#### Mission report for 3rd mission to Guinea Bissau December 9<sup>th</sup> – 17<sup>th</sup> 2015 a Consultancy carried out for

ADPP, Guinea Bissau



Project tittle "Renewable Energy for Local Development Bissora Sector, Oio Region, Guinea Bissau"

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**Appendix A:** Daily notes and observations for NA's 3rd Mission to Guinea Bissau for ADPP. From 9/12-17/12 2015

Appendix B: Guide for titration to determine acid number in the vegetable oil (NA 11/12 2015)

**Appendix C:** ASG laboratory test report of Jatropha seeds, press cake and oil.

Appendix D: Raw and calculated data from testing oil pressing, incl. indications of sample ID's.

# 2 Introduction

### 2.1 Project background

The activities in this report relates to the Action:

"Renewable Energy for Local Development, Bissorã Sector, Oio Region, Guinea Bissau", a project of 48 months duration – November 2011 to October 2015 – agreed in a contract between Fundacion Pueblo para Pueblo Spain and the European Union in contract number: FED / 2011/ 232-430, and with ADPP Guinea Bissau as implementing local organisation.

### The Action has as its overall objective:

"Improve the living standards and local economic conditions in rural, low-income areas of Guinea-Bissau"

### The Action has as it specific objectives:

Introduce and establish renewable energy systems for local development in Bissorã Sector, Oio Region in Guinea-Bissau. O1: Increase in solar and sustainable bio-fuel energy supply in rural areas.. O2: Increased human capacities to encourage renewable energy uptake in rural areas, wherever cost-effective. O3: Reduce environmental impact by promoting sustainable sources of energy.

### The target groups are:

First level: 24 extension workers, 4 technical extension workers.

Second level: 14.274 community members of 24 communities in Bissorã Sector including 2.600 poor rural smallholder households.

### **2.2** The specific tasks for DAJOLKA/Niels Ansø on this 3<sup>rd</sup> mission:

The following specific tasks are listed in the ToR.

- a) Design any training material / manuals and maintenance systems and schedules necessary to secure the sustainability of the installation;
- b) Design and train in an oil quality control system, including recommendations for how to select and store seeds before extraction and eventually how to "adjust" for not so good oil quality;
- c) Together with the ADPP team draw a conclusion about the use of Jatropha oil
- d) Conduct a general refresher course for the team who participated in the first two trainings about how to take care of the generators and secure safety measures of the Gensets
- e) Produce a recommendation report which can be presented to EU about the Jatropha oil and its considerations on the future.
- f) Installation of the Frequency convertor and test its functionality
- g) Train the Jatropha machine operator how to regulate the frequency convertor to the right speed
- h) Make a general control for the generators
- i) Draft of a pocket manual for Jatropha oil experiences
- j) Train ADPP team on how to conduct a titration and preparation of the testing solution in order to detect the acid levels for the Jatropha oil

# 2.3 DAJOLKA's background

DAJOLKA is a Social Enterprise, established in Denmark in 1997, by Niels Ansø. (B.sc.Mec). Until 2005 DAJOLKA carried out non-profit activities parallel to Niels Ansø's job as project engineer at the Danish NGO, Nordic Folkecenter for Renewable Energy, where he has worked with Pure Plant Oil (PPO) technology since January 1999. Since 2005 DAJOLKA has turned its activities to be profit oriented and Niels Ansø working full time at DAJOLKA within Renewable Energy Technologies.

# **2.4 Training program for 3<sup>rd</sup> mission**

The main program for the 3<sup>rd</sup> mission

- 1. Check and ensure Jatropha seed quality
- 2. Make the Hybren oil expeller work together with the frequency converter.
- 3. Optimize settings of the oil expeller and frequency converter for best yield and oil quality
- 4. Check the Jatropha Oil quality
- 5. Follow up and solve any problems with the gensets, electric installations, grinding machines etc.
- 6. Check Jatropha seeds and Oil quality etc. after returning to Denmark

### 2.5 PPO fuel quality

The quality of the PPO fuel used in converted diesel engines is essential for the safe and long operation of the engine, and for keeping exhaust gas emissions low. This topic is explained in details in the 1<sup>st</sup> mission report.

One of the main interests for this mission is to conclude if it's possible to produce Crude filtered Jatropha oil which can meet or at least come close to the limits specified in PPO fuel norm DIN 51605, or if it is necessary with some degree of refining the crude oil.

We have seen a wide variety of test results of Jatropha oil quality, and we have tried to draw parallels from our experiences with rape seed oil to Jatropha oil. It seems that high acidity, which often is combined with high levels of phosphor, Ca and Mg, is the main challenge with Jatropha oil. From rape seed we know that there are clear relation between seed quality, including seed ripeness, and the quality of the expelled PPO. We have previously made test of Jatropha seeds of different ripeness, but further experiments has to be done to toughly understand the relations between seed quality and PPO quality.



4 different Jatropha seeds with different ripeness collected at 1 field in Mali.

We have discussed this issue with the project team in the previous 2 missions, and attention have been paid to collect only good and ripe seeds.

# **3** Preparations

From Denmark we had prepared the following before this mission

- Guide for titration to determine acid number in the vegetable oil
- Suggest new oil expeller to purchase
- Suggest soft starter for staring 2<sup>nd</sup> processing machines
- Visit the manufactory of the Hybren oil expeller, to discuss how to operate and optimize the H60 expeller.

# 4 Training Sessions

The following main training sessions were carried out.

# 4.1 Check and ensure Jatropha seed quality

There have been collected new Jatropha seeds from the fields in August/September 2015, and these had been sun dried every day for about 1 month. In addition there were some bags of older Jatropha seeds left from harvest 2013.

