoh: Daftar Isi

DAFTAR ISI

Prakata

Intisari

1. Pengantar

Latar Belakang

Tinjauan Pustaka

Landasan Teori

Hipotesis

Rencana Penelitian

2. Cara Penelitian

Bahan Baku

Alat

Jalan Penelitian

Analisis Hasil

3. Hasil Penelitian

4. Kesimpulan

Daftar Pustaka

Lampiran

A. Penerapan Flowmeter

B.

C.

Daftar Hasil Menyeluruh

Keterangan

Intisari:

1. Dalam Makalah: ditulis dalam satu alenia.
2. Dalam Thesis/Laporan: dalam 3 alinea dan harus cukup dalam satu halaman (ketik 1 spasi) dan buat seringkas mungkin.

Gunanya Intisari:

1. Membantu orang yang waktunya terbatas.
2. Membantu orang yang awam dalam bidang tersebut, makanya dalam intisari jangan terlalu banyak memakai penyajian kuantitatif (matamatis), memakai kualitatif saja.

VI.2. Bagian Utama

Bagian ini terdiri dari:

1. Pengantar, berisi tentang: latar belakang permasalahan dan pemecahan, keaslian dan faedah (untuk pembangunan dan ilmu/ teknologi). Mutu penelitian dinilai dari berapa % keasliannya.

2. Tujuan Penelitian

Untuk S-2: mempelajari bidang ilmu tertentu (titik berat program S-2 di UGM memperdalam ilmu pengetahuan, tidak terlalu melebar).

3. Tinjauan Pustaka

- a. Keterangan penunjang (sistematis)

Mulai dari yang umum sampai mendekati topic yang diteliti.

- b. Sumber keterangan (nama dan tahun)

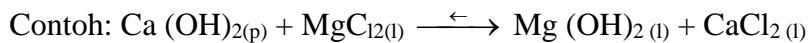
Buat kerangkanya dulu supaya sistematis.

Sumber Keterangan bila diacu satu kali, no halaman dicantumkan didaftar pustaka, dan bila diacu berkali-kali no halaman dicantumkan di sumber keterangan (Griffin, 1923, pp. 36-41) dan didaftar pustaka tidak perlu ditulis lagi no halaman. Mengacu makalah yang belum diterbitkan, tidak perlu menulis no halaman.

4. Landasan teori

a. Model Matematis

b. Uraian Kualitatif



↑↓

↓↑

Ca (OH)_{2(p)}

Mg (OH)_{2(p)}

(selalu dalam kesetimbangan)

Kualitatif: karena Ca (OH)₂ dalam larutan selalu dalam kesetimbangan dengan Ca (OH)₂ padat, maka reaksi bisa dianggap berorder semu terhadap MgCl₂ saja. Dan harus ada penjelasan lebih lanjut tentang mekanisme reaksi untuk membuktikan penjelasan kualitatif diatas.

5. Hipotesa

6. Rencana penelitian

Rencana atau cara penelitian

1. Bahan → Spesifikasi lebih lengkap daripada di usul penelitian (asal, penyiapan, sifat fisis, susunan kimia).

Contoh: Ca (OH)₂ untuk reaksi dengan MgCl₂ dalam spesifikasi kadra aktif lime (CaO aktif) total bukan Ca total karena Ca total termasuk CaCO₃ (tidak aktif).

Untuk analisis aktif lime:

- $\text{Ca}(\text{OH})_2$ + larutan gula pasir dikocok

Dan tentukan jumlah Ca Sakarat (CaCO_3 tidak bisa membentuk Ca Sakarat) hanya $\text{Ca}(\text{OH})_2$ yang bisa membentuk Ca Sakarat.

2. Alat: Menggambar mendekati keadaan sesungguhnya dan agak propisional.

Keterangan:

- Alat yang dibuat sendiri: sebutkan ukuran dan spesifikasi dengan lengkap
- Alat Standart sebutkan jenis dan kapasitas.

3. Jalan Penelitian

- uraian lengkap dan terperinci
- tindakan dan pengumpulan data

3. Analisa Hasil

- Uraian lengkap (kimiawi, fisis, statistik)

Untuk menentukan apa yang harus dianalisa:



- Ca: Sebelum reaksi ada dalam CaSO_4 (padat), setelah reaksi juga dalam padatan (CaCO_3) . tidak bisa dianalisis secara kimiawi.

- SO_4 : Terjadinya dalam padatan (CaSO_4) lalu setelah reaksi ada dalam larutan (K_2SO_4) jumlah SO_4 dalam larutan makin meningkat dan bisa dianalisis secara kimiawi.

CO_3 : Sebelum reaksi dalam larutan (K_2CO_3) setelah reaksi dalam padatan (CaCO_3). Penurunan konsentrasi CO_3 dalam larutan bisa dianalisis.

Contoh lain: pembuatan dekstrin dari pati

Pati + HCl dipanasi dalam waktu singkat

Analisis: - glukosa total dan glukosa bebas, selisihnya adalah pati yang terkonsentrasi menjadi dekstrin.

4. Kesulitan dan Pemecahan

- Agar peneliti selanjutnya tidak perlu mengalaminya

7. Hasi penelitian dan pembahasan

1) Hasil - daftar

Grafik

2) Pembahasan:

Penjelasan teoritis tentang hasil kualitatif dan kuantitatif, statistik.

Dibahas kuantitatif dulu untuk menjawab pertanyaan “mengapa” pembahasan dibuat untuk semua variabel yang dipelajari berpengaruh pada pengadukan, pengaruh suhu, dan pengaruh gabungan variabel. Setiap ada hasil daftar atau gambar langsung dibahas.

Perlu dibahas pula soal pemungutan hasil dan pengolahannya:

- kualitatif apakah yang diperoleh itu betul K_2SO_4

Misalnya: Dikelolah sampai mengkristal dan diperoleh zat padat kemungkinan masalah pengolahan hasil ini bisa jadi satu penelitian tersendiri.

8. Kesimpulan dan saran

- 1) Kesimpulan: Pernyataan singkat dan tepat (pembuktian kebenaran hipotesa) apa yang disebutkan dalam hipotesa dijawab dulu baru disusul kesimpulan lain.

- 2) Saran: Pengalaman dan pemikiran untuk dilanjutkan dan dikembangkan, saran ini tidak harus ada.

VI. 3. Bagian Akhir

Berisi daftar pustaka dan lampiran-lampiran

Daftar pustaka

- 1) kebawah menurut abjad nama akhir penulis pertama
- 2) kekanan:
 1. buku: nama penilis, tahun terbit, judul buku, jilid, terbitan ke, no halaman, penerbit, kota
 2. majalah: nama penulis, tahun terbit, judul tulisan, nama majalah, jilid, dan no halaman.

catatan: semua penulis harus disebutkan

nama akhir disusun huruf I nama depan, tengah dst.

Lampiran

1. Peneraaan alat
2. data awal/dasar
3. contoh perhitungan
4. daftar dan gambar yang terlalu besar

BAB VII. PENYUSUNAN ARTIKEL ILMIAH

Makalah lebih ringan dibandingkan dengan laporan penelitian dan keterangan pelengkap ditiadakan dan jumlah halaman dibatasi 10 halaman atau sesuai dengan aturan jurnal masing-masing.

VII.1. Susunan Makalah

1. Judul dan Nama Penulis

- Judul dalam bahasa Indonesia di bawahnya dalam bahasa Inggris.
- Nama penulis, peneliti, Pembimbing Utama dan pembimbing pendamping

2. Intisari

Dibatasi sekitar 100-250 kata dan harus informative, hanya satu alenia, bedanya dengan intisari tesis jangan dipecah jadi 3 alenia jadikan satu alenia saja. Aturan jumlah kata disesuaikan dengan aturan dari jurnal masing-masing.

3. Pengantar

- Latar belakang dan tinjauan pustaka tidak dipisahkan dalam sub bab tapi ditulis dalam alenia berbeda
- Tinjauan pustaka seperlunya saja, tapi teori sebaiknya ada hipotesis dan rencana penelitian tidak ada, pada akhir dicantumkan tujuan penelitian.

4. Cara penelitian

Bahan, alat, jalan penelitian dan analisis hasil dapat disajikan eksplicit menggunakan sub judul, tetapi dapat juga dijadikan satu uraian dan pemisahan hanya dengan alenia baru tergantung pada luasnya yang disajikan, gambar sebaiknya disajikan.

5. Hasil dan pembahasan

Bab ini disajikan secara meluas, lengkap dengan daftar grafik, pembahasan kualitatif dan kuantitatif.

6. Kesimpulan

Singkat dengan kalimat berlanjut tanpa perincian nomer.

7. Ucapan terimakasih

Seperlunya saja kalau tidak ada yang perlu diberi terima kasih bagian ini sering diganti daftar lambang.

8. Daftar pustaka

Daftar pustaka yang dipakai untuk membuat makalah bisa jadi daftar pustaka di thesis.

