## TANTALUM-NIOBIUM INTERNATIONAL STUDY CENTER

## PRESIDENT'S LETTER

Friends,

The T.I.C. plans to hold its Forty-seventh General Assembly on Monday October 16th 2006, as part of a meeting from October 15th to 17th in Innsbruck, Austria including a plant tour to the metallurgical facility of Plansee. The members of the Executive Committee, the Technical Promotions Officer and the Secretary General are busy developing what promises to be an interesting, diverse and compelling programme including, naturally, the technical programmes. The quality and interest generated by these papers is largely governed by the commitment of its members so I would call upon you all now to consider contributing to the success of the meeting with your own submission of abstracts. These can be submitted to any of the Executive Committee, the Technical Promotions Officer or the Secretary General. This is a great opportunity for the association's many talented, experienced, specialist and knowledgeable members to further our industry's understanding and appreciation of what it can provide.

Spring is usually a time associated with the first green shoots of new growth after a winter chill. For the first time in a number of years, in parts of our industry, we are welcoming such signs of renewed vigour. Please take the opportunity to build upon a feeling of renewed optimism and begin the process of encouraging customers, suppliers, colleagues and any non-members to join the association and better still offer to provide a contribution to the technical programme.

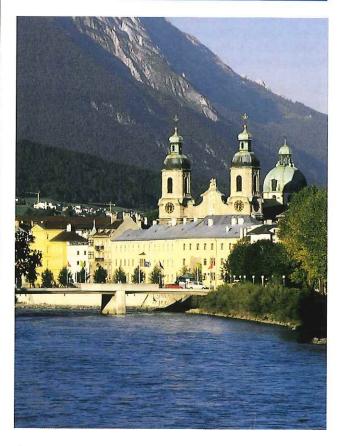
Members of the Executive Committee, the Technical Promotions Officer and the Secretary General will meet in Brussels in April to review and outline the programme for the forthcoming General Assembly. I would encourage you all to provide your suggestions on how we might improve our association and the service we provide to our members and would welcome your contributions to our deliberations.

William Millman T.I.C. President

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## **INNSBRUCK, OCTOBER 2006**



The Forty-seventh General Assembly of the Tantalum-Niobium International Study Center will be held in Innsbruck, Austria on Monday October 16th 2006: the full conference will run from October 15th to 17th. Following registration and a welcome reception on Sunday October 15th, the General Assembly of members will take place as the first event on the morning of October 16th. A programme of technical papers, with a break for lunch, will then be offered, and all delegates, guests and accompanying persons will be invited to a gala dinner in the evening. On Tuesday October 17th a field trip to Plansee SE will give delegates an opportunity to visit this metallurgical plant, by kind permission of the company.

The nominated delegates of member companies will be invited to attend: these letters of invitation will be sent about three months before the event. Others who are interested in attending should contact the T.I.C.

The technical sessions will be at the Hilton Hotel, where delegates will also stay – bookings are to be made through the T.I.C. Innsbruck is a charming Austrian town which welcomes visitors in both winter and summer. A sightseeing tour will be arranged for those people accompanying the delegates.

#### **DLA/DNSC**

The Annual Materials Plan of the U.S. Defense National Stockpile Center for the year to September 30th 2006, announced on October 4th 2005, includes

Columbium concentrates
Columbium metal ingots
Tantalum carbide powder
Tantalum metal ingots
Tantalum metal powder
Tantalum minerals
Tantalum oxide

560 000 lb Cb (Nb)
20 000 lb Ta
40 000 lb Ta
500 000 lb Ta
20 000 lb Ta

The Revised Plan announced on November 14th 2005 listed the same materials.

In October 2005, tantalum minerals containing 113 000 lb Ta were sold to DM Chemi-Met and Mitsui Mining and Smelting for approximately US\$4.4 million. Tantalum minerals containing 67 000 lb Ta were sold in November 2005 to Umicore, Raleigh, North Carolina, for approximately US\$2.7 million.