Since the seed humidity is essential for the performance of the oil expeller, we measured the humidity by evaporating the moisture by an improved drying system invented by Joe Grove.

For the purpose was used a metal can for keeping the Jatropha seeds, and a 1600W heat gun, placed in a distance 11cm from the can, to heat the seeds above 100°C, but still not heating the seeds too much, to avoid they would start burn or evaporate oil.

The system is shown on the photo to the right.

The drying system could work only in the day time when the genset is working. Approximately every 30minuttes the seeds were weighed on a balance.

In this way we measured the humidity of seeds from 2015 harvest to be 9,2% water, and 7,8% for the seeds from 2013 harvest.



9,2% seems high for oil pressing, and 7,8% seems acceptable. Later our practical tests will show the difference.



The curve shows the results of drying the Jatropha seeds from the harvest 2015. Initially the curve stabilized at 6,2%, but after increasing the drying temperature when we continued the next day, we got a new result showing 9,2% as result.

The issue about removing bad and broken seeds was repeated from the 2<sup>nd</sup> mission. Instead of sorting the seeds by hand picking and throwing them up in the wind, we invented a gravity separation system using an electrical fane from the office, as a prototype of a more efficient way.



Sorting broking and light seeds from good heavy seeds using a fane. The system worked really fast and fine, and was more healthy – no need for wearing respiratory protection.

# 4.2 Make the Hybren H60 oil expeller work together with the frequency converter.

The Hybren H60 oil expeller was purchased together with a frequency converter after our 2<sup>nd</sup> mission, where we concluded that the original oil expeller from Senegal was unable to work satisfactory with Jatropha Seeds. The Hybren H60 oil expeller was designed for pressing much smaller rape seed, with a nominal capacity of 60kg seeds /hour. Practical tests in Denmark had shown that it performed well also with Jatropha seeds. The Hybren expellers are normally giving a high oil yield of relatively clean crude oil, and generating less friction heat, which is positive with respect to oil quality.

The frequency converter was bought to use as tool to variate the speed of the oil expeller, to find optimal settings for pressing Jatropha seeds. At 50Hz the oil expeller runs the same speed as without the converter, and by lowering the frequency the speed drops proportional, and higher oil yield and lower temperature is expected.

Initially we had some problems to get the oil expeller running's continuously powered though the frequency converter, due to safety stops by the converter. These stops are to protect the electrical motor from overload. After changing the limits, as well as fixing loose connections



in the power line from the genset, we succeeded to get continuous operation between 30-48Hz.



The photo shows the Hybren H60 oil expeller opened for cleaning and preparing for start pressing. This has to be done after every stop, but is a very fast job because the press house consists of 2 half's, which are hinged near the gear box. The oil expeller has another 2 advantaged which are quite different from many other oil expeller designs, that the press cake thickness it not changed by opening and cleaning the expeller, and the oil expellers has crude oil pre filters integrated in the press casing. The frequency converter is the blue box on the wall behind.

# 4.3 Optimize settings of the oil expeller and frequency converter for best yield and oil quality

Several tests were performed with different seeds and different settings on the oil expeller and at different speeds. Besides variating the speed, we could variate the distance between the screw and the press cake nozzle, as well as we could variate the length of the pellet pipe. The raw measured data are shown in Appendix 4. We found that the optimal setting for press cake thickness was to adjust the initial distance between screw and nozzle to 0 (just contact, no pressure), giving press cake thickness of 0,5-0,8mm., and to choose the pellet pipe as long as possible, which was 22cm.

Please note the oil expeller should NEVER run dry with 0 distance between the screw and nozzle, because the friction metal against metal will destroy both parts.



The curve shows result for Throughput and Oil yield by variating the speed, pressing seeds from 2013.

The best oil yield seems to be 30% at around 37,5Hz corresponding 75% of nominal speed for the expeller. At the same time we can see that the throughput at this speed is around 60kg seeds / hours, which is the same as the press is designed for with rape seed at full speed. That means that this expeller can process around 85 kg Jatropha seeds at full speed.

This is a very useful experience which can be used next time ordering the same oil expeller for Jatropha oil production. Then it could be delivered with a higher exchange rate at the gear box, in order to reduce the nominal speed to optimal settings as shown from our test – and the oil expeller would run on these settings without frequency converter.



This curve shows the same data as previous curve, but also including the specific energy

consumption for producing 1 liter oil. Again the optimal setting is around 37,5 Hz where the energy consumption is around 0,22kWh/l or approximately 2,2% or the energy contents of the oil.

We noticed a big difference between pressing seeds from 2013 and 2015. The yield from the 2013 seeds was much higher. It is well known that new seeds are difficult to process – they need storing time for maturing – for rape seed around 1-2 months. For Jatropha we don't know how much time is needed. Another issue is high moisture content, which makes pressing difficult. Therefor we were not surprised that pressing 2013 seeds was much better. But later analysis showed that the 2015 seeds contained remarkable less oil than the 2013 seeds, which also explains the difference in yield. See more in chapter 4.6.

# 4.4 Check the Jatropha Oil quality

During the training sessions we analyzed the Jatropha oil to determine the amount of free fatty acids - by titration, which we trained at the 2<sup>nd</sup> missions. See Appendix B for guidelines how to do.



Unfortunately we found the FFA level still to be too high to be used for engine fuel. We measured 4,50 % FFA, which is 4,5 times the official limit, and more than twice as high as we can recommend to use, without damaging the injection system. Therefor we decided to make a test batch for reducing the FFA level by adding a mixture of water and NaOH – same ingredients as used to make soap. The amount of water and NaOH is calculated from the amount of oil you want to neutralize, and the actual FFA concentration. See spreadsheet below.