VII.2. Persyaratan seminar

Persyaratan seminar nasional

1. seleksi oleh redaksi
2. biaya pemuatan
3. sanggahan pembaca
4. tunduk pada pedoman redaksi

Tulisan untuk pertemuan ilmiah

Susunan dan isi hampir sam dengan tulisan untuk majalah ilmiah

Syarat untuk majalah ilmiah:

1. seleksi oleh panitia
2. pedoman oleh panitia
3. biaya untuk menhadari pertemuan

4. Tanya jawab pada waktu diskusi

VII.3. Penyajian makalah dalam pertemuan ilmiah

Pembicara:

1. waktu terbatas: 15 menit uraian, apa yang mendorong dilakukan penelitian, bagaimana hasilnya
2. cara penyajian
 - a. Ringkasan
 - permasalahan
 - cara penelitian
 - hasil daftar secara grafik
 - b. alat peraga

Perhatian:

1. Jangan membaca teks
2. Usahakan agar hadirin tertarik
3. jangan mengajari peserta (jangan sok tahu)

VII.4. Pimpinan sidang

1. Susunan : Ketua / wakil
: Sekretaris
2. Tugas : Membuka Sidang
: Memperkenalkan pembicara
: Mengatur jalanya sidang
: Menutup

: Membuat laporan

Tanya jawab

1. Penanya: - identitas (nama dan instansi)
- pertanyaan (lisan dan tertulis)
2. Pembicara: - menjawab (lisan dan tertulis)

VII.5. Tata cara

Bahan dan Ukuran:

1. Naskah: kertas HVS 80 gram
2. Sampul: kertas buffalo atau manila yang baik
3. Warna: biru tua (warna biru teknik)
4. Tulisan Sampul: judul dan keterangan, lambang, peneliti yang dilapori, tahun pada punggung buku juga ditulis.
5. Ukuran: 21cm x 28 cm

Pengetikan:

1. Huruf: Pica 10 pitch (10 huuuruf /inch)
miring diberi garis bawah untuk mesin ketik mekanik dan lambang dibuat dengan tinta hitam.
2. Jarak Baris: 2 Spasi, intisari, daftar pustaka
3. Batas Tepi
4. Pengisian Ruang
5. Alenia baru mulai ketikan ke 6
6. Permulaan kalimat
7. Judul

Penomoran

1. Halaman

- Bagian depan Romawi kecil
- Bagian utama /isi + bagian belakang angka arab
- Letak nomor: umum kanan atas

Judul kanan bawah

Kalau halaman dicetak bolak – balik, nomer ganjil sebelah kanan, nomer ganjil sebelah kiri.

2. Daftar Pustaka

Gambar: Angka Romawi Besar

Persamaan diberi no urut dibawah disebelah kanan kalau daftar gambar banyak boleh di tulis.

Daftar dan gambar

1. Daftar

- nomer dan nama disebelah atas tidak boleh dipenggal. Pemenggalan diijinkan kalau memang terpaksa, tapi heading table harus ditulis kembali
- kolom terpisah tidak perlu digaris
- daftar melebar bagian atas di sisi kiri halaman
- garis pemisah
- pengetikan simetris

2. Gambar

- Nomer dan nam disebelah bawah
- Tidak boleh dipenggal

- Keterangan ditempatkan di halaman yang sama
- Gambar melebar analog dengan dftra melebar
- Ukuran tidak melebihi ukuran jertas
- Skala disesuaikan agar mudah interpolasi dan ekstrapolasi.
- Garis lengkung → mulus (smooth)
- Tinta Hitam
- Simetris.

Bahasa

Bahasa Indonesia baku (subjek + predikat + objek + keterangan)

Bentuk kalimat (tidak boleh memakai: saya, kami, kita dll)

Istilah: Indonesia /di Indonesiakan

Asing (cetak miring / garis bawah)

Kesalahan

- a. kata hubung: sehingga, sedangkan, akan tetapi
- b. kata depan: pada
- c. dimana, dari, mengacu pada tempat
- d. awalan: di dan ke
- e. tata bahasa

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LAMPIRAN

1. Paper Publish di Jurnal Internasional terindek Scopus
2. Paper Publish di Jurnal Nasional terakreditasi Sinta

Effects of Temperature and Catalysts on the Yield of Bio-oil During the Pyrolysis of *Spirulina platensis* Residue

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Abstract- *Spirulina platensis* (SP) is aquatic biomass potentially grown as an energy source in the future. Extracting algae oil from SP will leave solid residue called *Spirulina platensis* residue (SPR). In this study, SPR processed by the pyrolysis process, both catalytic and non-catalytic. This study focused on the effects of different catalysts on the yield of bio-oil obtained from the pyrolysis process. Pyrolysis experiments conducted at a temperature range of 300 – 600°C using three different catalysts: Ni-oxide, Fe-oxide, and silica-alumina, and performed at 550°C in various amounts of 5%-30 wt. % for each catalyst. The effect of temperature on SPR catalytic and non-catalytic pyrolysis was studied. The result shows that the optimum yield of non-catalytic pyrolysis was 27.34 wt.% at 550°C. The yield of bio-oil within catalytic pyrolysis indicates that the maximum pyrolysis oil obtained using Fe-oxide as catalyst with 30.00 wt.% bio-oil, followed with 23.37 wt.% using Ni-oxide catalyst, and 18.41 wt.% using silica-alumina catalyst. The water phase yield for each catalyst was relatively different for every catalyst in various amounts ranging from 5 - 30%. Char and gas yields were almost the same for each type of catalyst in all amount variations. The highest gas yield produced was using the silica-alumina catalyst, followed by Fe-oxide and Ni-oxide catalyst. The SPR catalytic pyrolysis process improves the bio-oil yield with the increasing of the bio-oil yield 2.66 wt.% compared to the SPR non-catalytic pyrolysis.

Keywords: *Spirulina platensis* residue, Pyrolysis, Ni-oxide, Silica-alumina, Fe-oxide.

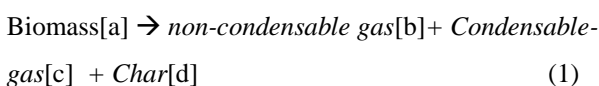
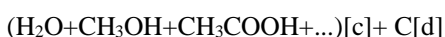
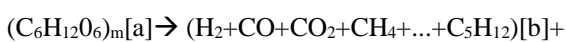
1. Introduction

Interest in the development of renewable energy is increasing along with the increasing world energy demand. The current energy source is still very dependent on fossil fuels, but it has limited resources and environmental problems such as global warming and air pollution. One of the abundant and quickly developed renewable energy sources is biomass, including wood, herbaceous plants, aquatic plants, and manure [1]–[8]. One of the biomass-derived from marine plants is microalgae. Biofuel from microalgae has several advantages, such as (i) high growth rates (up to 20 g of dried algae per m² per day), (ii) high yields per area, 15 times higher than oil palm [9], [10], (iii) high efficiency in absorbing CO₂ and solar energy conversion (8%) [6], (iv) no competition with agriculture and food [11]–[15]. Also, microalgae can grow in open waters (seas and ponds) and photo-bioreactors in non-agricultural land [16], [17].

Algae biomass contains three main components; they are carbohydrates, proteins, and natural oils (lipids). Nowadays, biodiesel from algae is produced by lipid extraction with organic solvents, such as hexane, followed by methanol trans-esterification using an essential catalyst [18]–[20]. Solid residues from this process contain minerals (up to 10%) that can be applied back to the algae growth cycle. However, the remaining 60% is a residue that not utilized, making it less economically attractive. Therefore, it is necessary to find a solution to the residual wastes, using the pyrolysis process to process this spirulina residue. The pyrolysis of microalgae [18], [19] and macro-algae [20], [21] produce hydrocarbons mixture, has shown to have the potential for large scale applications. Interestingly, Çoban et al (2015)[7] have demonstrated that pyrolysis of algae (*chlorella*) produces high bio-oil yields, above 40% at the temperature range of 300-500°C.

Microalgae conversion technologies to produce renewable energy can classify into four ways, biochemical conversion, thermochemical conversion, chemical reactions, and direct combustion or pyrolysis [22]-[24]. Microalgae with high lipid content are more suitable to be converted to biodiesel by chemical reaction (transesterification). In contrast, microalgae with low lipid content can treat by biochemistry or thermochemistry processes [23]. Pyrolysis is a thermochemical decomposition of biomass at a temperature range of 400-600 °C, taking place without the presence of oxygen so that the wood components broke down and produced three product forms: solid, liquid, and gas [22]-[24]. Pyrolysis technology is the most beneficial because the process is simple, does not require additives/solvents, operates at 400-600 °C and low pressure [25]. Pyrolysis produces fuel products and leaves no waste [25]-[28].

The chemical reaction of pyrolysis is [29]:



From Equation (1), biomass pyrolysis products are non-condensable gas (H_2 , CO , CO , short-chain hydrocarbons such as CH_4 , C_2H_6 , C_2H_4 , C_3H_8 , and other gases such as NH_3 , NO_x , SO_2), condensable gas (H_2O , CH_3OH , CH_3COOH , etc.), and char. The condensed gas is a thick black liquid called bio-oil or pyrolysis oil, bio-crude oil, bio-fuel oil, wood liquid, wood oil, liquid smoke, wood distillates, or pyro-ligneous tar [28]-[30]. In bio-oil production, no waste produced because all of the raw materials converted into bio-oil and charcoal. At the same time, non-condensable gas returns to the process as an energy source [29], [30].

The effects of catalyst in pyrolysis process are (i) significantly decreases the biomass decomposition temperature, (ii) influences the reaction tissue, for example, deoxygenation which allows the increase of bio-oil yield by reducing oxygenate compounds, (iii) reduces polymerization to stabilize bio-oil, the reaction more effective, (iv) releasing more CO , CO_2 , and H_2O , and (v) increases coke formation [29]-[31].

This paper discussed the effects of temperature, type, and concentration of catalyst on the yield of bio-oil, the conversion, and the product composition. There are two types of catalysts used. The first one is an acid catalyst, silica-alumina, and the second type is a metal catalyst, Ni-oxide, and Fe-Oxide. This paper also compared pyrolysis performances between non-catalytic and catalytic pyrolysis. The concentration of catalyst used in pyrolysis was ranging from 5 - 30% wt. Pyrolysis carried out on fixed bed reactors at temperatures ranging from 300 to 600°C, the pressure of 1 atm, and the heating rate of 20 - 35°C / min.

