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Effect of *Senna obtusifolia* Organic Solvents Extracts on Soda Pulping of Sudanese Sugarcane Bagasse

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ABSTRACT

Matured *Senna obtusifolia* (kawal) was collected as whole plant from Blue Nile state (Southern Sudan). Leaves, stem, and roots were been separated, air dried and ground in a standard star mill. The selection of representative raw material was due to its highly contents of anthraquinones. Bagasse was collected from White Nile sugarcane factory (White Nile State). Bagasse was air-dried and screened from sand and dust by dry debiting and screened by standard sieve. Five organic solvents were chosen to use in this study namely ethanol, ethyl acetate, chloroform, petroleum ether 60 - 80 and dichloromethane. Their chosen according to their polarity discrepancies. Full extraction was performed in a Soxhlet extraction unit. Each crude extracts were evaporated to dryness on rotator evaporator. *Senna obtusifolia* leaves, stem and roots were extracted with the five organic solvents; ethanol always has the highest yield % extractives (15.0053), (8.1733) and (6.1586) respectively. Bagasse employing three different alkaline pulping processes namely soda, soda-AQ and soda –AQ crude extract in certain conditions. 160 ^{o}C and active alkali 10.9% resulted screened yield% (49.84) and kappa number (15.57). Addition of 0.1 AQ to the same pulping conditions gave kappa number (8.26) and screened yield (53.68%). Stem extractives of senna obtusifolia attained screened yield ranged between (37.57%) and (55.44%) with kappa number ranged between (15.58) and (64.27). Leave extractives have been attained screened yield ranged between (33.58%) and (57.22%) with kappa number ranged between (17.89) and (30.57). Root extracts were achieved the promising results. It gave screened yield ranged between (49.5%) and (61.36%) with kappa number ranged between (16.01) and (28.02). Organic solvents crude extracts which are used as AQ source has attained yield ranged (55.44 % – 61.36 %) and kappa number ranged (15.58–17.89).

1. Introduction

Natural products are chemical substances produced by living organisms and could be classified into four main classes, carbohydrates, lipids, proteins and nucleic acids [1]. Anthraquinones are natural pigments that are found in plants e.g. *aloe latex*, senna, rhubarb, also they are found in fungi, lichens and some insects [2]. Senna species which are highly content of anthraquinones are usually used as medicinal herbs [3]. They are gaining importance in recent years due to environmental pollution caused by synthetic dyes [2, 3].

As general most quinines are chemically stable, so it is difficult to recommend any completely general extraction but; many quinones can be taken up in non polar solvents such as chloroform, ether [4]. Also it could extract even by ethanol [5]. Several anthraquinones have earlier been isolated and identified from the seeds of *Senna obtusifolia* [6].

Cassia review reported that the phytochemistry of *S.obtusifolia* leaves content mainly emodin, however the roots contents O-methyl-chrysophanol, aloe-emodin, chrysophanol, physicon, 1-hyroxy 7methoxy 3 methylanthraquinone, 8 –O methylchrysophanol, 1-O methylchrysophanol 1, 2, 8 trihydroxy 6, 7 dimthoxyanthraquinone, emodin, iso- landicin, helminthospoin, obtusifolin and xanthorin [2].

80%of approximately 350 species of *Senna* (L.) is widely distributed In Sudan, *Senna* (L.) is widely distributed in central, western, eastern Sudan, on the Nubian Desert and along the river Nile course from Khartoum to Dongola and it occurs on all types of soils with best yield on clay soils [7].

Fermented leaves of *Senna obtusifolia* (named Kawal) are used as a substitute of meat or an appetizing agent by people of eastern of Chad and western Sudan [8].The use of anthraquinone as catalyst in chemical pulping was first reported in 1977 for its effectiveness in accelerating delignification, decrease carbohydrate degradation and preserving pulp yield by enhancing the efficiency of the Kraft Process and thus reducing the number of trees harvested [9, 10]. Added About 0.1 % on wood of anthraquinone and results in a 1-3 % increase in pulp yield [9]. Although lignin is necessary to trees, it must be removed by pulping and bleaching processes for papermaking [11, 12 and 13]. Scientists found that it was possible to remove lignin from wood at high temperatures using strongly alkaline solutions based on sodium hydroxide (soda process) to produce pulp [14]. [15] observed that the cooking in the absence of anthraquinone yielded a high amount of uncooked material and consequently a low pulp yield.