We made a 1,5liter test batch, but we didn't have time to get the result – the result is presented in chapter 4.6.

Bissora, GB, 15/12 2015												
Titration results calculator												
Use green cells for	input for t	he calculation	n.									
density SVO	0,92	kg/liter										
Volume NaOH for blind titration	0,0	ml										
NaOH titration solution concentration, $C_{NaOH}$	4,00	g/liter	0,40%									
sample ID	titration solution $V_T[ml]$	mg NaOH/g	FFA%	mg KOH/g								
Oil from old seeds	2,9	12,61	4,42	8,85								
Oil from old seeds	2,7	11,74	4,12	8,24								
Oil from old seeds	3,0	13,04	4,58	9,15								
Oil from old seeds	3,2	13,91 4,88		9,76								
		-	-	-								
average	3,0	12,83	4,50	9,00								
Official limit for ACID value for fuel				2,00								
Neutralisation of need the following amount p	per liter oil											
Oil sample to netralise, V <sub>o</sub>	I	1,5										
Needed amount of NaOH	g	19,2	(V <sub>T</sub> x C <sub>NaOH</sub> +	+ 1) x V <sub>0</sub>								
Needed amount of water to mix	Ι	0,06										
Needed amount of water to mix	ml	60	4%									

After neutralisation wash out soap and NaOH, and check for remainings of these in the oil.





Preparation of the water+NaOH solution(left) for neutralizing the 1.1/2 liter batch (right)

# 4.5 Follow up and solve any problems with the gensets, electric installations, grinding machines etc.

There was not reported any specific problems with the gensets. They seemed to be worked very good.

But during the test with oil press and frequency converter we found loose connections in the power line between the gensets and the workshop with processing machines. That explained why we experienced problems with low voltage on some phases, and might also partly explain the challenges we had at the 2<sup>nd</sup> mission to get 2 processing machines working at the same time.

Nevertheless, the problem starting 2 processing machines was solved by installing a soft starter device, which we identified after the  $2^{nd}$  mission, and it is a well-known problem that 3 phase electrical motors are a challenge to start in the small genset grid.

But anyway loose connections is an issue which has to be addressed also in the future, while it is a common mistake to assemble electrical wires by twisting them together and put some insolation tape, but its not strong and safe enough in systems with high current peaks from starting electrical motors.



The photo shows loose wire connections in the genset control box.

Another issue we found the electrical cables was that the power cable to the gensets we not a flexible type, so vibrations had made some mechanical wear to the wire insulations, so the conducting wires came in contact with the metal casing of the control box on the gensets – which is both a danger for humans, for fire and for the installation itself.

We noticed that still the operator of the processing machines has difficulties to adjust the load, so the machines are over loaded, which again load the electrical grid.



Here is example of the measured power consumption on the maize mill with the sieve with smallest holes for the finest flour, measured on our  $2^{nd}$  mission. The power consumption was up to twice as high as the much 5,5kW nominal power of the motor, which is the same as the full capacity of the genset (~11kW). The lesson learned was that the load on the motor is strongly depending on the way it is operated, and that the operator has to learn and care about not to force the maize mill too much, over loading the system.

### 4.6 Check Jatropha seeds and Oil quality etc. after returning to Denmark

Samples of Jatropha oil, press cakes and seeds were sent to the ASG laboratory in Germany after returning to Denmark. All test results are shown in Appendix C, and the corresponding sample ID's can be seen in Appendix D, from where is also appear when the samples was taken.

### Seeds:

Jatropha seeds from both 2013 and 2015 harvest were tested for humidity and oil content, with the following results.

2013: Humidity 7,7% (we measured 7,8%), Oil content 36,1%

2015: Humidity 7,3% (we measured 9,2%), Oil content 29,4%

The difference in oil content between 2013 and 2015 harvest is remarkable, and it can explain the difference in oil yield, besides that moisture content and stage of maturing.

### Jatropha Oil:

The result of testing the oil showed that the FFA level is still far too high for using it as fuel, ~7% for 2015 seeds and ~6% for 2013 seeds, and also the amount of phosphor(P) and metals (Ca+Mg) was too high. Our experience is that FFA, P and Ca+Mg follows the same tendency, so if the FFA level is brought down, normally P and Ca+Mg will be reduced as well.

The 1.1/2 liter sample which we prepared for neutralization in Guinea Bissau had become nearly solid and was further processed in Denmark. It needed to be heated the separate the soaps from the oil.



The neutralized sample was heated near to boiling, and the soaps separated from the oil and collected at the top layer, from where it could be removed.

The final result was that from the original 1.1/2 liter sample (1.380g), 560g (~40%) of soap was collected, and 820g (~60%) of clean oil was left.

The neutralized oil was also tested at ASG in Germany, and the result showed close to 0 FFA, P and Ca+Mg, so it the result with respect to fuel quality was very good.

The neutralization process is really simple, but the loss of 40% oil to soap might be a challenge. Since I  $\dot{m}$  not an expert in chemistry and soap production, it might be that this process can be improved a lot.



The soaps were removed and the remaining oil drained off, as much as possible.

### **Press cakes:**

The press cakes contained 13,5-15,1% oil, which is normal for cold pressing.

The energy content of press cakes were around 21 MJ/kg dry matter.

# 5 Follow-up

The following issues can be followed up.

- study and pay attention to the seed ripeness at harvest, and the seed condition during storing.
- Improve neutralization process of Jatropha oil, to be combined with soap production.
- Pay attention to electrical wire connection in the power lines from gensets for processing machines.