2. Material and Method

2.1. *Spirulina Plantesis Residue (SPR)*

Dry *Spirulina platensis* residue (SPR) was obtained from *Spirulina platensis* (SP) solid residue extraction, while SP received from Nogotirto Algae Park, Yogyakarta, Indonesia. SPR samples were analyzed to obtain proximate value, the ultimate value, and high heating value (HHV). Proximate analysis carried out to receive a percentage of moisture content, ash, volatile matter, and fixed carbon. Ultimate study (C, H, O, N, and S with standard D-2361) conducted at Laboratorium Pengujian, Puslitbang Tekmira, Bandung (Testing Laboratory, Research Center Tekmira, Bandung). High Heating Value (HHV) was analyzed by bomb calorimeter at the Food and Agricultural Product Laboratory, Department of Agricultural Technology, and the Laboratory of Food and Nutrition of the Inter-University Center, UGM. The analyzed SPR composition was protein, carbohydrates, and proteins. Protein analysis used was the Kjeldahl method; the carbohydrates analysis used was the Anthrone method; lipids analysis used was the Soxhlet method.

2.2. Catalysts

There were three catalysts used in this experiment, Ni-oxide, and Fe-oxide, which were metal catalysts and silica-alumina, which is an acid catalyst. The alumina-silica catalyst was obtained from PT Pertamina Balongan in powder form, while Ni and Fe-oxide catalyst was from PT Pupuk Kujang, Cikampek, Indonesia. Each of the catalysts was analyzed by SEM and SEM-EDX (Scanning Electron Microscope – Energy Dispersive X-Ray) at Laboratorium Penelitian dan Pengujian Terpadu (LPPT) UGM (UGM Integrated Research and Testing Laboratory). SEM analysis performed to learn more about the shape and size of the catalyst particles. SEM-EDX analysis performed to identify the elemental composition of catalysts' materials.

2.3. Sample Preparations

The *Spirulina platensis* residue (SPR) obtained in wet conditions. The SPR was dried under the sun for three days to reduce water content, then cleaned of dirt and lumps of SPR beads. SPR stirred to homogenize the size distribution; then, it stored in a dry and closed place.

The silica-alumina catalyst made into the pellet-shaped catalyst. Pellets were prepared by mixing silica-alumina 95 wt.% with kaolin five wt.% and then adding distilled water. After the mixture was homogeneous, it molded into a pellet-shape with 4 mm in diameter and 6 mm in height. The pellet-shaped catalyst was dried in a furnace at 500°C for 2 hours and then cooled in a desiccator. In contrast, Ni and Fe-oxide catalysts were pellet-shaped catalysts that had the same size as silica-alumina.

2.4. Pyrolysis Experiment

2.4.1. Instrument

SPR microalgae pyrolysis experiment, both catalytic and non-catalytic reactions, were performed in a fixed-bed reactor with an inner diameter of 40 mm, the outer diameter of 44 mm, and a height of 600 mm. The reactor made of

stainless steel. The reactor equipped with a heater, with two cylinders, the first cylinder was for biomass SPR (R1), and the second cylinder was for silica-alumina (R2) catalyst. The pyrolysis gas produced from R1 directly flowed to the catalyst stack in R2. The reactor arrangement sees in Figure 1, and the diagram for the fixed-bed reactor system see in Figure 2.

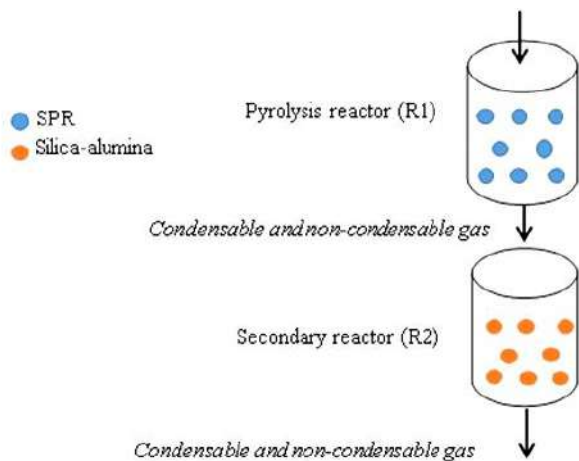


Fig. 1. Fixed-bed reactors arrangement [32]

2.4.2. Product Yield and Conversion Calculations

The total liquid or tar product (bio-oil and water phase), and gas were calculated by the equation [10], [23], [26], [32]:

$$Y_L = (W_L / W_M) \times 100\% \quad (2)$$

$$Y_{BO} = (W_{BO} / W_M) \times 100\% \quad (3)$$

$$Y_A = (W_A / W_M) \times 100\% = Y_L - Y_{BO} \quad (4)$$

$$Y_C = (W_C / W_M) \times 100\% \quad (5)$$

$$Y_G = 1 - (Y_L + Y_C) \quad (6)$$

The following equation calculated SPR pyrolysis conversion:

$$X = \frac{W_{BO} + W_A + W_G}{W_M} \times 100\% \quad (7)$$

In this case, Y_L , Y_A , Y_{Bo} , Y_C , and Y_G notations are respectively, liquid product yields, water phase, bio-oil, char, and gas. In contrast, W_M , W_L , W_A , W_{Bo} , and W_C are respectively the weight of the initial SPR, the yield of the liquid product, the water phase, bio-oil, and char.

2.4.3. Non-Catalytic Pyrolysis

Fifty grams of SPR was fed into reactor R1, tightly closed and heated. The R1 reactor was heated externally by an electric furnace, and the temperature-controlled by NiCr-Ni thermocouple placed outside of the reactor. The SPR samples heated with a heating rate in the range of 20 - 35°C/min from 30°C to the desired temperature (300 - 600°C). Then, the temperature was held constant for 1 hour. The temperature was

recorded every minute, and the heating rate and installed at the specified range. The pyrolysis gas was condensed, the liquid product came out, the condenser collected in the accumulator, and the produced gas was measured. The bio-oil and the water phase separated by decantation. After the experiment finished, the remaining char was taken and weighed. The yield of bio-oil, water phase, char, and gas was calculated by equation (2) - (6), while conversion calculated by equation (7). The experiment was repeated three times for each variable tested.

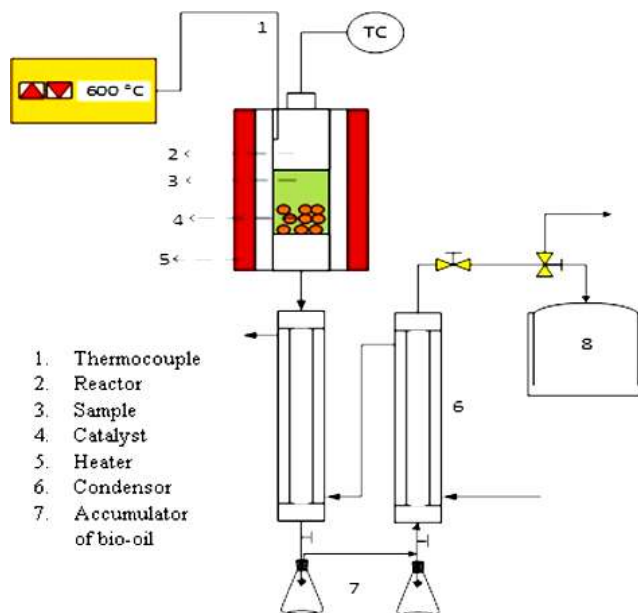


Fig. 2. The SPR pyrolysis system [32]

2.4.4. Catalytic Pyrolysis

Fifty grams of SPR fed into the reactor R1, and silica-alumina catalyst (5 - 30 wt.%) supplied in R2, then closed tightly and heated. The reactor heated externally by an electric furnace, and the temperature-controlled by a NiCr-Ni thermocouple placed outside of the reactor. The SPR sample heated with a heating rate in the range of 20-35°C/min from 30°C to 550°C; then, the temperature was held constant for 1 hour. The same method and equation as the non-catalytic pyrolysis used to calculate the yield of bio-oil, water phase, char, gas, and conversion to catalytic pyrolysis [32], [33]. The experiments repeated using the same method for each Ni-oxide and Fe-oxide catalysts.

3. Result and Discussion

3.1. Compositions of Spirulina Plantesis Residue (SPR)

Table 1 shows the results of the proximate analysis of the SPR. The proximate analysis performed to determine the weight percentage of moisture content, ash, volatile matter, and fixed carbon. The proximate analysis shows the highest rate of SPR is the volatile matter (68.31%) and leaves relatively low content of ash (8.93%).

Table 1 also shows the results of the ultimate analyses of the SPR feedstock. The ultimate analysis carried out to determine the weight percentage of elemental component,

carbon, nitrogen, hydrogen, and oxygen. The highest percentage was carbon (41.36%), followed by oxygen (35.33%), nitrogen (7.17%), and hydrogen (6.6%).

The high heating value of SPR found to be almost 18.21 MJ/kg. This result indicated that SP would be a better candidate if bio-energy production from renewable resources. The composition analysis shows that nearly half of the SPR composition is a protein (49.56%), followed by carbohydrates (38.51%), and a small amount of lipid (0.09%). This low lipid content in SPR is because the *Spirulina Plantesis* has been extracted and leaves this residue, which used in this experiment.

