Tri-reforming of Methane and CO₂: A Novel concept for Catalytic Production of Solid Waste Syngas with Desired H₂/CO Ratios for Liquid Biofuels

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Project Abstract: Proposed is a novel tri-reforming process which involves a synergetic combination of CO₂ reforming, steam reforming and partial oxidation of methane in a single gasification reactor for cost effective production of industrially useful synthesis gas for use in Fischer-Tropsch synthesis (FTS). Municipal solid waste biomass gasification processes (H₂ and CO₂ are available in a 1:1 effluent) are just entering the early commercial phase and offer many opportunities for improvement. These improvements are urgently needed to reduce capital cost and facilitate commercial deployment, thus creating new industry and new employment for Florida. Here is directly where the proposed effort is targeted. The novel tri-reforming concept represents a new way of thinking for both conversion and utilization of CO2 and CH4 without separation that can be applied to industrial flue gas as well. The tri-reforming catalytic system proposed can not only produce synthesis gas $(CO + H_2)$ with desired H₂/CO ratios (1.5– 2.0), but also could eliminate carbon formation which is usually a serious problem in the CO_2 reforming of methane. Therefore, the proposed tri-reforming can solve two important problems that are encountered in individual processing. The incorporation of low partial pressures of O2 in the partial oxidation reaction generates heat *in-situ* that can be used to increase energy efficiency and O₂ also reduces or eliminates the carbon formation on the reforming catalyst. The selection of catalyst support is critical; a justification and explanation is presented. Our group at USF has already developed a process that converts MSW to Diesel and JP-8. This project will optimize and leverage this effort.

Project Impact: Municipal solid waste (MSW) biomass offers tremendous opportunity as a major, near-term, carbon-neutral energy resource. Florida has more MSW biomass resources than any other state, $\sim 7\%$ of the U.S. total. As such, harnessing these resources should be a key component of Florida's energy strategy. Projecting a future use of 7.5 million acres for biomass production in the State of Florida and forecasting an annual production



of 30 barrels per acre of bioenergy liquid fuels, one can project annual revenues of \$22.5 billion annually. Assuming that liquid fuel conversion will require the annual labor input of one person to generate \$225,000 of revenues, one arrives at a figure of 100,000 new jobs relative to converting bioenergy into fuel liquids. *Another very attractive function of the catalyst proposed is with flue gases from electric power plants.* The tri-reforming concept represents a new way of

thinking both for conversion and utilization of CO_2 and CH_4 in effluent biomass gasification without separation, and for production of industrially useful synthesis gas with desired H₂/CO ratios for FTS.

Dissemination and Technical Commercialization: Disseminating knowledge gained through publications in refereed journals and presentations at local, national and international meetings is paramount. Past performance in this respect attests to this commitment. Targeted conferences include MRS, AVS, ACS, AIChE and others are suitable and anticipated. Technical and technology commercialization will be pursued via the USF Office for Technology Development and the Clean Energy Research Center (CERC) at USF to develop, evaluate and promote commercialization of new environmentally clean energy sources and systems.

		Start	Month		Deliverable	
No.	Task/Activity Description			Deliverables/ Outputs	/ Output	
	Description	WIOIIUI	Complete		Due Dates	
1	Experimental Studies on	1	9	Yield and kinetic data	9th month	
	Bio-gasifier generated			on biomass gasification,		
	syngas composition			and overall economic		
				feasibility		
2	Development and design	3	11	Characterization and	11 th month	
	of the tri-reforming Ni-			activity analysis of the		
	based catalyst system			developed catalytic		
				system		
3	Optimization of catalyst	4	12	Initial yield and	12 th month	
	utilizing a PFR with			composition data on		
	controlled feed			H ₂ :CO ratios obtained		
4	Experimental studies	6	12	Yield and composition	12 th month	
	utilizing actual biomass			data on H ₂ :CO ratios		
	generated syngas			obtained, catalyst		
				activity and lifetime		
5	Commercial Scale Plant	6	12	Plant Design Report and	12 th month	
	Design and Economic			Sensitivity Studies		
	Profitability Study					

Timeline & Deliverables

Existing Collaborators and External Support: The PI's are currently working in partnership with several academic as well as commercial collaborators. These include Drs. B. Joseph, Y. Goswami, V. Gupta, N. Alcantar, C. Miller, V. Bhethanabotla, and others at USF along with

Helena and Jason Weaver at UF. Commercial and national laboratory collaborators include Tino Prado of Prado engineering, Derek Benson of Catalyst Renewables, Shaun Reeve of Bio Energy, Inc., and Matt Yung, NREL and Paul Matter of PH Matter, and Justin Wang of Sud Chemie.