# 6 Conclusion

All technical tasks were completed within the planned time schedule. Problems were solved or solutions suggested to be implemented later. That includes

# 6.1 Check and ensure Jatropha seed quality

We found a practical way to determine the seed moisture content in the field, and designed an efficient system to sort out bad seeds, dust and other impurities, using an electrical fan.

# 6.2 Make the Hybren oil expeller work together with the frequency converter.

After solving some initial problems the Hybren H60 oil expeller and the frequency converter worked well together, and the system was very efficient for finding optimal settings for the Jatropha Oil expelling. The oil expeller performed very well and worked at low temperature around 70°C.

# 6.3 Optimize settings of the oil expeller and frequency converter for best yield and oil quality

By variating the speed, the distance between screw and nozzle and changing the length of the pellet pipe, we found the optimal settings for the Hybren H60 expeller with Jatropha seeds from 2013 harvest to be:

- Speed : 37,5 HZ or 75% of nominal speed
- Distance : initial 0, corresponding a press cake thickness of 0,5-0,8mm
- Length pellet pipe : 22cm

# 6.4 Check the Jatropha Oil quality

We tested the level of Free Fatty Acids(FFA) in the field, and found that it was still 2-4,5 times too high to be used as fuel. We conclude that this issue is too challenging, also based on experiences with other projects, so we recommend to neutralize the crude Jatropha Oil, before using it as fuel. The neutralization process can be combined with soap production.

# 6.5 Follow up and solve any problems with the gensets, electric installations, grinding machines etc.

The gensets are still performing very well.

We identified some problems with loose connections in the power lines from gensets to processing machines.

We saw that the soft starter works good to start a 2<sup>nd</sup> processing machine without stalling the electrical grid.

We saw that the operator of the processing machines still have problems to control the load on the machines.

### 6.6 Check Jatropha seeds and Oil quality etc. after returning to Denmark

Samples of Jatropha Oil, seeds and press cakes were sent for analysis in Germany.

Main finding regarding the seed quality was that the seeds from 2015 contained 29,4% oil which is considerable less than the seeds from 2013, which contained 36,1% oil. It also explains the big difference in oil yield between to 2 kinds of seeds.

The neutralized oil sample was further processed in Denmark and analyzed in Germany. The oil quality was really good as fuel with FFA, phosphor and metal(Ca+Mg) content near to 0. But around 40% of the oil was lost as soap, and only 60% of the oil ended as engine fuel quality. This process can probably be improved, but it is beyond our capability and experience at this moment.

The press cake contains around 14-15% oil, which is normal. The press cake will be a valuable source for biogas production, and then an even better fertilizer. The biogas can be used in dual fuel engines.

Again the training workshop had been planned very well by ADPP, and it is a pleasure to work with them.

Dronninglund, Denmark,

April 2016

malfall

Niels Ansø

### Wednesday 9/12:

Depart from home 19:00 to take train Aalborg – Copenhagen, then take plane Copenhagen-Lisbon, Lisbon – Casa Blanca, Casa Blanca - Bissau

### Friday 11/12:

Arrived Bissau 05:20. Went to ADPP office for small rest waiting for the car to take me to Bissora.

Had short discussion with Rafael and Mette about Jatropha oil quality and production price, food<>fuel discussion etc. Rafael mentioned a production price of Jatropha oil of over 2 \$ / liter, which is considerable higher than fossil diesel, which is about 1,XX

Arrived Bissora at 15:00. Joined Joe, Tembo and project leaders, who was in meeting at ADPP office. Joe explained that the calculation of the production price of Jatropha oil is based on purchasing seeds at 155 CFA/kg (xxx  $\in$ ct/kg), estimated total oil yield of 23%, plus transport and pressing, but setting the press cake value to zero.

In my opinion the press cakes represents a considerable value both as energy and fertilizer, so its unfear for the energy- and economical balance not to value the press cake. One way to utilize the press cake is in biogas system, where the rest oil content and organic matter can be digested giving energy as biogas. The biogas could easily be used as dual fuel in diesel generator, but best in application with stabile and high load.

### Saturday 12/12:

Went to site in Waitine, to start oil pressing.

Initially some problems with the power supply had to be solved. Found loose connection at one phase at the power supply to in the pressing room, and possible loose connection at the connection to the generator. At the generator the neutral had been disconnected, so that explained why the light and the 220V sockets didn't work in the process building and shop.

Oil press was investigated before starting up. It appeared that it had been running without seeds after being cleaned last time, so the screw had been rotating up against the nozzle with direct contact metal to metal, leading to high temperature due to the friction – high enough to melt the metal, so the screw and nozzle was light welded together. After the safety pin between the screw and the drive shaft from gearbox had broken.

Thee safety pin was changed – there were 4 in spare coming with the oil expeller.

The damaged nozzle and screw has to be polished to see if they can work again, but it was decided to do that later, not to lose time. Instead the new spare screw and nozzle were installed.

The new frequency converter was installed.

Started pressing powering the oil press from the frequency converter. But the frequency converted didn't allow to operate long time before stopping with alarm for over current – typically about 25A – which we couldn't understand while the average current red in the FC display showed only around 8-10A. Also 1 time it stopped with Alarm for under voltage – which was coursed by started the rise mill in the other room.

There's naturally a reason why the frequency converter stops with alarm – to protect the equipment and to protect the frequency converter it self. But the stopped is quit disturbing, while the oil press has to be cleaned each time before re started. Luckily the that is very fast to do, and the nozzle can be removed and cleaned without changing the nozzle setting settings (gab).