3.2. Characterization of Catalysts

Characterizations of catalysts were carried out by SEM and SEM-EDX analysis. The results presented in Table 2 and Figure 3. From Table 2, it can be seen that Ni-oxide consists of 4 components, they are O (46.01%), Al (34.39%), Ca (7.03%), and Ni (12.56%). Fe-oxide consists of 5 components, namely C (8.04%), O (28.75%), Cr (4.93%), Fe (56.64%) and Cu (1.64%). The silica-alumina consists of 4

components, namely C (8.41%), O (55.78%), Ca (24.64%) and Si (11.17%). Among the three types of catalysts, the most metal component is Fe at 56.64% in Fe-oxide catalysts with the addition of Cr 4.93% promoter.

Figure 3 explains the 10,000 times magnification of each catalyst. In Figure 3(a), macroscopically, the Ni-oxide catalyst is a fine gray powder. As for microscopically, each particle in Ni-oxide powder looks like a three-dimensional net with irregular size and shape of pores. It is structurally and thermally stable and has a large BET surface area (100 m²/g catalysts). Figure 3(b) is silica-alumina, it appears that SiO₂ mixed with Al₂O₃ in one grain, which is shown by transparent white granules such as glass and visible cavities in the catalyst with a pore surface area of 240.553 m²/g catalysts [32], [33]. As for Fe-oxide in Figure 3(c), from the results of 10,000 times magnification, they are not homogeneous. The presence of cluster groups suspected because of the magnetic nature of Fe₂O₃, so it tends to cluster. The color of Fe₂O₃ tends to be black; Cu is orange, while the intensity of Cr determined by the ligands attached to the center of Cr with a pore surface area of 1.963 m²/g catalysts.

Table 1. The proximate analysis of SPR

The proximate analysis of SPR		The ultimate analysis of SPR		The composition analysis of SPR	
Properties	wt. %	Properties	wt. %	Properties	wt. %
Moisture	9.99	C	41.36	Lipids	0.09
Ash	8.93	H	6.60	Carbohydrates	38.51
Volatile matter	68.31	N	7.17	Proteins	49.56
Fixed carbon	12.77	O ^a	35.33	HHV	18.21 MJ/kg

^aOxygen calculated by the difference

Table 2. Specifications of catalysts with SEM-EDX

Nickel oxide	Mass, %	Ferro oxide	Mass, %	Silica-alumina	Mass, %
O	46.01	C	8.04	C	8.41
Al	34.39	O	28.75	O	55.78
Ca	7.03	Cr	4.93	Al	24.64
Ni	12.56	Fe	56.64	Si	11.17
		Cu	1.64		

3.3. Non-catalytic Pyrolysis Result

The effects of temperature on non-catalytic pyrolysis see in Figure 4. The pyrolysis process was performed at a temperature from 300 to 660°C. The maximum pyrolysis oil obtained at a temperature of 550°C. The result shows that the maximum pyrolysis oil was 27.34 wt.%, then the pyrolysis oil drops of 27.02 wt.% slightly at a temperature of 600°C (27.02 wt.%). The water yield obtained ranging from 22.17 wt.% to 28.82 wt.%. Tar is a liquid product from the addition

of the bio-oil and water phase. The maximum tar yield obtained 56.16 wt.%. The char yield decreased significantly from 44.41 wt.% to 27.81 wt.% at temperatures of 300-600 °C. The gas yield was ranging from 14.57 wt.% to 24.88 wt.% at temperatures of 300 - 600°C.

The reduction of tar yield above the optimum temperature caused by secondary cracking (cracking, polymerization, condensation) reactions, the tar product in first cracking will partly decompose into gas so that the tar yield drops [34]-[36]. The amount of water in pyrolysis

products comes from the SPR (9.99% free water) and the reaction of water formation during pyrolysis (dehydration). According to [33]-[37], the average water content in tar in biomass was above 20 wt.%.

From equation 7, we can calculate the SPR pyrolysis conversion, i.e., the total weight of bio-oil, water phase, and gas divided by the initial weight of the SPR. Conversion of

SPR pyrolysis without catalyst at a temperature of 300-600°C can see in Figure 5. With the increase in pyrolysis temperature, the thermal decomposition of the SPR is more effective so that the weight of the SPR decreases, increasing conversion. Improvements in liquid and gas products indicated that the speed of the decomposition reaction rises as long as temperature increases.

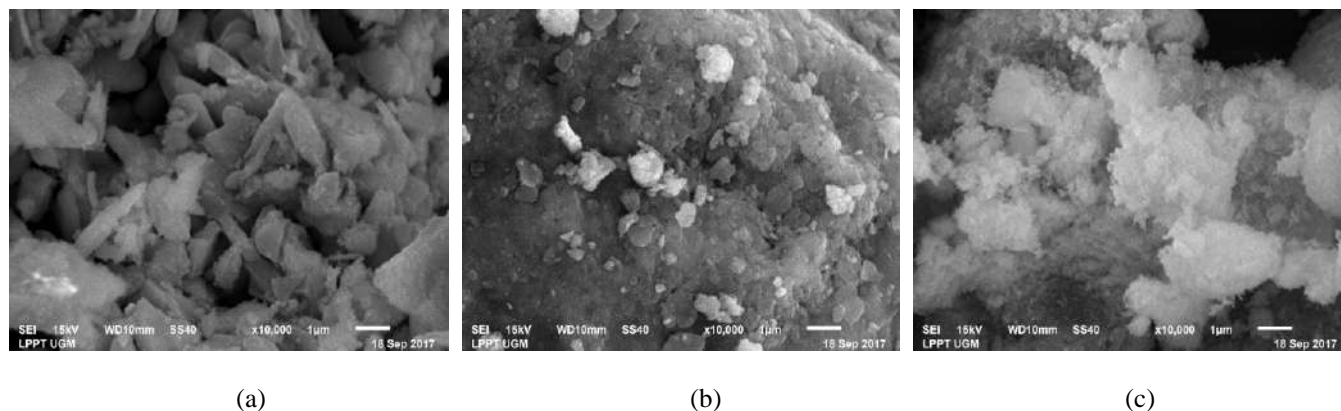


Fig. 3. SEM analysis at magnification 10,000 times (a) Ni-oxide, (b) Silica-alumina, (c) Fe-oxide

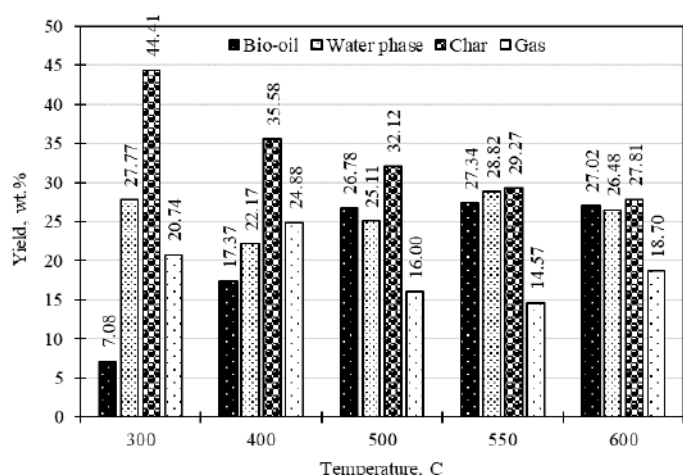


Fig. 4. The effects of temperature on the SPR non-catalytic pyrolysis yield

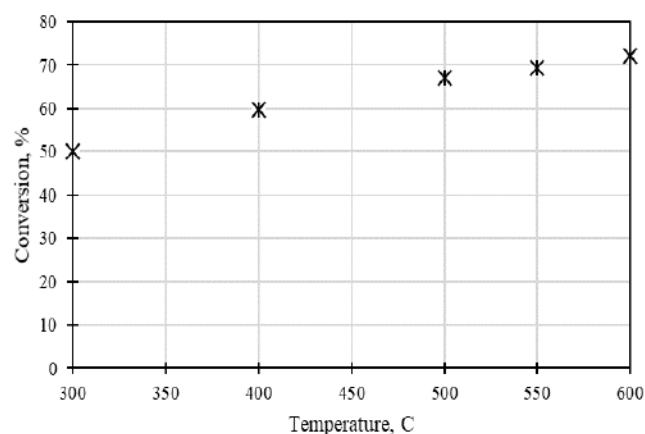


Fig. 5. The effects of temperature on conversion of the pyrolysis.

3.4. Catalytic Pyrolysis Result

The effects of catalyst on SPR pyrolysis using three types of catalyst: Ni-oxide, Fe-oxide, and silica-alumina with varying amounts of 5 - 30 wt.% at 550°C presented in Figure 5. At 550°C, the optimum temperature obtained in pyrolysis without a catalyst.

From Figure 6 (a), we can see the effects of the amount of catalyst on the yield of bio-oil products. Bio-oil mostly obtained from a 10% Fe-oxide catalyst. The amounts of bio-oil products at 550 °C with a variation of the quantity of catalyst 5-30% for Ni-oxides, silica-alumina, and Fe-oxides are respectively in the range of 18.86 - 23.38; 11.53 - 18.41, and 14.67 - 27 wt.%. It can conclude that the highest yield of bio-oil obtained by the use of a Fe-oxide catalyst, followed by Ni-oxide and the least was by silica-alumina.