On December 12th 2005 the DNSC announced that it had suspended sales of several materials, including columbium (niobium) metal ingots and tantalum, until the Defense Authorization Bill for fiscal year 2006 had been passed by Congress and signed into law by the President. The DNSC had reached the limit of its authority under the existing law. There were no sales in December 2005.

On January 11th an immediate resumption of sales was announced, following the passage of the Bill through Congress and its signature as required. Sales for January 2006 under the Basic Ordering Agreement (BOA) procedure were reported on February 6th: 20 000 lb Ta in capacitor grade metal powder, 174 000 lb Ta contained in minerals, and 9900 lb columbium in vacuum grade metal ingot were sold to H.C. Starck Inc., Mitsui Mining and Smelting and Umicore for about US\$7.2 million. DNSC stated that these sales 'exhausted DNSC's inventory of capacitor grade tantalum powder'.

Tantalum minerals in seven lots containing 638 393 lb Ta stored at Warren, Ohio, were offered for sale in third week of February, but earlier in the month and again on February 23rd no material was offered.

Companies wishing to make offers for DNSC material should take careful note of the conditions for BOA pre-qualification and of the amendments such as no. 006 of October 20th 2005 which gives an extension to the period of offers (as they now close at 11.30a.m. on the Wednesday following a posting on the preceding Thursday), and details of the means of transfers for payment. A note to amendment 006 states that no materials contain more than 0.05% uranium and thorium, and all references to materials of Category 2 (with more than 0.05% uranium and thorium) and requirements for NRC licences were therefore deleted. Amendments 002-005 were also deleted. Amendment 007 should also be noted, as it refers to the wood packaging which may be used for tantalum and columbium materials and its failure to meet U.S. Department of Agriculture regulations.

www.tanb.org e-mail to info@tanb.org

## BIODIESEL PRODUCTION USING NIOBIUM-BASED CATALYSTS

by António T. Pereira, Kensley A. Oliveira and Robson S. Monteiro of Companhia Brasileira de Metalurgia e Mineração (CBMM), and Donato A.G. Aranda, Rafael T.P. Santos and Rafael R. João of Escola de Química da UFRJ, Centro de Tecnologia

This paper, Biodiesel Production Through the Esterification of Free Fatty Acids of Palm Oil by Niobium-based Acid Catalysts, was given by Dr Robson Monteiro (robson.monteiro@cbmm.com.br) during the technical sessions of the T.I.C. International Symposium Tantalum and Niobium World, October 2005.

#### 1. INTRODUCTION

The quest for alternative sources of energy other than fossil fuels has become nowadays public policy and it features in government-driven agenda associated to the need for sustainable growth of modern societies. Among several programmes worldwide, the feasibility of using bioethanol and biodiesel as substitutes for gasoline and diesel, respectively, has been successfully demonstrated in countries such as Brazil and Germany, where one can find entire car fleets running on biofuels only. The advantages are renewability, reduction of carbon dioxide emissions, and a significant decrease in particulate matter and sulfur compound releases from tailpipes [1].

Most of the currently produced biodiesel comes from the transesterification of vegetable oil triglycerides with methanol through the use of a homogeneous liquid-base catalyst. Typical liquid-base catalysts are NaOH, KOH and alkoxides such as CH<sub>3</sub>ONa. However, the base-catalyzed transesterification of triglycerides presents many shortcomings that lead to high production cost for biodiesel. The main issue of this process is the rigid specification for the triglycerides, which should not have acidity as free fatty acids (FFA) above 0.5 wt.%. Otherwise soap is formed, giving rise to the formation of gels that heighten significantly the viscosity. Another important fact is that glycerol is formed as a by-product of transesterification of vegetable oils, which causes the product separation cost to be greatly increased and the fuel grade quality of the biodiesel to be affected. In order to meet such demanding feedstock specifications, highly refined vegetable oils are required, whose price may account for 60 to 75% of the final cost of the biodieseľ [2-3].

