OPTIMIZATION THE SYNTHESIS CONDITION AND CHARACTERIZATION OF METHYLCELLULOSE OBTAINED FROM JACK BEAN SEED HULL

(*Canavalia ensiformis* L. (DC.))

ABSTRACT

Jack bean (*Canavalia ensiformis* L. (DC.)) has a seed hull containing lignocellulose component. Isolation of cellulose from that hull and synthesis of its derivates expected to increase the economic value and supporting zero waste program. One of cellulose derivates which was widely used in food industry is methylcellulose (MC). The aims of this study are : (1) to identify the effect of NaOH concentration, volume of dimethyl sulphate as methylation agent, and time of methylation on methylcellulose synthesis toward degree of subtitution (DS) (2) to optimize the synthesis condition of foodgrade methylcellulose based on combination of degree substitution (DS), solubility, water holding capacity, and oil holding capacity, and (3) to characterize the methylcellulose obtained from optimum condition.

Cellulose isolated from jack been's seed hull is used as material for methylcellulose synthesis. Synthesis of methylcellulose is started from determining the solvent and the zero point in various NaOH concentration, volume of dimethyl sulphate, and time of methylation. Optimization of methylcellulose synthesis condition obtained by *Response Surface Methodology* (RSM) *central composite design* at NaOH concentration 27,59; 31; 36; 41, and 44,41% (w/v), volume of dimethyl sulphate 3,32; 4, 5, 6, and 6,68 ml, and time of methylation 4,32; 5; 6; 7; and 7,68 hours with remethylation in same condition, based on combination of degree substitution (DS), solubility, water holding capacity, and oil holding capacity. Then, methylcellulose obtained from the optimum synthesis condition is characterized of its degree of subtitution (DS), solubility, water holding capacity (WHC), oil holding capacity (OHC), viscosity of 2% solution, moisture content, ash content, lightness, pH 1% solution, cellulose yield, loss of drying, functional group, cristallinity index, and the ability to form thermoreversible gel.

Characteristic of jack been's seed hull cellulose similar with commersial cellulose with content of cellulose 83,93%(w/w), lignin 3,11%(w/w), and hemicellulose 6,03%(w/w). MC can be synthesized from it with toluene as solvent. The increasing of NaOH concentration, volume of dimethyl sulphate, and time of methylation give the increase of DS until the certain point and then decrease gradually with the further increase of them. The optimum condition based on combination of degree of substitution, solubility, water holding capacity, and oil holding capacity response is NaOH 35,54%; 5,55 mL dimethyl sulphate; and 5,97 jam hours with remethylation in same condition. MC obtained from that synthesis condition has DS value 1,73±0,02, solubility 73,04±0,33%, WHC 15,32±0,18 g water/g dry sample, OHC 4,38±0,09 g oil/g dry sample, viscosity of 2% solution 363,23±5,77 cPs, moisture content 9,02±0,10%(db), ash content 2,79±0,31%(db), lightness 88,21±0,03, pH 1% solution 7,18±0,06, cellulose yield 105,21±2,15%, loss of drying 7,25±0,20%, cristallinity index 63,91%, and has ability to form thermoreversible gel at 60^{o}C.

Keyword : jack bean's seed hull, cellulose, methylcellulose, response surface methodology (RSM)

OPTIMASI SINTESIS DAN KARAKTERISASI METIL SELULOSA DARI KULIT KORO PEDANG PUTIH (*Canavalia ensiformis* L. (DC.))

INTISARI

Koro pedang putih (*Canavalia ensiformis* (L.) DC) memiliki kulit biji yang mengandung lignoselulosa. Isolasi selulosa dari kulit koro pedang putih dan pembuatan turunannya diharapkan mampu meningkatkan nilai ekonomi dan mengurangi pencemaran (*zero waste*). Turunan selulosa yang banyak digunakan dalam bidang pangan adalah metil selulosa (MC). Tujuan dari penelitian ini adalah (1) mengidentifikasi pengaruh variasi konsentrasi NaOH, konsentrasi dimetil sulfat (DMS), dan waktu metilasi pada sintesis MC terhadap derajat subtitusi MC, (2) mengoptimasi kondisi sintesis MC yang diklasifikasikan *food grade* berdasarkan kombinasi respon DS, kelarutan, w*ater holding capacity* (WHC), dan *oil holding capacity* (OHC), dan (3) mengkarakterisasi MC yang diperoleh dari kondisi optimum.

Selulosa diisolasi dari kulit koro pedang putih dan digunakan sebagai bahan baku pembuatan metil selulosa. Pembuatan metil selulosa diawali dengan penentuan pelarut terbaik kemudian penentuan titik nol pada 3 variabel yaitu konsentrasi NaOH, DMS, dan waktu metilasi. Optimasi kondisi sintesis MC diperoleh dengan *Response Surface Methodology* (RSM) *central composite design* dengan variasi NaOH yang digunakan yaitu 27,59; 31; 36; 41, dan 44,41% (b/v), variasi DMS 3,32; 4, 5, 6, dan 6,68 ml, dan waktu metilasi 4,32; 5; 6; 7; dan 7,68 jam dengan remetilasi pada kondisi yang sama berdasarkan kombinasi respon DS, kelarutan, w*ater holding capacity* (WHC), dan *oil holding capacity* (OHC). MC yang diperoleh dikarakterisasi derajat subtitusi, kelarutan, WHC, OHC, viskositas larutan 2%, kadar air, kadar abu, derajat putih (*lightness*), pH larutan 1%, rendemen, *loss of drying*, gugus fungsional, indeks kristalinitas, dan kemampuan pembentukan gel.

Berdasarkan penelitian, selulosa yang diperoleh memiliki karakteristik yang menyerupai selulosa komersial, dengan kadar selulosa sebesar 83,93%(b/b), dengan kandungan lignin dan hemiselulosa yang rendah yaitu 3,11%(b/b) dan 6,03%(b/b). MC dapat disintesis menggunakan selulosa yang diisolasi dari kulit koro pedang putih menggunakan pelarut toluene. Peningkatan konsentrasi NaOH, dimetil sulfat dan waktu metilasi akan meningkatkan derajat subtitusi MC. Namun, penggunaan konsentrasi NaOH, volume DMS, dan waktu metilasi yang terlalu tinggi akan menurunkan nilai derajat subtitusi. Kondisi optimum yang diperoleh berdasarkan kombinasi respon DS, kelarutan, WHC, dan OHC adalah 35,54% NaOH; 5,55 mL DMS; dan 5,97 jam dengan remetilasi dengan kondisi sintesis yang sama. MC kulit koro pedang putih tersebut memiliki karakteristik mendekati MC komersial, yaitu memiliki nilai DS 1,73±0,02, kelarutan 73,04±0,33%, WHC 15,32±0,18 g air/g sampel kering, OHC 4,38±0,09 g minyak/g sampel kering, viskositas larutan 2% 363,23±5,77 cPs, kadar air 9,02±0,10%(db), kadar abu 2,79±0,31%(db), derajat putih (*lightness*) 88,21±0,03, pH larutan 1% 7,18±0,06, rendemen 105,21±2,15%, *loss of drying* 7,25±0,20%, dan indeks kristalinitas 63,91% serta mampu membentuk gel *thermoreversible* pada suhu 60^{o}C.

Kata kunci : kulit biji koro pedang putih, selulosa, metil selulosa, *response surface methodhology* (RSM)