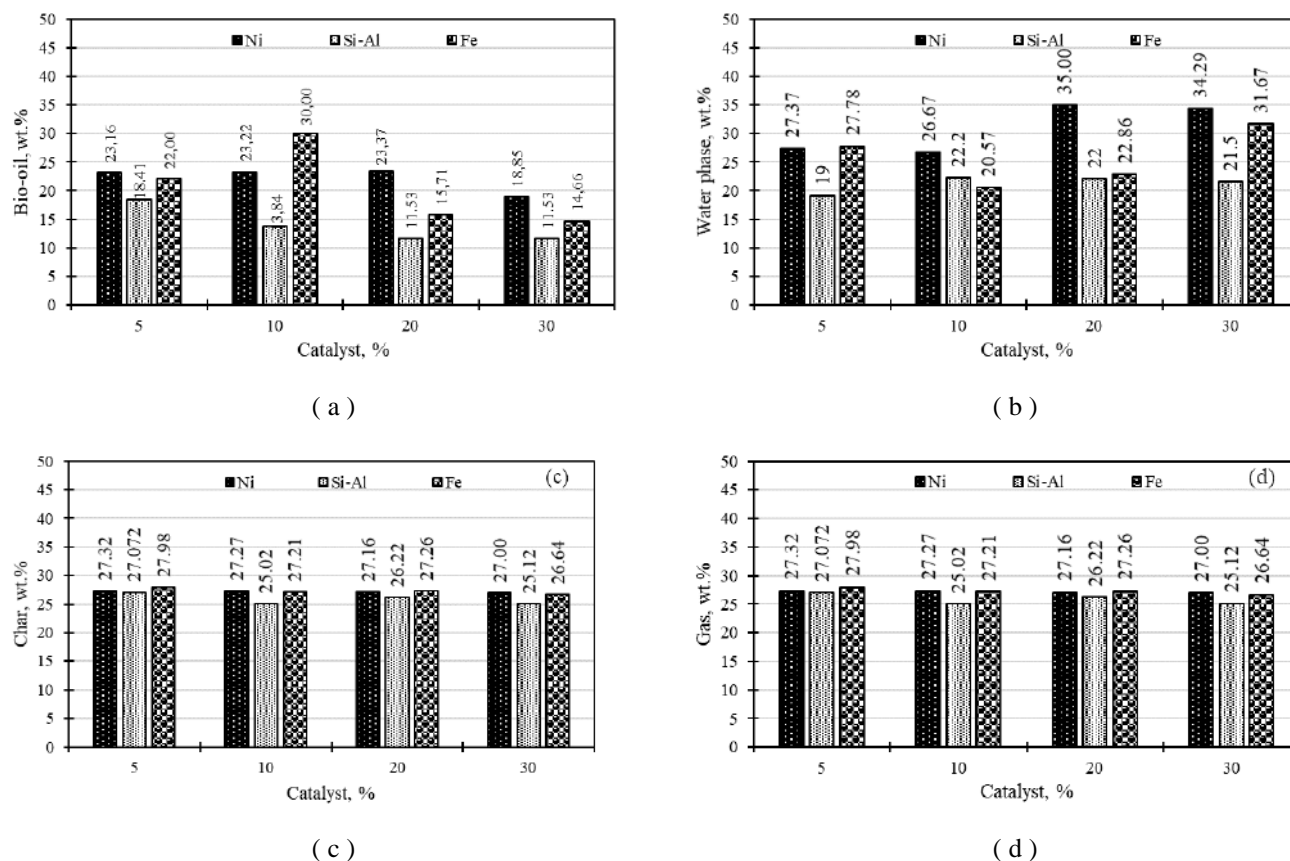


Fig. 6. The effects of catalyst (Ni oxides, silica-alumina, and Fe oxides) on product yields at 550°C: (a) Bio-oil; (b) Water phase; (c) Char and (d) Gas

The yield of the water phase in various types and amounts of catalyst can be seen in Figure 6 (b), the yield of the water phase for multiple types of catalysts at the same amount is significantly different. The range of water phase yields with catalysts Ni-oxides, silica-alumina, and Fe-oxides for the amount of catalyst 5-30% is 26.67 - 35, respectively; 19 - 22.2 and 20.57 - 31.67 wt.%. This difference in yield can be due to the water content in the SPR sample and the reactions of water formation during pyrolysis. The use of catalysts can increase the dehydration reaction, which produces a certain amount of water [38]-[42]

From Figure 6(c) and (d), it can see that the char and gas yield in the use of various types and amounts of catalyst, the yield of char and gas is relatively the same. For the yield of char produced in the use of Ni-oxide catalysts, silica-alumina, and Fe-oxide, the amounts are 27 - 27.32%; 25.02 - 27.07% and 26.64 - 28 %, Meanwhile, for gases, the measures are respectively 14.47 - 22.84%; 35.52 - 41.85% and 22.24 - 34.17 wt.%. The most gas produced is in pyrolysis using a silica-alumina catalyst, the next is Fe-oxide, and the least one is with Ni-oxide catalyst. According to [33], the Ni-oxide has effected on the activation energy.

Pyrolysis conversion can achieve by using various types and amounts of catalysts is presented in Figure 7. From this figure, it can see that the more catalyst used, the higher the conversion. Pyrolysis use Ni-oxide, silica-alumina, and Fe-oxide catalysts in the amount of 5 - 30% obtained in the range of 45.36 - 46.11%; 45.86 - 49.76% and 44.04 -

46.72%. The highest conversion is achieved in the pyrolysis of silica-alumina catalysts, followed by Ni-oxides and Fe-oxides with almost the same range.

4. Conclusion

The maximum pyrolysis yield in the non-catalytic pyrolysis is 27.34 wt.% at a maximum temperature of 550°C. Then yield dropped at 27.02 wt.% as the temperature increase in the temperature of 600°C. In the temperature ranging from 300 to 600°C, the yield of the water phase, char, and gas was 16.6 - 19.04 wt.%; 27.92 - 49.86 wt.% and 25.02 - 42.10 wt.%. The pyrolysis conversion will increase from 50.15 to 72.08 wt.% as the pyrolysis temperature increases.

Pyrolysis with the catalyst conducted at 550°C using various catalysts (Ni-oxides, silica-alumina, and Fe-oxides), and the amount of catalyst was 5 - 30 wt. %. The highest yield of bio-oil was found from the use of Fe-oxide catalysts in the range of 14.67 - 27 wt.%, followed by Ni-oxide of 18.86 - 23.38 wt.% and the lowest by silica-alumina of 11.53 - 18.41 wt.%. The water phase yield was significantly different from each catalyst. The range of water phase yields with catalysts Ni-oxides, silica-alumina, and Fe-oxides for the amount of catalyst 5-30% are 26.67 - 35 wt.%; 19 - 22.2 wt.% and 20.57 - 31.67 wt.% respectively.

The char and gas yield was relatively similar. The yield of char produced in the use of Ni-oxide catalysts, silica-

alumina, and Fe-oxide was 27 - 27.32 wt.%; 25.02 - 27.07 wt.% and 26.64 - 28 wt.% respectively. While gases yields were 14.47 - 22.84 wt.%; 35.52 - 41.85 wt.% and 22.24 - 34.17 wt.% respectively. Most of the gas produced was in the use of silica-alumina catalyst, then Fe-oxide, and Ni-oxide catalyst.

The more amount of catalyst used, the higher the conversion. Conversions with Ni-oxide, silica-alumina, and Fe-oxide catalysts are in the range of 45.3 - 46.11 wt.%; 45.86 - 49.76 wt.% and 44.04 - 46.72 wt.%. The highest conversion obtained using silica-alumina catalysts, followed by Ni-oxides and Fe-oxides with almost the same range.

Acknowledgments

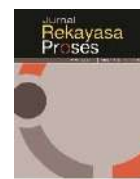
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Biochar from Slow Catalytic Pyrolysis of *Spirulina platensis* Residue: Effects of Temperature and Silica-Alumina Catalyst on Yield and Characteristics

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ABSTRACT

The use of biochar varies on its ability as an adsorbent which adsorbs liquid or gas molecules. Biochar from *Spirulina platensis* residue (SPR) as an energy source, as its richness in nutrients, can be used as fertilizer and maintain water resources in plantations. Biochar can be used as an intermediary for the synthesis of nanotubes, activated carbon, carbon black, and carbon fiber. One of the essential things to be considered in the application of activated carbon from SPR is char's characteristics. This study aimed to obtain data on the biochar and components from the pyrolysis of *Spirulina platensis* residue. The study was conducted in a fixed-bed reactor with electric heaters with a variety of temperatures (300-700 °C) and the amount of silica-alumina catalyst (0-20%). The biochar weight was obtained by weighing the char formed at the end of the pyrolysis. The char characteristics were obtained by the surface area, total pore volume, and pore size analysis. Based on the study results, the relationship between temperature and the amount of catalyst on the characteristics of biochar was studied. The higher the pyrolysis temperature, the less biochar. Also, the use of catalysts can reduce the amount of biochar. The higher the temperature, the higher the surface area and the total pore volume while the pore radius was reduced. The optimum condition for maximum biochar yield in non-catalytic pyrolysis at a temperature of 300 °C was 49.86 wt.%. The surface area, the total pore volume, and the pore radius at 700 °C catalytic pyrolysis with 5% silica-alumina was obtained as 36.91 m²/g, 0.052 cm³/g, and 2.68 nm, respectively.