Therefore, biodiesel is heavily subsidized. To be economically viable and to compete commercially with petroleum-based diesel fuel, the following are necessary: use of low cost feedstocks, continuous processes, few reaction and separation steps and the use of a heterogeneous solid and recyclable catalyst [3]. For low cost feedstocks with high acidic content as free fatty acids, esterification is becoming increasingly important. Besides lowering production cost, it also adds value and allows the use of biomass waste such as used frying oils, fatty acidic sewage or fatty acids from refined animal fats and vegetable oils [4]. To carry out the esterification either a liquid or solid acid catalyst can be used, but the use of a heterogeneous solid catalyst has its own benefits, such as ease of separation and recyclability.

This contribution aims to demonstrate that niobium-based solid acid is an industrially proven heterogeneous catalyst for the esterification of free fatty acids of refined palm oil. Among the vegetable oils, the content of free fatty acids of palm oil is one of the highest, averaging 4 to 8 wt.% as crude oil. For some

palm oil producers such as the Brazilian company AGROPALMA, FFA obtained after oil refining are treated as residues with low economic value. Upgrading the FFA through their transformation into biodiesel by esterification is an attractive and elegant way to aggregate value to this low-grade by-product.

## 2. BRAZILIAN BIODIESEL NATIONAL PROGRAMME

Brazil has the world's highest use of renewable sources of energy. It is around 41% of the energy pool, mainly due to the input of hydroelectric power and biomass with the world-renowned bioethanol fuel programme for automobiles. The major push for the use of biodiesel came recently from the Brazilian government with the introduction of Federal Law 11,097/2005. According to this legislation, the biodiesel programme will be implemented in three distinct phases, depicted in Figure 1. There are strong indications of an increase in volume to 20 vol.% (B20) in 2020.

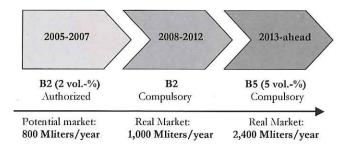


Figure 1: Three-phase implementation of biodiesel programme for Brazil, Federal Law 11,097/2005 (current diesel consumption 40 000MLiters/year)

In order to meet demand in coming years, Brazil is looking for several kinds of vegetable oils. Oils other than soya such as palm, castor, tame nut, turnip, sunflower and cotton are most preferred for lower cost, high productivity per land hectare and better adaptability to harsh weather such as highly humid or arid conditions. Another relevant aspect of the Brazilian Biodiesel National Programme is the policy of bringing development and generation of wealth to poverty-stricken rural areas in the northern and northeast parts of the country. Therefore, Brazil's biodiesel will not only promote environmental sustainability but also improve social conditions [5].

## 3. PALM OIL AND ITS CHARACTERISTICS

Palm oil crops grow well in the region within 10 degrees of latitude from the Equator. Although only a small part of the globe is available for production, with producer countries in the northern part of South America, Central Africa and the Malay Archipelago, palm oil has the second largest production of vegetable oil in the world. According to Figure 2, for world production of 100.9 millions of metric tons in 2003/2004, palm oil contributed 27.9%, slightly less than soybean oil, 31.6%. The reason is the remarkable oil productivity of palm tree crops of 7250 liters per hectare per year, that is seven to ten times higher than soybean crops. Malaysia and Indonesia are so far the world's largest producers (Table 1), but the highest growth potential is in Central Africa and South America.

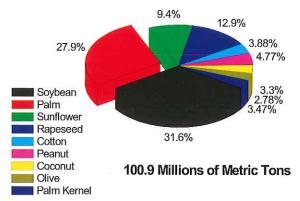


Figure 2: Vegetable oils: world production 2003/2004 Source: Foreign Agriculture Service, USDA

Country	1996/2000	2001/2005	2006/2010	2011/2015
Malaysia	9.0	11.0	12.7	14.0
Indonesia	5.4	8.3	11.4	14.8
Nigeria	0.7	0.8	0.9	1.0
Ivory Coast	0.3	0.4	0.4	0.5
Colombia	0.4	0.4	0.5	0.5
Brazil	0.0	0.1	0.2	0.3
Others	2.1	2.5	3.1	3.8
WORLD	17.9	23.5	29.2	35.0