The under voltage monitoring was disengaged at the frequency converter, to avoid this problem to appear.

It was a problem that no detailed description was found in the oil press manual about how initially to adjust the gap between the screw and the nozzle. Therefor we were guessing from experiences with other oil expellers. The distance is adjusted by turning the nozzle completely against the screw, and then turn it out an exactly number of turns. This day we tested with 1 turn, then  $1\frac{1}{2}$  turn, and finally 0,75 turn.

# Full Belly grinder.

Joe tested the Full Belly grinder, to see if it can be used to de-shell the Jatropha seeds, to save manual work. The test show that the gab has to be adjusted for maximum in order not to brake or crack the seeds. We could conclude that the deshelling works fine, but further test should be done investigating if the seeds are getting broken.

### **Testing seed humidity**

Joe was testing the seed humidity by evaporating moisture using a hot air gun, checking the weight and temperature regular every ½h. The conclusion was that 8% water was evaporated during 5 hours – after that there was no more evaporation detected.

The interesting question is now if that shows that the kernel has dried, or if it need even more time.

### Sunday 13/12

Today we started testing the oil expeller with much smaller gap between the screw and the nozzle. Initially <sup>1</sup>/<sub>4</sub> turn, which gave an oil yield of only 7 % - then reduce the gap to 1/8 turn, which increased the yield to about 12%. Further adjustments were done by increasing length of the pellet pipe, and reducing the speed by lowering the frequency fed to the motor from the frequency converter.

The Kamstrup energy meter was installed and used to monitor the power consumption from the oil press.

The best result showed an oil crude oil yield around 19 %, which still is far too low (it come close to 30%).

### Monday 14/12

Went to Waitine to continue work with the oil expeller, while Joe and Tempo stayed at the office for other work and meetings.

We found that there were 8-9 bags Jatropha seeds from 2013 harvest – the same harvest which we used to test the Chinese oil expeller in February/march 2014. We decided to test these older seed, to compare with new seed, and to compare with the previous results with the old seeds.

We started pressing the seeds with the exact same settings as the day before, and we measured yield of 17% with the same speed and settings where we measured 12,3% the day before with new seeds, so the older seeds obviously works better.

First we increased length of pellet pipe from 18 to 22cm, which increased yield to 24,1%, and then we reduced the gap further, so that the nozzle was just in light contact with the screw. With this setting the yield increased to 28%.

Then we switched back to new seeds, and measured 19,6% yield with exactly same settings.

At the end of the day we could conclude that the oil expeller performs best with the old seeds from 2013 and with the nozzle adjusted with light contact to the screw, resulting in a gap around 0,5-0,8mm between the nozzle and the screw when the oil press is working with seeds. It should be stressed, that the oil expeller never must run dry with that tight adjustment, because the screw and nozzle can heat up due to friction and now cooling from press cakes passing.

# Tuesday 15/12

Seed dryer for humidity measurement (invented by Joe), 1600Watt, step 2, disctance 11cm.

Cleaning Jatropha seeds with electrical fan.

Pressing Jatropha seeds with variable speed.

Testing oil for acidity (Titration)

### Wednesday 16/12

Continuing seed drying of old seeds, finding that the moisture content is 7,8%, which with respect to oil pressing is considerable lower than the 9,2% moisture in the new seeds. Regarding the new seeds they might be easier to press after some further maturing.

Making neutralization of 1<sup>1</sup>/<sub>2</sub> liter Jatropha oil from pressed from the old seeds the day before, using the titration results to calculate the amounts of caustic soda(NaOH) and water to use for that process.

Summarizing the last days work and conclusions with the Tembo and the participants, about oil press and settings, using the frequency converter, doing titration, and possible neutralization of Jatropha oil with high acidity.



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NA 11/12 2015

DAJOLKA

#### Guide for titration to determine acid number in the vegetable oil

Pure vegetable oil must comply with certain quality standards to be used as motor fuel, so the engine is not damaged, and exhaust emissions will keep you within the norms.

One of these quality parameters are vegetable oil's acid number. The acid number is a measure of the number of free fatty acids (FFA) which are in the oil, and the number is expressed in mg KOH / g of oil, i.e. the number of mg of KOH (Potassium Hydroxide) needed to neutralize the free fatty acids in the oil. Alternatively, the acid value is expressed by the number of mg of NaOH needed to neutralize the FFA's, and then convert the number to mg of KOH.

The limit on the use of vegetable oils for engine fuels is officially 2.0 mg KOH / g of oil, which corresponds to 1% FFA in the oil. If the acid value is too high, it can have an abrasive effect on the metal surfaces, e.g. at the piston(s) in the fuel injection pump and fuel injectors, which are manufactured with very fine tolerances, so if attacked by abrasive fuel, it will change the performance of the engine. High acid fuel can also have a negative effect on the engine lubrication oil-

Titration is a method to determine the level of FFA in the oil. The method involves dissolving a precisely measured volume of vegetable oil in isopropyl alcohol, and adding color indicator (phenol red). Titration solution is then added, consisting of distilled water with a precisely measured concentration of KOH or NaOH dissolved. Titration solution is added slowly, while the whole mixture is stirred until all of the solution changes color to pink, permanent for at least 30 seconds.

### Ingredients for titration:

 Titration solution consisting of 0.1 or 0.4% KOH or NaOH dissolved in distilled water. The best results are achieved with the lowest concentration.