Keywords: biochar; pore radius; silica-alumina; surface area; total pore volume

ABSTRAK

Penggunaan biochar bervariasi pada kemampuannya sebagai adsorben dalam menyerap molekul cairan atau gas. Biochar dari residu *Spirulina platensis* merupakan sumber energi, karena kaya akan unsur hara, dapat digunakan sebagai pupuk dan pemeliharaan sumber daya air di perkebunan. Biochar dapat juga digunakan sebagai perantara untuk sintesis nanotube, karbon aktif, carbon black, dan serat karbon. Salah satu hal penting yang harus diperhatikan dalam aplikasi karbon aktif dari SPR adalah karakteristik arang. Penelitian ini bertujuan untuk mendapatkan data biochar dan komponen dari pirolisis residu *Spirulina platensis*. Penelitian dilakukan di reaktor fixed-bed dengan pemanas listrik dengan variasi suhu (300-700 °C) dan jumlah katalis silika-alumina (0-20%). Berat biochar diperoleh dengan cara menimbang arang yang terbentuk pada akhir pirolisis. Sedangkan karakteristik arang diperoleh dari analisis luas permukaan, volume pori total, dan ukuran pori. Berdasarkan hasil studi hubungan antara suhu dan jumlah katalis terhadap karakteristik biochar yang telah diteliti, semakin tinggi suhu pirolisis maka biochar semakin sedikit. Selain itu, penggunaan katalis dapat mengurangi jumlah biochar. Sebaliknya, semakin tinggi suhu semakin besar luas permukaan, dan volume pori total serta radius pori-pori semakin berkurang. Kondisi optimum untuk biochar maksimum pada pirolisis non katalitik pada suhu 300 °C adalah 49,86 wt.%. Luas permukaan, total volume pori, dan radius pori pada suhu 700 °C untuk pirolisis katalitik silika-alumina 5% diperoleh masing-masing sebesar 36,91 m²/g, 0,052 cm³/g, dan 2,68 nm.

Kata kunci: biochar; luas permukaan; radius pori; silika-alumina; total volume pori

1. Introduction

Pyrolysis is the most studied thermochemical technology to date and has proven to be one of the best techniques for producing biofuels and biochar from biomass feedstocks (Jamilatun et al., 2019; Li et al., 2016; Tripathi et al., 2016). Biomass sources influence biochar production through pyrolysis, biomass properties (e.g., particle size and moisture content), composition (e.g., cellulose, lignin, and ash content), and process parameters (e.g., temperature, heating rate, residence time) (Yu et al., 2017a). Dickerson and Soria (2013) explained the process parameters for slow pyrolysis; the heating rate is 0.1-1 °C/sec with residence time in the range of minutes to hours, and temperatures between 400-600 °C will produce around 33% char, 32% tar, and 35% gas. The intermediate pyrolysis at 400-500 °C,

a heating rate at 1-1000 °C/sec, hot vapor residence at 10-30 seconds will produce 25% char, 50% tar, and 25% gas. In contrast, fast pyrolysis can provide 12% char, 75% tar, and 13% gas with a heating rate of 10 to more than 1000 °C/sec, a residence time of fewer than 2 seconds, and an optimum temperature between 400-650 °C (Jamilatun et al., 2017; Suganya et al., 2016).

Non-catalytic pyrolysis produces low-quality liquid products with a relatively high oxygenated compound content, which can cause corrosion to the engine. Reduction of oxygenate compounds can improve quality; another way is to use a catalyst during pyrolysis (Jamilatun et al., 2019). One of the catalysts commonly used for cracking hydrocarbons is silica-alumina, the solid acid catalyst most widely used in supporting the production of petrochemicals, chemicals, and renewable energy. High acidity (low Si/Al) can

be used in the process of cracking petroleum; its function is to increase oxidation of CO (Wang et al., 2019). The silica-alumina catalyst is suitable for upgrading bio-oil, has a high melting point (1818 °C) and surface area (Cheng et al., 2016; Duan et al., 2013). The catalytic pyrolysis results can improve bio-oil and biochar; it is essential to know the yield and characteristics (surface area, total pore volume, pore radius) of biochar produced in biochar application.

Microalgae is currently a third-generation raw material for biofuel production. It also produces several pharmacologically necessary and nutritious chemicals such as pigments and fatty acids. The simultaneous production of biofuel raw materials and fine chemicals in microalgae biorefinery can improve the economy (Elkhalifa et al., 2019). Biochar from the pyrolysis of microalgae has a lower surface area and carbon content than biochar from lignocellulose. However, biochar has excellent characteristics such as higher pH, its ability to balance soil acidity, and higher nutrient content, including minerals such as nitrogen, ash, and inorganic elements compared to another biomass. Other characteristics of biochar from microalgae such as surface area, total pore volume, and pore radius are still rarely discussed; for this reason, it is necessary to identify with the BET method (Chen et al., 2018; Ido et al., 2019).

Biochar is a solid residue from pyrolysis, formed from primary and secondary pyrolysis reactions, containing carbon and stable elements with high carbon content. The use of biochar varies because of its inert nature and ability to absorb liquid or gas molecules, as a gasification substrate for energy generation. It has excellent potential to be used directly for combustion because it emits

much lower CO₂ with higher energy than fossil fuels (Lee et al., 2020). Biochar can be used directly as a solid form of biofuel without the complicated extraction process required by liquid-based biofuels. Another advantage of biochar is that the nutrients can be used as fertilizer and to maintain water resources on plantations. As an intermediate material, biochar can be used as a raw material for the synthesis of nanotubes, activated carbon, carbon black, and carbon fiber (Lee et al., 2020).

Biochar with a relatively high surface area can be used as a catalyst and for wastewater treatment. Some biochar from microalgae such as *Chlorella sp.*, *Chlamydomonas sp.*, *Coelastrum sp.*, *Spirulina platensis* has a surface area of 6.163, 2.122, 15.032 and 167 m²/g with pyrolysis time of 0.5, 0.5, 0.5 and 2 hours, respectively (Choi et al., 2020). Wang et al. (2013) reported that the surface area of biochar originating from *Chlorella vulgaris* (2.4 m²/g), macroalgae *Eucheuma sp.* has a much higher surface area (30.03-34.82 m²/g) than other species ranging from 1.29 to 8.87 m²/g (Yu et al., 2017b).

Research on the characteristics of biochar from microalgae and their applications is still not widely found, therefore data on pore area, total pore volume, and pore radius is essential to determine the superiority of biochar from microalgae. This paper aims to study the effect of temperature and the amount of silica-alumina catalysts on product yields and product biochar characteristics from the pyrolysis of residual *Spirulina platensis* (SPR). Biochar characteristics were analyzed by the Brunauer-Emmett-Teller (BET) method and catalysts by the BET method and XRF. The SPR is obtained from solid residue extraction of *Spirulina platensis*. The study was conducted

with fixed bed reactors with a variety of temperatures (400-700 °C), and the amount of silica-alumina catalyst (5-20 %). The catalyst was in the form of pellets. Based on the characteristic data obtained, it is expected that there will be a follow-up treatment from biochar so that it can be appropriately utilized.

2. Research Methodology

2.1 Materials

Dry *Spirulina platensis* residue (SPR) was obtained from *Spirulina platensis* (SP) solid residue extraction, while SP was obtained from Nogotirto Algae Park, Yogyakarta, Indonesia. Wet *Spirulina platensis* residue (SPR) was first sun-dried for three days, then cleaned, and stirred for homogenization. The SPR was stored in a dry and closed place. Then, the SPR was analyzed for the ultimate and proximate content (Jamilatun et al., 2019).

Silica-alumina was obtained in powder form from PT Pertamina Balongan, Indramayu, Indonesia. For applications in pyrolysis, it needs to be formed in the form of pellets. Pellets were created by mixing silica-alumina (95 wt.%) with kaolin (5 wt.%). Distilled water was added to the homogeneous mixture and it was formed into pellets of 4 mm in diameter and 6 mm high. The catalyst pellet was dried in a furnace at 500 °C for 2 hours, then cooled in a desiccator.

2.2 Procedures

Fifty (50) g SPRs were put into the reactor, then tightly closed, and heated with electricity. Temperature controlled with a NiCr-Ni thermocouple placed outside the reactor. Heating was carried out at a heating rate of 5-35 °C/minute from room temperature (30 °C) to the desired

temperature (300-700 °C). Pyrolysis gas was condensed, liquid products coming out of the condenser were collected in the accumulator, and the amount of gas production was measured. Biochar products were obtained after the experiment was completed. The amount of biochar was measured by weighing. The biochar yields were calculated by Equation (1).

2.3 Instrument and data analysis

The experiment was carried out in a fixed-bed reactor made of stainless steel with the inner diameter of 40 mm, the outer width of 44 mm, and the height of 600 mm, equipped with a heater of nickel wire wrapped around the reactor's outer cylinder (Jamilatun et al., 2019). The pyrolysis system's diagram with the fixed-bed reactor is presented in Figure 1.

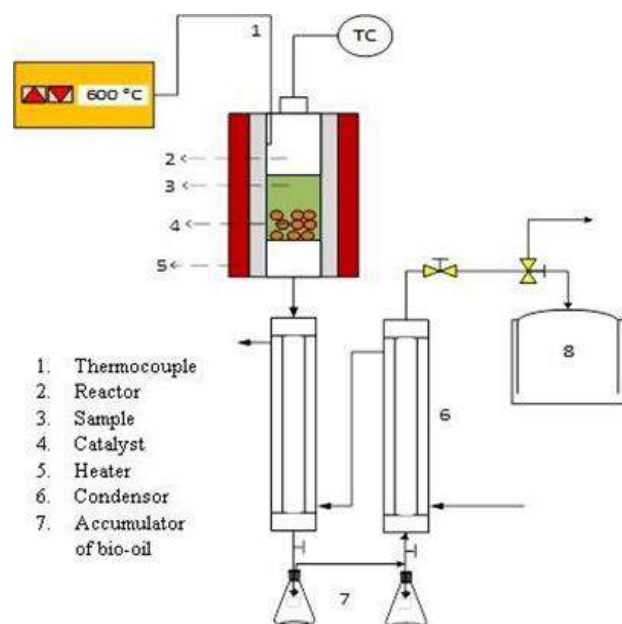


Figure 1. The Pyrolysis system of SPR (Jamilatun et al., 2019)

The reactor is constructed of two cylindrical reactors in vertical series (Reactors 1 and 2). *Spirulina platensis* residue of 50 g is put into the top of reactor one and the catalyst to the bottom of the two reactors,

then tightly closed and heated. The temperature of the reactor was controlled by a NiCr-Ni thermocouple that was placed outside of the reactor. The SPR samples were heated with a heating rate in the range of 5-35 °C/min from 30 °C to the desired temperature range of 300-700 °C. The condenser unit condensed the pyrolysis gas. The liquid yield was collected in the accumulator, and the produced gas was measured. After the experiment finished, the remaining solid product (biochar) was taken and weighed. The bio-oil yields were calculated by Equation 1.