Table 1: World's largest producers of palm oil and its derivatives, in millions of metric tonnes (Source: Oil World)

In terms of percentage by weight, palm oil typically consists of 43.7% palmitic acid, 39.9% oleic acid, 10.3% linoleic acid, 4.4% stearic acid and 1.0% myristic acid. As a crude oil it has one of the highest amounts of free fatty acids among the vegetable oils, which averages from 4 to 8 wt.% according to the harvest conditions. The free fatty acids obtained after refining the palm oil constitute a low cost raw material that can be transformed into biodiesel.

## 4. ESTERIFICATION OF FREE FATTY ACIDS

The esterification reaction of free fatty acids is an acidcatalyzed reaction, proceeding only through the use of acid catalysts. The reaction is carried out in the presence of an alcohol, added in excess – methanol or ethanol are the most common. The reaction products are methyl or ethyl esters of fatty acids, popularly known as biodiesel, and water – see reaction pathways below.

By using methanol:

R-COOH + CH<sub>3</sub>OH 
$$\rightleftharpoons$$
 R-COOCH<sub>3</sub> + H<sub>2</sub>O (fatty acid) (methanol) (methyl ester) (water)

By using ethanol:

R-COOH + CH<sub>3</sub>CH<sub>2</sub>OH 
$$\rightleftharpoons$$
 R-COOCH<sub>2</sub>CH<sub>3</sub> + H<sub>2</sub>O (fatty acid) (ethanol) (ethyl ester) (water)

where R denotes the hydrocarbon chain peculiar to the fatty nature of the raw material, containing 6 to 24 carbon atoms. H<sup>+</sup> is the proton representation of the catalyst acid sites.

The esterification reaction of fatty acids does not produce glycerol, which is commonly found as by-product in the base-catalyzed transesterification of vegetable oil triglycerides. Water is the only by-product, which makes the final operations of purifying biodiesel much simpler and more efficient.

## TANTALUM AND NIOBIUM WORLD



Welcome reception



From Symposium sponsor H.C. Starck: Dr and Mrs Heinz Heumüller and Dr Axel Hoppe (right

# Tantalum and Niobium World International Symposium 16-20 October 2005



General Assembly: T.I.C. Executive Committee



William Millman, AVX, T.I.C. President



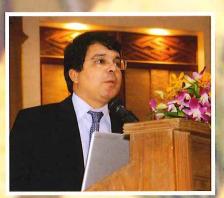
Symposium session Front left; T.I.C. Technical Promotions Officer Ulric Schwela







Michael Tamlin, Sons of Gwalia



Robson Monteiro, CBMM



Gaurav Aggarwal, Penn State University

With thanks to H.C. Starck (Thailand) for photographs

## **INTERNATIONAL SYMPOSIUM 2005**



President, sponsors, distinguished guests



Entertainment at the Gala Dinner: puppets and their puppeteers, from the Joe Louis Theatre, Bangkok



Speakers and participants



Suwichai Laohapaisan (left) and Joshua Loeffelholz, H.C. Starck (Thailand) who organized the plant tour and helped with the meeting



After dinner, everyone floated on the water of the pool a leaf 'boat' with candles and flowers



Splendid and colourful Thai decorations

Acid catalysts in commercial use are sulfuric acid, hydrofluoric acid and p-toluene-sulfonic acid. However, homogeneous liquid acid catalysts lead to contamination and generation of waste that requires neutralization, making product separation and purification more costly [6]. Thus, the use of a solid acid catalyst may bring several benefits such as ease of separation from the products and its own regeneration and reutilization.

Various solid acid catalysts have been commercially used for esterification of fatty acids. Good results were obtained for ion-exchange resins such as Amberlyst-15 and Nafion. However, at temperatures higher than 140°C those resins are not stable, which limits their applications. At high temperatures inorganic solids are more suitable. Zeolites, silica molecular sieves, heteropoly acids and sulfated metal oxides (ZrO<sub>2</sub>, SnO<sub>2</sub>, etc.) have been extensively used, but for all these catalysts the formation of water during the esterification hampers their ability of carrying out catalysis by slowing the reaction rates, sintering and leaching out of active acid sites [7]. Thus, an effective solid acid catalyst must be able to operate in watery conditions given that water is an inevitable reaction by-product.