External support currently consists of pending proposals to NSF and DOE. Proposals to NSF for the MURI and RESTOR programs have and/or will be submitted in the next few months.

Where we are now...

Currently we have optimized a novel Fischer-Tropsch (FT) Co/SiO_2 catalyst and reactor design that is tunable, producing diesel and J-P8 jet fuel as shown in fig 1 below. We can produce any hydrocarbon cut required, from gasoline to jet fuel using biomass derived synthesis gas. Fig 2 shows a schematic of the FT process. Our current source of syn gas is pine chips, however, any biomass feed stock can be utilized; from municipal waste, demolition waste, animal waste, etc. The problem lies with the resulting composition of syn gas produced biomass. A 2:1 H₂: CO syngas composition in required for optimal performance. Biomass derived syn gas has a typical H₂: CO ratio of 1-1.3:1.

Fig 1: GC results of product using 2:1 H₂:CO enriched biomass derived hydrocarbon fuel. Note the tight hydrocarbon distribution.



Fig 2. A schematic of the overall Fischer Tropsch process

Fig 3 shows our fuel product in stages of increasing H_2 concentration to the biomass derived syn gas up to the 2:1 optimum ratio. One can clearly see the optimized product at the far right has no phase separation and was used to produce the GC results of fig. 1. Conclusion; the H₂:CO ratio for biomass derived syn gas must be brought up to the optimal 2:1 ratio for best possible product results. What is need is a catalytic system where upon gasification of the biomass, a 2:1 H₂: CO syn gas is directly produce so that enrichment with hydrogen is no longer required.



Fig 3. Fuel samples 1-7 showing the change in phase separation as the optimal 2:1 H_2 :CO ratio is reached. Sample #7 is the resulting optimized fuel as seen in the GC results of Fig 1.

In addition, our technology was showcased at the 2010 Global Venture Challenge held at Oak Ridge National lab where the technology placed second in the US. Our team picture is shown below.



Project Description: *Statement of purpose:* The purpose of this effort is to eliminate a current **bottleneck** in the development of non-feedstock biomass gasification systems to produce an optimal H₂: CO ratio of 2:1 for Co-based FTS. Typical biomass syngas analysis is H₂ 32.7%, CO 42.5%, CO₂ 19.6% and CH₄ 5.2% (mol % dry-basis) as a result, the H₂: CO ratio is approximately 1:1 [1]. **Hypothesis:** By exploiting this mixture, one can increase the H₂: CO ratio via the development and optimization of a novel tri-reforming catalyst process. This is a synergetic combination of CO₂ reforming, steam reforming, and partial oxidation of methane in a single gasification reactor for effective production of industrially useful synthesis gas (syngas). **The CO₂, H₂O, CH₄, and O₂ in the exit gas need not be pre-separated because they will be used as co-reactants for tri-reforming.** The novel tri- reforming, as proposed, has the potential

to be performed within the biomass gasification stage as the temperatures, conditions, and constituents are optimal.

Program Objectives: (1) Synthesize and develop a cost effective joint tri-reforming catalytic system capable of producing non-feedstock syngas with desired H_2 : CO ratios of 2:1 complimentary to ongoing FTS at the University of South Florida. (2) Evaluate catalyst reducibility, activity and attrition. (3) Plant design including mass and energy balance using actual non-feedstock biomass generated syngas and (4) Evaluate the environmental burden/advantages and identify technological innovation opportunities.

Background and Significance: CO_2 and CH_4 conversion and utilization are an important component in chemical research on sustainable development; not only due to greenhouse issues but because CO_2 and CH_4 also represent an important source of carbon for fuels and chemical feedstock in the future [1–3]. The prevailing thinking for CO_2 conversion and utilization begins with the use of pure CO_2 , which can be obtained by separation. Even the recovery of CO_2 from concentrated sources requires substantial energy input [4,5]. According to US DOE, current CO_2 separation processes alone require significant amount of energy which reduces a power plant's net electricity output by as much as 20% [4,6]. Proposed in a novel method where CO_2 generated via biomass gasification can be utilized in one step by novel tri-reforming. However, there are two serious problems: deactivation of the catalyst by carbon formation and consumption of high energy due to endothermic processes (Eq. 1).