Isopropyl alcohol, 99%

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- vegetable oil
- 1% phenol red powder dissolved in isopropyl alcohol
- Glass for mixing and blending alcohol, oil, phenol red and titration solution.
- 3 pieces 10ml pipettes with pumping bulb, one to each, respectively, alcohol, oil and titration solution.
- 3 glasses for storing smaller quantities, respectively, alcohol, oil and the titration solution, for filling of the pipettes. The excess alcohol and titration solution is discarded and should not be poured back into the bottles with alcohol and titration solution.

#### **Procedure:**



1) Fill approximately 10ml isopropyl alcohol in the mixing glass



2) add 4 drops of phenol red solution to the alcohol



3) add exactly 1ml of vegetable oil



4) Add titration solution slowly with constant stirring. Initially, the mixture is yellowish. Later the mixture briefly starts to switch color to pink, but only when the mixture remains pink for at least 30 seconds the FFA's in the oil have been neutralized by the KOH or NaOH. Note precisely how much ml titration solution there was added to neutralize.

Repeat the process at least 3 times to ensure against fluctuations in results due measurement uncertainty.

#### **Blank titration:**

Since some alcohols may contain a minor amount of acid which may interfere with the measurement results, it is advisable from time to time to check the alcohol used. This is done with a blank titration, which is carried out as described above, but without the oil. If it is only required a few drops of the titration solution to neutralize the acid in the alcohol, it is of no importance. However, if it requires, for example. 0,5ml for neutralizing, blank titration should be done prior to each titration of the oil, or compensate the measurement result hereinafter, for example, if the blank titration shows that 0,5ml titration solution is need to neutralize the acid in the alcohol, then subtract 0,5ml from the titration result with oil.

### **Calculation:**

For calculating the acid value from the measured volumes during the titration, the following values and formulas are used.

k: concentration titration solution [g / I]  $\rho$ : density of the vegetable oil(= 0,92) [kg / I] v: volume titration solution [ml] v<sub>b</sub>: volume titration solution for blind titration [ml]

AN (Acid Number ) AN<sub>NaOH</sub>: Acid value based on titration solution of NaOH [NaOH mg / g oil] AN<sub>KOH</sub>: Acid value based on KOH titration solution [mg KOH / g oil]  $k_{FFA}$ : concentration of FFA in the vegetable oil [%]

Formula calculation of acid number:

$$AN = \frac{(v - vb) * k}{\rho}$$

Formula to convert from  $AN_{NaOH}$  to  $k_{FFA}$ 

$$k \ FFA = \frac{AN \ NaOH}{2,85}$$

Formula to convert from  $k_{\text{FFA}}$  to  $AN_{\text{KOH}}$ 

$$AN KOH = k FFA * 2$$

We have made Excel calculation sheets for calculation of AN and  $k_{\text{FFA}}$  from the measured volumes.

Titration results calculator												
Use green cells for input for the calculation.												
density SVO	0,92	kg/liter										
Volume NaOH for blind titration	0,0	ml										
NaOH titration solution concentration	4,00	g/liter	0,40%									
sample ID	titration solution [ml]	mg NaOH/g	FFA%	mg KOH/g								
	0,0	-	-	-								
		-	-	-								
		-	-	-								
		-	-	-								
		_	-	-								
average	0,0	-	-	-								

Example of calculation using. DAJOLKA's Excel spreadsheet.

### Hint's to measure precise volumes with pipette and pump ball.

A) Use **3 different marked pipettes**, 1 for each liquid, in order not to contaminate the different liquids with each other.



- B) The **pipette pump ball** has 3 air release valves which are marked A, E and S. The pump ball is used in the following way.
  - a. Press A and press the ball, to release the air from inside the ball. Now there will be under pressure inside the ball, ready to suck liquid into the pipette.
  - b. place the open end of the pipette in the liquid, and press S until liquid has filled into the pipette as much as desired.
  - c. Take the pipette up from the liquid, and adjust the liquid level in the pipette by pressing E
  - d. Now the pipette is ready to release liquid in to the mixing glass. Keep the pipette over the mixing glass and press E to release liquid.
- C) To release a precisely volume from the pipette, never use the last liquid in the pipette, but always work between the number on the scale, .e.g. to release 1ml, release from 7,0 to 6,0 on the scale

Please observe that the liquid has a curved surface, and you should always read the bottom of the curve.

ASG Analytik-Service Gesellschaft mbH Trentiner Ring 30 • 86356 Neusäss • Germany

DAJOLKA Dalmosevej 2 9330 Dronninglund DENMARK

Your reference	: Niels
Your order no.	: 4079
Date of order	: 21.12.2015
Sample Receipt	: 23.12.2015
Sender	: Customer
Start of test period	: 28.12.2015
End of test period	: 07.01.2016
Report date	: 08.01.2016
Page	: 1 of 1