## 5. NIOBIC ACID AS A SOLID ACID CATALYST

Niobic acid, the hydrated form of niobium pentoxide  $(Nb_2O_5.nH_2O)$ , is a well-known solid acid that shows a fairly high acid strength in spite of large amounts of water being contained (~20 wt.%). It is an amorphous material with BET surface area around 150 to 200 m²/g and average pore volume of 0.15 cm³/g. Its acid strength of  $H_0 \le -5.6$  is equivalent to the acid strength of  $H_2SO_4$ . Niobic acid shows high catalytic activity for various acid-catalyzed reactions in which water molecules participate or are liberated [8-9].

In this paper, niobic acid produced by Companhia Brasileira de Metalurgia e Mineração (CBMM) was used as a heterogeneous catalyst for the esterification of free fatty acids of refined palm oil. A mixture of FFA supplied by AGROPALMA (1.2 wt.% of lauric acid, 0.1 wt.% of myristic acid, 45.4 wt.% of palmitic acid, 40.5% oleic acid, and 11.1 wt.% of linoleic acid) and methanol (99%) from PROSINT at a 3:1 methanol/FFA molar ratio were used as reactants. Three different samples of niobic acid (Niobia HY-340) from CBMM in powdery, pellet and extrudate forms were used as solid acid catalysts. The amount of catalyst per reaction batch was 1 and 2 wt.% of FFA weight. The reaction was carried out at 130°C in a 500-ml Parr reactor stirred at 500 rpm. As reference samples for comparison were used extrudates of HY, ZSM-5 and Mordenite zeolites.

Figure 3 shows the kinetic curves for 1 and 2 wt.% of catalyst for samples of Niobia HY-340 powder and a HY zeolite (SAR = 60). In both plots the initial activity is high, but as the esterification reaction is constrained by the thermodynamic equilibrium the conversion levels off after 30 minutes of reaction. For 1 wt.% of catalyst (Figure 3a), no significant difference was observed among the samples in the time range investigated. However, the increase of catalyst amount for 2 wt.% (Figure 3b) showed clearly a difference of sample activities as the level conversion for the Niobia HY-340 powder jumped ahead of HY zeolite after 5 minutes of reaction, reaching a final conversion of about 70%, compared with 50% for the zeolite. The performance of Niobia HY-340 is yet more pronounced when comparison is made with ZSM-5 and Mordenite acid zeolites for a catalyst amount of 2 wt.% under the same reaction conditions, Figure 4.

Tantalum-Niobium International Study Center 40 rue Washington, 1050 Brussels, Belgium. Tel.: +32 2 649 51 58 • Fax: +32 2 649 64 47 e-mail: info@tanb.org The T.I.C. is an association internationale under Belgian law.

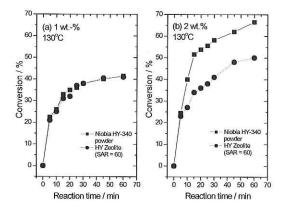


Figure 3: Kinetic curves for the esterification of free fatty acids of palm oil at 130°C for Niobia HY-340 and HY Zeolite: (a) 1 wt.% and (b) 2 wt.% of catalyst

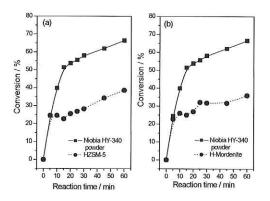


Figure 4: Kinetic curves for the esterification of free fatty acids of palm oil at 130°C for Niobia HY-340 in comparison with: (a) HZSM-5 and (b) H-Mordenite zeolites for 2 wt.% of catalyst

A possible interpretation of the activity of the Niobia HY-340 being better than that of the zeolites resides in its capacity to catalyze in the presence of water. It is well known that solid acid catalysts decrease their activity when water is in the reaction medium. Water absorbs on acid sites hampering their ability to carry out catalysis [7]. As mentioned above, the esterification of fatty acids with methanol produces water as a by-product. At a lower content of catalyst (Figure 3a), the influence of the water is hardly noticed, but when the amount of catalyst is increased (Figure 3b) the activity increases, which in turn increases the water content. Consequently, the HY zeolite does not keep up with Niobia HY-340 activity at a higher water concentration. Similar behavior was observed for ZSM-5 and modernite zeolites (Figure 4).