$$CH_4 + CO_2 \rightarrow 2CO + 2H_2$$
 $\Delta H^{o}_{298} = 247.3 \text{ kJ/mol}$ (1)

During commercial dry reforming, several side reactions of coke formation occur simultaneously (Eqs. (2) and (3))

$$CH_4 \rightarrow C + 2H_2$$
 $\Delta H^{o}_{298} = 74.9 \text{kJ=mol}$ (2)
 $2CO \rightarrow C + CO_2$ $\Delta H^{o}_{298} = -172.2 \text{ kJ=mol}$ (3)

Comparatively, the proposed joint tri-reforming process is far superior over current dryreforming both thermodynamically and in the elimination of carbon (coke) formation. Joint trireforming is a combination of endothermic CH₄ reforming (Eq. (1)), steam reforming (Eq. (4)) and exothermic oxidation of CH₄ (Eqs. (5) and (6)), which have a greater impact in regard to both industrial and environmental aspects [7-9].

$$CH_4 + H_2O \rightarrow CO + 3H_2$$
 $\Delta H^o_{298} = 206.3 \text{kJ/mol}$ (4)

$$CH_4 + \frac{1}{2}O_2 \rightarrow CO + 2H_2$$
 $\Delta H^o_{298} = -35.6 \text{ kJ/mol}$ (5)

$$CH_4 + 2O_2 \rightarrow CO_2 + 2H_2O$$
 $\Delta H^{o}_{298} = -880.3 \text{kJ/mol}$ (6)

RESEARCH APPROACH

The proposed investigation has been designed to enhance and expand funded research in this area, as well as exploiting PI's prior work [10-14] and collaborations established under the Binational Science Foundation with the Tel Aviv University and Weizmann Institute of Science and NREL. A support letter from Dr. Matthew M. Yung at NREL is attached.

Experimental: Various supported Ni catalysts will be prepared in our laboratory for trireforming in the proposed work. The supports include CeO₂, ZrO₂, MgO, SiO₂ and Al₂O₃, prepared using the soft chemistry technique reported by Rossigonol et al. [15] with Ce(NO₃)₃ - $6H_2O$ and Zr(OCH₂CH₂CH₃)₄ as the precursors. Ni will be dispersed on these supports by wet impregnation method using nickel nitrate Ni(NO₃)₂ -6H₂O under agitation for 1 h, followed by drying in an oven at 60 °C overnight. The dried solid will then be ground into power and calcined at 870 °C for 6 h in air. The Ni/MgO/CeZrOx catalyst will be prepared by the same procedures as described above except Mg(NO₃)₂-6H₂O (99% purity) was used as the precursor of MgO and both Ni(NO₃)₂-6H₂O and Mg(NO₃)₂-6H₂O will be dissolved into distilled H₂O to form an aqueous solution. The weight percentage of MgO in the Ni/MgO/CeZrO catalyst will be ca. 10 wt.%. A commercial Ni-Al₂O₃ catalyst (ICI Synetix 23-4,R15513) and mesoporous SiO₂ supported catalysts will also tested for comparison.

The supports were selected based on the following considerations: Supports with basic properties and/or high oxygen storage properties may promote the adsorption of CO₂ on catalysts and, consequently, enhance the CO₂ conversion. Based on a simplified mechanism of CO₂ conversion in the CO_2 reforming reaction [16-18], the reaction starts from the activation of methane followed by the surface reaction with surface CO₂ species or adsorbed oxygen atoms derived from CO_2 ($CO_2 + * = CO + O^*$, * denotes an active site). Compared with H₂O and O₂, CO₂ is more acidic. Basic supports may preferentially interact more strongly with CO₂ than H₂O and O₂. Once CO₂ is adsorbed on the catalyst surface, it may have more chance to react with CH₄ and form CO and H₂. Similarly, supports with more oxygen storage capacity may facilitate the dissociative adsorption of CO_2 into CO and adsorbed oxygen by $CO_2 + * = CO + O^*$, leading to the enhanced conversion of CO₂. MgO is a basic support which has been reported for CO₂ reforming [19–22], steam reforming [23,24], and methane partial oxidation [25]. However, no reports have been found on the comparison of CO₂ conversion in the presence of H₂O and O₂ as in the proposed tri-reforming process. The H₂/CO ratio in the products is related to the conversion of H₂O, CO₂, and O₂. The mixed oxide of Ce and Zr has been reported to have a larger oxygen storage capacity although the oxygen storage capacity of CeO_2 and ZrO_2 themselves is very little [24]. The application of this material has not yet been studied in trireforming, although similar support material has been tested by Roh et al. [23] in oxy-steam reforming. CeO_2 and ZrO_2 were chosen as supports for the purpose of comparison.