Sample	ASG-ID	Parameter	Method	Result	Unit
Sample   Jatropha seeds S0   Jatropha seeds S1   Jatropha oil 01   Jatropha oil 02   Jatropha press cake   P0   Jatropha press cake   P6   Jatropha oil   Jatropha oil   neutralized 03	0000//0_001	Oil content	DIN EN ISO 659	36,1	% (m/m)
Jatropha seeds SU	2308648_001	Moisture & volatile matter	DIN EN ISO 665	7,7	% (m/m)
	0000//0_000	Oil content	DIN EN ISO 659	29,4	% (m/m)
Jatropha seeds ST	2308648_002	Moisture & volatile matter	DIN EN ISO 665	7,3	% (m/m)
		Acid value	DIN EN 14104	Result   Unit     2   36,1   % (m/m)     5   7,7   % (m/m)     5   7,7   % (m/m)     6   29,4   % (m/m)     5   7,3   % (m/m)     6   71,6   mg/kg     12,13   mg/kg   12,13     9   12,13   mg/kg     6,5   % (m/m)   6,5     6,5   % (m/m)   6,4     9   15,2   % (m/m)     9   15,2   % (m/m)     9   13,5   % (m/m)     9	mg KOH/g
SampleJatropha seeds S0Jatropha seeds S1Jatropha oil 01Jatropha oil 02Jatropha press cake P0Jatropha press cake P6Jatropha oil neutralized 03	2308648_003	Phosphorous content	DIN EN 14107	121	mg/kg
		Metal Content II (Ca+Mg)	DIN EN 14538	71,6	mg/kg
		Acid value	DIN EN 14104	12,13	mg KOH/g
Sample Jatropha seeds S0 Jatropha seeds S1 Jatropha oil 01 Jatropha oil 02 Jatropha press cake P0 Jatropha press cake P6 Jatropha oil neutralized 03	2308648_004	Phosphorous content	DIN EN 14107	40,8	mg/kg
		Metal Content II (Ca+Mg)	DIN EN 14538	27,8	Unit % (m/m) % (m/m) % (m/m) mg KOH/g mg/kg mg/kg mg/kg mg/kg mg/kg % (m/m) % (m/m) J/g dm % (m/m) % (m/m) % (m/m) J/g dm % (m/m) J/g dm % (m/m) J/g dm % (m/m) dm % (m/m) g KOH/g mg/kg mg/kg mg/kg
		Water content (Humidity)	DIN 51718	Result   Unit     36,1   % (m/m)     7,7   % (m/m)     29,4   % (m/m)     29,4   % (m/m)     7,3   % (m/m)     13,99   mg KOH/g     113,99   mg KOH/g     121   mg/kg     121   mg/kg     12,13   mg KOH/g     40,8   mg/kg     27,8   mg/kg     6,5   % (m/m)     15,2   % (m/m)     13,5   % (m/m)     22650   J/g dm     5,4   % (m/m) dm     6,4   % (m/m)     13,5   % (m/m)     22385   J/g dm     20938   J/g dm     5,1   % (m/m) dm     0,199   mg KOH/g     0,199   mg KOH/g     0,199   mg KQ     3,0   mg/kg	
		Oil content	DIN EN ISO 659	15,2	% (m/m)
Jatropha press cake	2308648_005	Calorific value, upper	DIN 51900-1 mod.	22650	J/g dm
		Calorific value, lower	DIN 51900-2 mod.	21180	J/g dm
	ASG-IDParameter502308648_001Oil content512308648_002Oil content512308648_003Phosphorous content2308648_003Phosphorous conten2308648_004Phosphorous conten2308648_004Phosphorous conten2308648_004Phosphorous conten2308648_004Phosphorous conten2308648_005Oil content II (Ca+2308648_005Calorific value, upperCalorific value, lowerAsh content (B15 °C)Calorific value, lowerAsh content (B15 °C)Calorific value, lowerAsh content (B15 °C)Calorific value, lowerAsh content (B15 °C)2308648_007Acid valuePhosphorous contenCalorific value, upperCalorific value, lowerAsh content (B15 °C)Ash content (B15 °C)Ash content (B15 °C)2308648_007Acid valuePhosphorous contenMetal Content II (Ca+Sodium (Na)Saponification value	Ash content (815 °C)	DIN 51719	5,4	% (m/m) dm
		Water content (Humidity)	DIN 51718	6,4	% (m/m)
		Oil content	DIN EN ISO 659	13,5	% (m/m)
SampleJatropha seeds S0Jatropha seeds S1Jatropha oil 01Jatropha oil 02Jatropha press cake P0Jatropha press cake P6Jatropha oil neutralized 03	2308648_006	Calorific value, upper	DIN 51900-1 mod.	22385	J/g dm
		Calorific value, lower	DIN 51900-2 mod.	20938	J/g dm
		Ash content (815 °C)	DIN 51719	5,1	% (m/m) dm
		Acid value	DIN EN 14104	0,199	mg KOH/g
		Phosphorous content	DIN EN 14107	0,9	mg/kg
Jatropha oil	2308648_007	Metal Content II (Ca+Mg)		<1	mg/kg
		Sodium (Na)	UIN EN 14538	8,0	mg/kg
		Sanonification value	150 3657	191	ma KOH/a

Report No. : 2308648-2

dm = result related to dry matter

Jürgen Bernath (Technical manager)

This report is related only to the samples stated above and may not be reproduced except in full, without approval of the testing laboratory. Storage of the samples: 4 weeks from report date For further information, please refer to our terms and conditons at www.asg-analytik.de

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+49 (0) 821 450423-0 +49 (0) 821 486 2519 info@asg-analytik.de Testing Hybren H60 oil expeller with Jatropha Seeds, Bissora, Guinea Bissau