According to studies reported by Batamack et alii [10] on the acidity of niobic acid by interacting it with water, it was observed that the concentration of hydronium ion (H3O+) continuously increased with the increase of the concentration of adsorbed water molecules. Figure 5a shows that the number of hydronium ions per acid site ( $n_{\rm H3O}$ +/bas) increases monotonically with the number of water molecules per acid site (n<sub>H2O</sub>/bas) for niobic acid. In contrast with non-dealuminated and dealuminated HY zeolites, the variation of hydronium ion concentration as a function of the water concentration per acid sites was different. For dealuminated HY zeolite, nH<sub>3</sub>O<sup>+</sup>/bas gradually increases until the number of water molecules equals 1, becomes constant when  $1 < n_{H2O}/bas \le 2$  and rises significantly after  $n_{H2O}/bas > 2$ , but it is not capable of reaching the niobic acid behavior. For non-dealuminated HY zeolite, the number of  $H_3O^+$  ions is similar to niobic acid for  $n_{H2O}/bas \le 1$ , and it then remains constant, so more added water molecules per acid site do not play any further role in increasing the ionization of the zeolite.

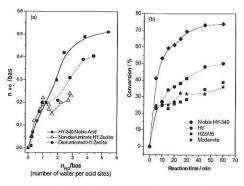


Figure 5: The role of hydronium ions (H<sub>3</sub>O<sup>+</sup>) in the performance of Niobia HY-340 in comparison to acid zeolites: (a) number of H<sub>3</sub>O<sup>+</sup> as a function of number of water molecules per acid site according to reference [10], and (b) kinetic curves for the esterification of free fatty acids of palm oil for Niobia HY-340 and HY, HZSM5 and Modernite acid zeolites

Putting in the same plot Niobia HY-340 and HY, HZSM5 and Mordenite zeolites for the esterification of free fatty acids of palm oil, Figure 5b, it is noticed, remarkably, that all catalysts present the same trend as that observed in Figure 5a. As the reaction proceeds in time, the level of conversion of Niobia HY-340 increases, and the water produced in the reaction hydrates the niobia, generating H<sub>3</sub>O<sup>+</sup> ions that keep the activity going despite more water being produced in the reaction. Conversely, all the zeolites fall short for not having the same ability as niobic acid to create hydronium ions. These results confirmed that under watery conditions niobic acid has a great advantage as a solid acid catalyst in comparison to zeolites.

## 6. NIOBIA HY-340 AS AN INDUSTRIAL CATALYST FOR BIODIESEL PRODUCTION

In order to apply Niobia HY-340 in an industrial process for biodiesel production, its powder form needed to be transformed into pellets or extrudate shape forms. Before loading it in a fixed-bed reactor on an industrial scale, tests were carried out to evaluate the best shape format. Table 2 shows the textural properties of pellet and extrudate in comparison with the powdery counterpart. Pellet format showed the lowest BET surface area, whereas no meaningful difference is observed for powder and extrudate. To form a pellet, powder is hard pressed in the presence of a binder, which might lead to the collapse and closing of pores. Pore volume presented similar results for all samples, but as expected pellet has the lowest value. The bulk density is quite comparable for all samples with minor differences. Due to the endogenous pressure in the esterification reaction because of methanol high vapor pressure at 130°C, the pellet or extrudate form of the Niobia HY-340 must withstand pressure up to 20 N/mm. Both prepared samples have enough crush strength to perform the reaction without being destroyed.