References:

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 Gunardson, Industrial Gases in Petrochemical Processing, Marcel Dekker, New York, 1998.
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DR. JOHN T. WOLAN

Education: Ph.D. University of Florida, June 1998; M.S. University of Florida, January, 1996; B.S. Chemistry, University of Central Florida, May 1981

Employment

2005	- Associate Professor of Chemical Engineering, USF, Tampa
2002-2005	- Assistant Professor Chem. Engg., Univ. of S. Florida, Tampa
1998-2001	-Assistant Professor of Chemical Engineering, Mississippi State University
1986-1998	- Lecturer and Research Associate, Department of Chemical Engineering, UF.

Awards and Honors

Honorable mention at the 2010 Global Venture Challenge Elected Director of Materials Science and Engineering Program, University of South Florida, 2009 Engineering Professor Research Award, University of South Florida, 2004 Engineering Professor of the Year, Mississippi State University, 2001 "Best Graduating Chemical Engineer", University of Florida, 1998

Selected Publications

A.H. Kababji, B. Joseph, J.T. Wolan; "Silica-Supported Cobalt Catalysts for Fischer–Tropsch Synthesis: Effects of Calcination Temperature and Support Surface Area on Cobalt Silicate Formation;" Catal. Lett (2009) 130: 72-78

Jonathan Mbah, Burton Krakow, Elias Stefanakos, and John T. Wolan, "A Study on H₂S Permeability of CsHSO₄ Membrane", International Journal of Hydrogen Energy; 34 (5) 2009, 2460-2466

Jonathan Mbah, Burton Krakow, Elias Stefanakos and John Wolan "Influence of High Energy Planetary Milling on the Ionic Conductivity of CsHSO₄", Electrochem. Solid-State Lett., Volume 12, Issue 7, pp. E12-E16 (2009)

Elias Stefanakos, Burton Krakow, Jonathan Mbah, and John T. Wolan: "Hydrogen Production from Hydrogen Sulfide in IGCC Power Plants", (DOE Information Bridge), http://nsdl.org/resource/2200/20080812094136518T

A.H. Kababji and J.T. Wolan, "Porous silicon Carbide and Gallium Nitride; "SiC Catalysis Today," book chapter 10, Wiley Press, editors Randall M. Feenstra and Colin Woods, ISBN 978-0-470-51752-9, copyright © 2008 Wiley Press & Sons Ltd.

46 scholarly articles in refereed journals
More than 50 technical presentations at national and international conferences
12 Invited technical seminars at major universities
18 Graduate students supervised
Consultant to industry and government

DR. JOHN N. KUHN

EDUCATION & PROFESSIONAL EXPERIENCE

2009-present Assistant Professor of Chemical and Biomedical Engineering, USF

2007–2009 Postdoctoral Fellow in Chemistry, University of California, Berkeley (joint appointment with Lawrence Berkeley National Laboratory in Divisions of Chemical Sciences and Materials Sciences)

POSTDOCTORAL SPONSOR

Prof. Gabor A. Somorjai; University of California, Berkeley and Lawrence Berkeley National Laboratory; (510)-642-4053; somorjai@berkeley.edu

2002-2007 Doctor of Philosophy in Chemical Engineering, Ohio State University Ph.D. ADVISOR

Prof. Umit S. Ozkan; Ohio State University; (614) 292-6623; ozkan.1@osu.edu

- 2001-2002 Research Assistant in Materials and Manufacturing Directorate at Air Force Research Laboratories, Wright-Patterson Air Force Base
- 1998-2002 Bachelor of Science in Chemical Engineering, University of Dayton

SELECTED PUBLICATIONS

1. **Kuhn, J.N.**, Huang, W., Tsung, C.-K., Zhang, Y., and Somorjai, G.A., "Structure sensitivity of carbonnitrogen ring opening: Impact of platinum particle size from below 1 to 5 nm upon pyrrole hydrogenation product selectivity over monodisperse platinum nanoparticles loaded onto mesoporous silica", *Journal of the American Chemical Society* 130 (2008) 14026-14027.

2. Kuhn, J.N., Zhao, Z., Felix, L.G., Slimane, R.B., Choi, C.W., and Ozkan, U.S., "Olivine catalysts for methane- and tar-steam reforming", *Applied Catalysis B: Environmental* 81 (2008) 14-26.