2.3.1 Biochar Yield

The biochar yield was calculated using Equation (1).

$$Y_C = \frac{W_C}{W_M} \times 100\% \quad (1)$$

In this case, Y_C notation is the yield of charcoal products, while W_M and W_C are the initial SPR weighting and charcoal weight, respectively.

2.3.2 Sample of *Spirulina platensis* residue

Spirulina platensis residue sample analysis included proximate, ultimate, and higher heating value (HHV). Proximate analysis (protein with the Kjeldahl method; carbohydrates with the Anthrone method; lipids with the Soxhlet method) and HHV (Bomb calorimeter) were carried out at the Laboratorium Pangan dan Hasil Pertanian, Departemen Teknologi Pertanian dan Laboratorium Pangan dan Gizi, Pusat Antar Universitas (PAU), UGM Indonesia. The ultimate analysis (C, H, O, N, and S with a standard D 2361) was conducted at the Laboratorium Pengujian, Puslitbang Tekmira, Bandung Indonesia.

2.3.3 Silica-Alumina

Measurement of the surface area, total pore volume, and radius pore were carried out using the BET method (Brunaur, Emmett, and Teller) by Quantachrome NovaWin - Data Acquisition and Reduction NOVA instruments 1994-2013, Quantachrome Instruments version 11.03. The content of C, O, Al, Si, and silica-alumina ratio $\text{SiO}_2/\text{Al}_2\text{O}_3$ with SEM-EDX (Scanning Electron Microscope-Energy Dispersive X-ray), each conducted at the Laboratorium Penelitian dan Pengujian Terpadu (LPPT), UGM. (Jamilatun et al., 2019). The X-ray fluorescence (XRF) analysis was carried out at The International Frontier Division, Dept. Transdisciplinary Science and Technology School of Environmental and Society, Tokyo Institute of Technology, Japan. The XRF microscopy analyzes were carried out at normal atmospheric pressure with currents of 50 kV and 1.0 mA, in the Horiba Scientific XGT-5200 Analytical X-ray Microscope, with high spatial resolution from 1.2 mm to 10 μm .

2.3.4 Biochar Product

Prior to the adsorption test, biochar was degraded at 150 °C for 10 hours. The specific surface area of BET (S_{BET}) was determined by the Brunauer-Emmett-Teller equation (BET). Total pore volume (V_{total}) was established by a single point adsorption total pore volume analysis. The $4V/S_{\text{BET}}$ determines the average pore diameter (D) based on the BET method (Chen et al., 2018). BET analysis was carried out at the Laboratorium Analisis Instrumental (ANINS), Chemical Engineering Department, UGM by Quantachrome Nova Win-Data Acquisition and Reduction for NOVA instruments 1994-2013, Quantachrome Instruments version 11.03.

3. Results and Discussions

3.1 Analysis Results

The SPR sample ultimate analysis revealed the percentage of C, H, N, and O of 41.36, 6.60, 7.17, and 35.33 wt.%, respectively. On the other hand, the proximate analysis resulted in the lipids, proteins, and carbohydrates composition of 0.09, 49.60, and 38.51 wt.%, respectively. While the HHV of the SPR is 12.8 MJ/kg.

Silica-alumina was analyzed by Scanning Electron Microscope-Energy Dispersive X-ray (SEM-EDX). The results are shown in Figure 2.

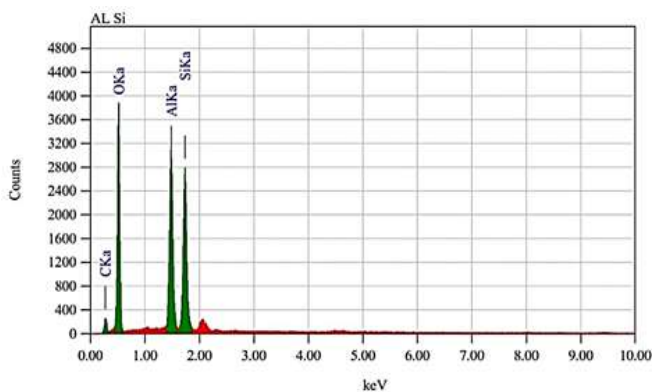


Figure 2. Silica-alumina SEM-EDX results

Figure 2 shows the magnitude of the voltage (keV) used, and each catalyst constituent (C, O, Al, and Si). Si obtained at a voltage of 1.739 keV with a complete counting of 2400 counts, while Al is received at a voltage of 1.486 keV with 6800 counts. Based on the ZAF Standardless Quantitative Analysis Method and using the Fitting Coefficient: 0.0684, the weight percent values of C, O, Al, and Si were 12.33, 55.73, 15.42, and 16.51%, respectively. Meanwhile, from the BET measurement, surface area, average pore volume, and pore diameters were 240.53 m²/g, 0.199 cm³/g total pore volume, and 3.3 nm. The XRF analysis results of SiO₂, Al₂O₃, and ratio SiO₂/Al₂O₃ were 60.28 wt., 35.25 wt.,

and 1.71%, respectively (Jamilatun et al., 2019).

3.2 Biochar Yield

The biochar yield data of pyrolysis with fixed-bed reactors at various temperatures and the amount of silica-alumina is shown in Figure 3

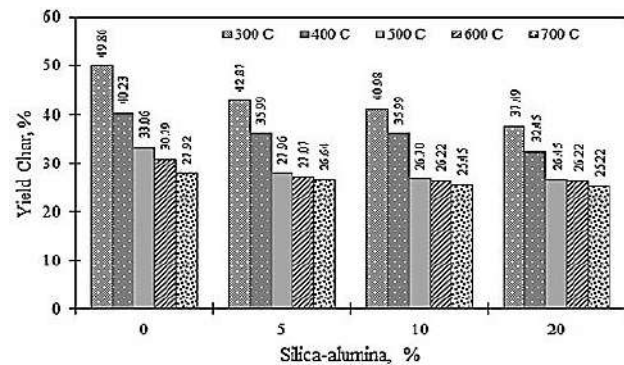


Figure 3. The influence on temperature and amount of catalyst on biochar yield

Based on Figure 3, the pyrolysis temperature rise of non-catalytic and catalytic pyrolysis has a similar trend. The increasing pyrolysis temperature will affect the biochar yield to decrease further. The increase in pyrolysis temperature will reduce the biochar yield. It causes thermal cracking of the heavy hydrocarbon material, leading to an increase in fluids and gases and a decrease in the biochar yield. The primary pyrolysis reaction happens at low temperature, and decomposition occurs gradually to produce charcoal. While at high temperatures, rapid evaporation occurs, which leads to the formation of volatiles. The high temperature causes a secondary pyrolysis reaction. The biochar formed during the primary reaction is decomposed into liquefied and non-condensed gas, reducing the charcoal (Tripathi et al., 2016). In non-catalytic pyrolysis, temperature increases from 300,

400, 500, 600, and 700 °C will produce biochar of 49.86, 40.23, 33.06, 30.79, and 27.92%, respectively.

The addition of silica-alumina to pyrolysis will increase secondary reactions, causing a slight decrease in biochar. In the secondary reaction, the tar will undergo further decomposition into gas to increase the amount of non-condensable gas yield and reduce the amount of condensable-gas and biochar yield. At the temperatures of 300 and 400 °C, the increasing amount of catalyst has little effect on yield decreasing of biochar. Whereas at 500-700 °C, the use of catalysts (5, 10, and 20%) had no impact on reducing the charcoal amount. The optimum conditions of pyrolysis to produce high biochar yield is achieved at a temperature of 300 °C without a catalyst. However, to produce biochar with better characteristics (surface area, total pore volume, and pore radius), a catalyst is recommended.

3.3 Biochar Surface Area

The effect of temperature and the amount of silica-alumina on the biochar surface area is shown in Figure 4. Based on Figure 4, the impact of temperature rise for both non-catalytic and catalytic pyrolysis is quite significant on the surface area. As the temperature gets higher, the surface area tends to increase. The optimum condition is obtained at 700 °C for non-catalytic, the biochar surface area is 23.45 cm²/g, while for catalytic pyrolysis (5% silica-alumina) the surface area is 36.91 m²/g. The biochar surface area for the use of 5% silica-alumina at 400, 500, 600, and 700 °C was 3.99, 4.18, 23.45, and 36.91 m²/g, respectively.