Materials/ Properties	Powder	Pellet	Extrudate
Dimensions			
-Diameter	-	6.2 mm	1.5-2.5 mm
-Height		3.6 mm	3-5 mm
BET Surface Area	160 ± 20	120 ± 10	200 ± 10
$(m^2/g)$			1
Pore Volume (cm <sup>3</sup> /g)	0.15	0.12	0.13
Bulk Density (g/cm <sup>3</sup> )	1.2	1.30	1.10
Crush Strength (N/mm)	-	30	25

Table 2: Physical properties of Niobia HY-340 for different shape formats

Figure 6 reports the results for the esterification of free fatty acids of palm oil for pellet and extrudate of Niobia HY-340 in comparison with its powder form. It is noticeable that the pellet format presented the best performance reaching the highest conversion, around 70% at reaction temperature of 130°C, after 60 minutes of reaction. Unexpectedly, the extrudate showed a very poor activity with results comparable to the behavior of zeolites. Forming Niobia HY-340 into pellets helped to improve the powder activity, which makes it a strong candidate for industrial use.

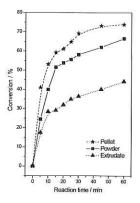


Figure 6: Kinetic curves for the esterification of free fatty acids of palm oil at 130°C for Niobia HY-340 in form of pellets and extrudates in comparison with powder

Niobia HY-340 in its pelletized form was then applied to an industrial fixed-bed reactor operated by AGROPALMA (Belém, PA) for the production of biodiesel from the esterification of free fatty acids of palm oil. AGROPALMA is Brazil's largest producer of palm oil and its derivatives. The industrial unit has the capacity to produce 24 million liters of biodiesel per year. A picture of the industrial plant is shown in Figure 7. The reaction temperature is carried out at higher temperature (~ 160°C) than laboratory tests in order to achieve conversion levels superior to 90% of methyl ester formation. The biodiesel plant started operations on May 2005 and the properties of the produced biodiesel are presented in Table 3.



Figure 7: Industrial plant for the production of biodiesel from the esterification of free fatty acids of palm oil operated by AGROPALMA (Belém, PA Brazil).

(Courtesy: AGROPALMA GROUP)

The quality of the biodiesel made from fatty acids of palm oil, registered as PalmDiesel®, meet the specification of Europe's EN14214 norm. For some properties such as ester purity, cetane number and stability to oxidation, the PalmDiesel® considerably exceeds the European specification making it a premium grade biodiesel fuel. One important characteristic is the absence of glycerol, even at levels of ppm, whereas the presence of glycerol is a very common problem for biodiesel

produced by the transesterification of vegetable oils. The use of a heterogeneous catalyst also minimized the presence of alkaline metals such Na, K, Ca and Mg that are typical contaminants in the base-catalyzed transesterification process.

#### 7. FINAL WORDS

Niobic acid, the hydrated form of niobium pentoxide (Nb<sub>2</sub>O<sub>5</sub>.nH<sub>2</sub>O), commercialized by CBMM as Niobia HY-340, is a proven industrial solid acid catalyst for the production of fuel-grade biodiesel from the esterification of free fatty acids of palm oil. Niobia HY-340 activity surpasses the performance of several commercial zeolites due to its unique ability to catalyze the reaction in the presence of water. This remarkable behavior may be related to the creation of hydronium ions (H<sub>3</sub>O<sup>+</sup>) as Bronsted acid sites on the surface of the catalyst as the water concentration increases in the reaction medium. Conversely, on acid zeolites the ionization effect in the presence of water is not as effective as for the niobic acid. For the first time in the world, a premium fuel-grade biodiesel is industrially produced through the esterification of free fatty acids of palm oil by using a heterogeneous acid catalyst.