3. Kuhn, J.N., Tsung, C.-K., Huang, W., and Somorjai, G.A., "Effect of capping agent upon activity for ethylene hydrogenation and carbon monoxide oxidation over platinum nanoparticles supported on mesoporous silica", *Journal of Catalysis* 265 (2009) 209-215.

4. Kuhn, J.N., Lakshminarayanan, N., and Ozkan, U.S., "Effect of hydrogen sulfide on the catalytic activity of Ni-YSZ cermets", *Journal of Molecular Catalysis A: Chemical* 82 (2008) 9-21.

5. **Kuhn, J.N.**, Zhao, Z., Senefeld-Naber, A., Felix, L.G., Slimane, R.B., Choi, C.W., and Ozkan, U.S., "Ni-olivine catalysts prepared by thermal impregnation: structure, steam reforming activity, and stability", *Applied Catalysis A: General* 341 (2008) 43-49.

6. **Kuhn, J.N.**, and Ozkan, U.S., "Surface properties of Sr-and Co-doped LaFeO₃", *Journal of Catalysis* 253 (2008) 200-211.

7. Tsung, C.-K., **Kuhn, J.N.**, Huang, W., Aliaga, C., Hung, L.-I., Somorjai, G.A., and Yang, P. "Sub-10 nm platinum nanocrystals with size and shape control: Catalytic study for ethylene and pyrrole hydrogenation", *Journal of the American Chemical Society* 131 (2009) 5816-5822.

8. Zhang, Y., Grass, M.E., **Kuhn, J.N.**, Tao, F. Habas, S.E., Huang, W., Yang, P., and Somorjai, G.A., "Highly selective synthesis of catalytically active monodisperse rhodium nanocubes", *Journal of the American Chemical Society* 130 (2008) 5868-5869.

9. Zhao, Z., **Kuhn, J.N.**, Felix, L.G., Slimane, R.B., Choi, C.W., and Ozkan, U.S., "Thermally impregnated Ni-olivine catalysts for tar removal by steam reforming in biomass gasifiers", *Industrial & Engineering Chemistry Research* 47 (2008) 717-723.

Budget Justification:

Senior Personnel: One-month and half-month summer salaries over the 12 month period are budgeted for the PI (Wolan) and Co-PI (Kuhn) respectively. This time is in recognition of student direction, mentoring and guidance as well as hands on experimentation.

Student Support: One graduate and two undergraduate students are budgeted for 12 month appointments. Several other graduate students work with the research groups of the faculty PIs of this proposal, and their involvement is implicitly assumed, although not budgeted here.

Domestic Travel: Travel funds at \$1,000 are budgeted for the USF faculty and their student to attend Hinkley Center reviews.

Materials, Supplies and Instrument Time: Funds are requested for consumable chemicals and laboratory supplies, reagents, etc. (as needed) metrology instrument time (XPS, XRD, and SEM), shop fabrication costs, and such. Projects are charged an hourly rate for shared facilities at a subsidized rate for the College of Engineering.

Graduate Student Tuition: In-state tuition for the one graduate student is requested at the rate of \$321 per credit hour for a total of 24 credits per student. Out-of-state portion is about three times more, and a tuition waiver will be requested through the College of Engineering in case the students are not Florida residents.

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6	OTHER				0.0							\$0
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	UTHER			-								ې ل
		TOTALOTH	ER DIRECT COSTS	5								\$17,968
H. TOTAL	DIRECT CO	STS (A THR	OUGH G)									\$56,379
I. F&A (INI	DIRECT) CC	STS - SPEC	IFY RATE									
			Base			Rate	F&A Costs					
vr		C	\$48,411			0%	\$0					
y		.0	\$0			- 0%	φ0 Φ0					
yr2			φ0 ¢0			0%	\$ 0					
yr:			\$U			47%	\$0					
yr2	1		\$0			47%	\$0					
yıt	5		\$0			47%	\$0					
TOTAI F&A	COSTS											\$0
I TOTAL		D F&A COST	S (H+I)							<u> </u>	<u> </u>	\$56 270
K DEOIDU										<u> </u>		φυυ,379 Φο
KESIDUA			K SUPPORT OF C	UKKENT PROJE	:015)					┝──		\$0
L. AMOUN	I OF THIS I	REQUEST (J	I) OR (J MINUS K)						<u> </u>		\$56,379
M. COST SHARING: PROPOSED LEVEL - MUST be pre-approved by Dept Chair, Associate Dean for Research, and Dean \$0												