NA 3rd mission to GB, Dec. 2015

Jatropha seed picked in August/september 2015, sun dried for 1 month, Humidity meassured to 9,2% And jatropha seeds 2 years old from 2013

			screw/						crude oil		de oil						Pellet				
date	time	Dt	nozzle		oil			cake	1	thoughput	yi	eld	Tempe	erature	Frequency	power	energy	pipe	Samples ID		
			distance	start	end	mass	start	end	mass				oil	press bead					oil	cake	seeds
		s	turn	g	g	g	g	g	g	kg	l/h	%	°C	°C	Hz	kW	kWh/l	cm			
13-dec	16:00	25.7	1/4	10.4	43.8	33.4	17.7	484	466.2	70.0	, 5.1	6.7%	55	78	50	3.3	0.65	4			
13-dec	16:57	28	1/8	26.6	96.1	69.5	17.8	512	493.7	72.4	9.7	12.3%	64	94	50	3.6	0.37	8			
13-dec	17:15	28.6	1/8	26.6	74.7	48.1	17.8	416	398.5	56.2	6.6	10.8%	64	96	45	3.5	0.53	8			<b>S1</b>
13-dec	17:25	22,9	1/8	15,9	75,5	59,6	18,7	392	373,3	68,1	10,2	13,8%	64	96	45	3,5	0,34	8			new
13-dec	17:40	29	1/8	13,6	73,6	60	19	388	368,7	53,2	8,1	14,0%	64	96	40	3	0,37	8	01		seeds
13-dec	18:00	29,3	1/8	17,5	92,9	75,4	20,2	364	344	51,5	10,1	18,0%	64	96	35	3	0,30	8			2015
13-dec	19:00	32	1/8	18,1	95,7	77,6	21,2	336	314,7	44,1	9,5	19,8%	64	106	35	3,3	0,35	18		P0	
						0			0	0,0	0,0	0,0%		106	35	3,6		22			
14-dec	13:25	22,3	1/8	16,5	89,6	73,1	18,1	375	356,5	69,4	12,8	17,0%	55	90	50	4,5	0,35	18			S0
14-dec	13:45	20,6	1/8	17,4	120,7	103,3	18,8	345	325,9	75,0	19,6	24,1%	64	95	50	4,7	0,24	22			old
14-dec	14:05	19	0	20,2	130	109,8	19,1	308	289	75,6	22,6	27,5%	65	100	50	5	0,22	22	02	P3	seeds
14-dec	14:10	19,9	0	26,8	153	126,2	19,1	335	316,2	80,0	24,8	28,5%	65	100	50	5	0,20	22		P3	2013
14-dec	16:30	24,2	0	23	107,7	84,7	18,9	315	295,8	56,6	13,7	22,3%	65	85	40	5	0,37	22		P4	new
14-dec	17:00	19,5	0	22,5	96,6	74,1	19,4	322	303	69,6	14,9	19,6%	76	115	50	4,8	0,32	22			seeds
14-dec	17:30	29	6/4	27,1	81,3	54,2	19,3	361	342	49,2	7,3	13,7%			40	1,7	0,23	22			2015
14-dec	18:15	26	1/8	21,5	81,8	60,3	19,4	350	331	54,2	9,1	15,4%	69	98	33	3,6	0,40	22		P5	dried
15-dec	14:00	29,6	1/32	20,2	149,9	129,7	17	531	514	78,3	17,1	20,1%	58	90	45	4,2	0,24	22			
15-dec		25,2	1/32	22,7	146,1	123,4	17,5	467	449,2	81,8	19,2	21,6%	65	97	47,5	4,7	0,25	22			
15-dec		26,1	1/32	22,7	132,2	109,5	18	457	438,6	75,6	16,4	20,0%	65	99	46	4,4	0,27	22			
15-dec		24,7	1/32	22,7	129,7	107	18	420	401,8	74,2	17,0	21,0%	65	100	45	4,3	0,25	22			
15-dec		24,6	1/32	21,3	134	112,7	18,1	437	418,5	77,7	17,9	21,2%	65	100	43,5	4,2	0,23	22			
15-dec		27,6	1/32	18,7	138	119,3	17,2	426	408,4	68,8	16,9	22,6%	65	100	42	4	0,24	22			
15-dec		28,2	1/32	19,5	134,8	115,3	17,4	393	375,1	62,6	16,0	23,5%	65	102	40,5	4	0,25	22			
15-dec		29,0	1/32	22,2	158,3	136,1	17,4	423	405,7	67,3	18,4	25,1%	67	105	39,5	3,9	0,21	22			50
15-dec		26,2	1/32	22,9	157	134,1	17,3	328	310,4	61,1	20,0	30,2%	67	105	38,5	4,3	0,21	22			old
15-dec		27,0	1/32	22,7	168	145,3	19,6	339	319,2	61,9	21,1	31,3%	67	110	38	4,2	0,20	22			seeds
15-dec		26,8	1/32	23,5	153,7	130,2	17,8	324	305,8	58,6	19,0	29,9%	67	105	37,5	4	0,21	22	03	P6	2013
15-dec		29,1	1/32	24,1	167,3	143,2	17,8	350	332,2	58,8	19,3	30,1%	60	105	37	4,2	0,22	22			2015
15-dec		28,7	1/32	25,7	157,7	132	18,1	331	313,3	55,9	18,0	29,6%	50	105	36,5	4,2	0,23	22			
15-dec		29,2	1/32	23,1	166,4	143,3	18,2	355	336,7	59,2	19,2	29,9%	60	105	36	4,2	0,22	22			
15-dec		30,2	1/32	25,6	164,2	138,6	18,1	362	343,4	57,5	18,0	28,8%	67	105	35,5	4,3	0,24	22			
15-dec		31,2	1/32	24,3	171,5	147,2	18,3	391	372,9	60,0	18,5	28,3%	67	105	35	4,3	0,23	22			
15-dec		28,2	1/32	26,4	170,6	144,2	18	359	341,2	62,0	20,0	29,7%	67	110	38	4,3	0,21	22			
15-dec		29,0	1/32	21,5	165,9	144,4	18	370	351,9	61,6	19,5	29,1%	67	110	38	4,3	0,22	22			
15-dec	15:30	29,0	1/32	23,2	154,9	131,7	18,2	367	348,4	59,6	17,8	27,4%	67	110	38	4,3	0,24	22			