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Properties	Unit	EN14214	PalmDiesel®
Density, 20°C	Kg/m <sup>3</sup>	0.86-0.90	0.87
Viscosity, 40°C	mm²/s	3.5-5.0	4-4.5
Water, max.	% vol.	0.05	0.00
Contamination	mg/kg °C	24	15
Flash point, min.	°C	100	177
Ester purity	%	96.5	99.5
Distillation, 90% vol.	°C	-	350
Carbon residue	%	0.30	0.01
Ashes, max.	%	0.020	0.001
Sulfur	%	0.001	0.001
Na + K, max.	mg/kg	5	2
Ca + Mg, max.	mg/kg	5	2
P, max.	mg/kg	10	1
Corrosion, 3 h, 50°C, max.		1	1
Cetane number		51	69
Aspect	( <del>-</del> )	-	Clean
Acidity, max.	mg KOH/g	0.50	0.50
Free glycerol, max.	%	0.02	0.00
Total glycerol, max.	%	0.25	0.00
Monoglycerides	%	0.8	0.01
Diglycerides		0.2	0.0
Triglycerides	%	0.2	0.0
Methanol or ethanol, max.	%	0.2	0.01
lodine	1000	120	50
Oxidation stability 110°C, min.	h	6	72

Table 3: PalmDiesel® properties and Europe's EN14214 biodiesel specification (Courtesy: AGROPALMA Group)

#### MEMBER COMPANY NEWS

**Cabot Supermetals** 

Operating profits for the December quarter were higher than for the preceding quarter, although lower than the December 2004 quarter. Mr Kennett Burnes commented that the company was 'encouraged by stronger demand for our tantalum products'. Following 'a very difficult year', a change in direction was announced in December for Cabot's sputtering targets, in spite of the recent establishment of its facility in Etna, Ohio. The company decided to 'stop its efforts to manufacture and sell finished tantalum sputtering targets' to end users, and focus efforts on the sale of tantalum plate to the target manufacturers, as it believed it could sell larger amounts of tantalum while incurring lower costs in this way. A labour dispute at the Boyertown plant which had begun in June 2005 was ended with agreements which the company thought satisfactory.

Cabot Supermetals / Sons of Gwalia

On February 8th the two companies announced settlement of their long-running negotiations which had ended in recourse to arbitration: both companies pronounced themselves 'pleased' with the result and its implied flexibility. Cabot would pay US\$27 million to terminate the existing supply agreement. Then the two companies entered into a new agreement for significantly reduced annual volume at higher prices.

#### Sons of Gwalia

On February 13th the company, which carries on business 'Subject to Deed of Company Arrangement', announced an operational restructuring which would maximize tantalum production at the 'lower cost' Wodgina Mine. The Greenbushes open cut mine would operate on reduced tonnages and the underground operation would be put on 'care and maintenance'. The company could 'respond on a timely basis to any increase in demand', it added. Further decisions on the future of the tantalum operations of Sons of Gwalia have yet to be announced: while some companies express interest in acquisition, others are reported to have sold their shareholding.

#### **Highland African Mining**

New address for correspondence:

P.O. Box 25343, East Rand, 1462, South Africa.

#### KEMET

On February 22nd KEMET announced that Mr Dave Reynolds (a member of the T.I.C. Executive Committee and a past President of the T.I.C.) was promoted to become Managing Director Suzhou China, the new production plant of Kemet.

Mitsui Mining & Smelting

Mr Hiroshi Yamamoto has been nominated as the T.I.C. delegate, as Mr Shunichiro Kachi has been appointed to other duties: h\_yamamoto@mitsui-kinzoku.co.jp.

Sogem/Traxys

T.I.C. member Sogem has been re-named Traxys, and its new

21 Avenue du Boulevard, bte 21, 1210 Brussels, Belgium. Tel. +32 2 227 23 21, fax +32 2 227 2376. Mr Frédéric Delforge succeeds Mr Bruno Deliens: frederic.delforge@traxys.com.

#### **EPCOS / KEMET**

The 'anticipated acquisition' by Kemet of the tantalum business of EPCOS (Bulletin 124) was the subject of an 'invitation to comment' by the British Office of Fair Trading issued on January 24th 2006, under the merger provisions of the Enterprise Act 2002 (comments by February 7th).