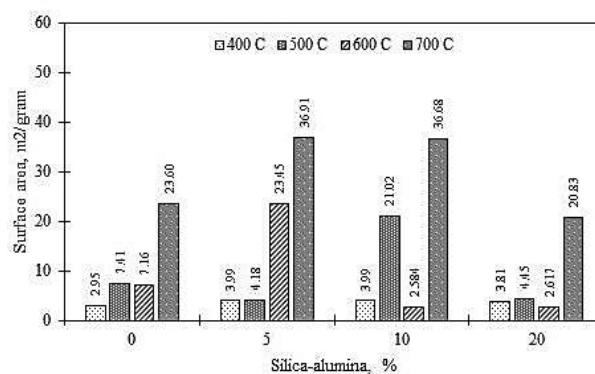


Figure 4. The influence of silica-alumina catalyst on surface area biochar

Microalgae-based biochar appears to be promising in terms of energy potential because of its higher HHV range (14.50-36.67 MJ/kg) and relatively higher surface area (up to 80 m²/g). The content of various functional groups and inorganic elements in biochar is useful in the process of adsorption for environmental control (Lee et al., 2020). Based on the surface area data obtained, it can be interpreted that biochar from microalgae has the potential to be developed into activated charcoal with physical or chemical activation to achieve a larger size of the surface area. The choice of pyrolysis temperature needs to be considered to obtain high biochar yield and surface area. According to Zheng et al. (2017), the pyrolysis of *Chlorella sp.* at a temperature of 600 °C produces biochar with a surface area of 6.16 m²/g. Wang et al. (2013) reported that the biochar surface area of *C. Vulgaris* was 2.40 m²/g, while Roberts et al. (2015) reported on biochar produced from macroalgae *Eucheuma sp.* has a much higher surface area (30.03-34.82 m²/g) than other species ranging from 1.29 to 8.87 m²/g. Compared to this study, the biochar produced by SPR pyrolysis with silica-alumina at the same temperature, namely 600 °C, has a much higher surface area, namely 36.91 m²/g.

3.4 Biochar Total Pore Volume

The effect of temperature and the amount of silica-alumina on the total pore volume are shown in Figure 5. The temperature and the amount of catalyst have significant effects on the total pore volume at 700 °C. The optimum total pore volumes were obtained at a temperature of 700 °C in non-catalytic and catalytic pyrolysis with 5, 10, and 20 % silica-alumina were 0.036, 0.052, 0.052, and 0.041 cm³/g, respectively. The total pore volume data of biochar from microalgae are not published as much as the data for the pore area. However, Chen et al. (2018) report that the pyrolysis of *Spirulina platensis* at 600 °C with a barium (Ba) catalyst produces a total pore volume of 0.004 cm³/g.

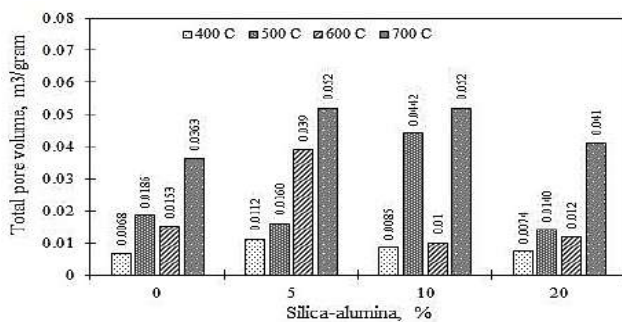


Figure 5. The influence of temperature and the amount of silica-alumina catalyst on total pore volume

3.5 Biochar Pore Size

The effect of temperature and the amount of alumina-silica catalyst on the biochar radius can be seen in Figure 6. Based on this figure, the pore radius for temperatures of 300-400 °C from non-catalytic and catalytic pyrolysis has the same trend. The rising temperature will increase the pore radius, but at a temperature of 700 °C, the pore radius drops sharply. The correlation between surface area and pore radius is that the higher temperature increases the surface area;

otherwise, the pore radius will decrease (Yu et al., 2017b). The pore radius ranges resulted from 400-700 °C for non-catalytic and catalytic pyrolysis with 5%, 10%, and 20% silica-alumina were 2.68-2.70, 2.68-2.72, 2.69-2.71 and 2.68-2.70 nm. The biochar product from non-catalytic and catalytic pyrolysis SPR leads to mesoporous sizes of 2-50 nm. Chen et al. (2018) reported that the biochar product of *Spirulina platensis* from catalytic pyrolysis with Barium (Ba) has the characteristics of pore area, total pore volume, and pore radius of 0.09 m²/g, 0.04 m³/g, and 177 nm, respectively.

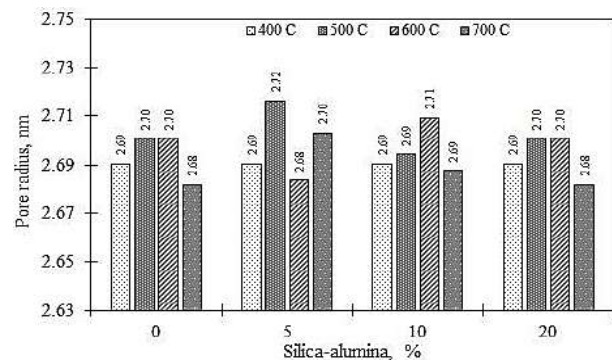


Figure 6. The influence of temperature and the amount of silica-alumina catalyst on pore radius biochar

3.6 Comparison of SPR Biochar Characteristics with the Other Findings

Table 1 compares the process of pyrolysis of different microalgae and the biochar products produced in fixed-bed reactors with slow pyrolysis. The pyrolysis of brown macroalga *Laminaria japonica* and green macroalgae *Cladophora glomerata* shows that the higher the pyrolysis temperature, the lower the yield of biochar (Jung et al., 2016), while the surface area is increasing (Bordoloi et al., 2016; Norouzi et al., 2016). Bordoloi et al. (2016) reported that an increase in temperature from 300-600 °C affected the increase in surface area from 1.72 to 123 m²/g.

Table 1. The pyrolysis process and characteristic biochar microalgae (Yu et al., 2017)

Pyrolysis Process	Biomass feedstock	Temperature (°C)	Biochar production	References
Slow pyrolysis	<i>Brown Laminaria japonica</i> macroalgae	200–800	<ul style="list-style-type: none"> • 78.34% at 200 °C • 63.64% at 400 °C • 37.96% at 600 °C • 27.05% at 800 °C 	Jung et al. (2016)
Fixed-bed pyrolysis	<i>Scenedesmus dimorphus</i>	300–600	Surface area of <i>biochar</i> increased from 1.72 to 123 m ² /g when temperature increased from 300-500 °C; reduced to 89 m ² /g at 600 °C	Bordoloi et al. (2016)
Fixed-bed pyrolysis	Green macroalgae <i>Cladophora glomerata</i>	400–600	<ul style="list-style-type: none"> • 44 wt% yield at 400 °C • 40 wt% yield at 500 °C • 39 wt% yield at 600 °C 	Norouzi et al. (2016)
Fixed-bed pyrolysis Slow pyrolysis	<i>Spirulina platensis</i> residue	300-700	<ul style="list-style-type: none"> • Yield <i>biochar</i> at catalyst 0, 5, 10 and 20 % in the range 27.92-49.86, 26.64-42.87, 25.45-40.98, and 25.22-37.49 %, respectively. • Catalyst 0, 5, 10 and 20 % (700 °C) surface area of 31.47, 36.905, 36.667, 20.826 m²/g, respectively • Catalysts with 0, 5, 10, and 20% (700 °C) total pore volume of 0.036, 0.052, 0.052 and 0.041 cm³/g, respectively • Catalysts with 0, 5, 10, and 20 % (700 °C) pore radius of 2.682-2.701, 2.684-2.717, 2.688-2.709 and 2.682-2.701 nm, respectively 	This experiment

The results of non-catalytic and catalytic SPR pyrolysis research at the temperatures of 300-700 °C with the variations of 0, 5, 10, and 20% alumina-silica catalysts yielded biochar yields in the range of 27.92-49.86, 26.64-42.8, 25.45 -40,98, and 25.22-37.49%, respectively.

Based on the yield of biochar, the optimum conditions in non-catalytic pyrolysis were obtained, at a temperature of 300 °C with 49.86 wt.% yield. Based on the characteristics of biochar, the optimum conditions for catalytic pyrolysis was obtained with 5% silica-alumina catalyst at 700 °C, which resulted in the surface area, total pore volume, and pore radius of 36.91 m²/g, 0.052 m³/g, 2.68 nm, respectively.

4. Conclusions

Non-catalytic pyrolysis produces bio-oil with a high content of oxygenated

compounds, so the use of silica-alumina catalysts improves the bio-oil quality. Catalytic pyrolysis will affect the quality of biochar, such as surface area, total pore volume, and pore radius. Non-catalytic and catalytic pyrolysis of *Spirulina platensis* residue (SPR) was carried out in a fixed bed reactor. The results of pyrolysis at the temperatures of 300, 400, 500, 600 and 700 °C with silica-alumina catalyst varied at 0, 5, 10 and 20% were biochar yields in the range of 27.92-49.86, 26.64-42.87, 25.45-40.98 and 25.22-37.49%; the surface area in the range of 2.95-23,60, 3.99-36.91, 3.99-20.83; the total pore volume in the range of 0.007-0.036, 0.011-0.052, 0.009-0.052 and 0.007-0.041 m³/g; and the pore radius in the range of 2.682-2.701, 2.684-2.717, 2.688-2.709 and 2.682-2.701 nm, respectively. The optimum condition for producing the maximum

biochar yield is non-catalytic pyrolysis at the temperature of 300 °C, which is 49.86 wt.%. However, to obtain better biochar characteristics, this condition does not apply. Based on the biochar characteristics, the optimum conditions were obtained at 700 °C by catalytic pyrolysis with 5 % of silica-alumina, which obtained the surface area, total pore volume, and radius pore as 36.91 m²/g, 0.052 m³/g, 2.68 nm, respectively.

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