Anthraquinone (AQ) is working as a redox catalyst, transferring electrons from wood carbohydrates to intermediate structures in lignin degradation, which results in higher yields and lower kappa number .This improve the selectivity with respect to lignin removal without significant carbohydrates degradation [16].

The redox mechanism is attributed to the aldehyde end groups of carbohydrates can be oxidized to acid groups, which stabilize the carbohydrates from further degradation. At the same time, the AHQ reacts with lignin in wood chips, which accelerates the delignification [17].

Sudan is rich country with pulp and papermaking raw materials that include non-woody plants, agricultural residue, recycled papers, as well as hard wood species [18].

Bagasse is the sugarcane (*Saccharum officinarum L.*) residue after extraction. It is one of the most lignocellulosic (nonwood raw) materials that used to produce pulp for papermaking because of its higher yields and better mechanical properties. By addition of AQ to soda pulping bagasse it accelerates the delignification to produce higher yields, lower Kappa numbers physical strength properties similar to kraft pulp grades [19, 20].

The Sudanese bagasse had very good cellulose content (54.8%) and comparatively to wood low lignin content (17.6%), however the favorable cellulose-to-lignin ratio of 3.1 predicted high yields by the alkaline pulping methods at moderate alkali charge [19].

The present study, deal with extraction of natural anthraquinones and anthraquinone derivatives from different parts of Sudanese *S.obtusifolia* using different organic solvents and use it as AQ catalyst to cook Sudanese bagasse to produced pulp for paper making.

2. Materials and Methods

Materials:

Plants Material:

Sample Collection and preparation:

Bagasse which is by product of sugar manufacturing was collected from White Nile sugarcane factory (White Nile State - Sudan). Bagasse was air-dried and screened from sand and dust by dry depithing and screened by standard sieve.

Matured *S.obtusifolia* was collected as whole plant from Al-Angassna area, Blue Nile state (Southern Sudan). Different parts (leaves, stem, and roots) were been separate and air dried. Stem, leaves and roots were ground in a standard star mill with a standard sieve and tied till used.

Methods:

Chemical Solvent:

Five organic solvents were chosen to use in this study namely ethanol, (E), ethyl acetate (E.A), chloroform (CH_3CL), petroleum ether 60-80 (P.E) and dichloromethane (DCM).Their chosen according to their polarity discrepancies. Previous studied used it to extract the phytoconsistituents of the under investigation plant species. [4, 21, 22, 23, 24].

Other chemicals:

Potassium permanganate ($KMnO_4$), Potassium Iodide (KI), Sodium Thiosulphate ($Na_2S_2O_3.5H_2O$), Sodium Hydroxide pellets purified (NaOH),Potassium Hydroxide (KOH) and Starch soluble from BDH chemical Ltd

Poole England. Sulphuric acid (H_2SO_4 98% AR) and Ammonia solution from Romil Pure Chemistry. Anthraquinone from Prolabo.
The above mentioned chemicals were used in pulping process, determination of kappa number and identification of anthraquinones.

Preliminary phytochemical screening for anthraquinone:
Identification of the anthraquinones in the plants species under investigation was carried on by take 2 gm of milled sample and boiled with 0.5N KOH, 10 ml of chloroform was added; this solution was shaken and then filtered. 3 ml of 10% ammonia was added to the filtrate. Formation of red or pinkish colour in ammonia layer indicates presence of anthraquinones [25].

Extraction:
30 gm of each part of milled *Senna obtusifolia*, leaves; stem and root were extracted with the above five different organic. Full extraction was performed in a Soxhlet extraction unit. Each crude extracts were evaporated to dryness on rotator evaporator.

Pulping Method:
Laboratory soda, soda-AQ and soda –AQ crude extract of bagasse cooks were performed in 5-litre rotary electrically heated digester. Pulping liquors were freshly prepared just before the beginning of the different cooks. The pulping conditions have been shown in the following table:

Pulping Conditions:
Table (1) Control condition for pulping

Control condition	
Oven –dry Weight/gm	500
Active alkali as Na_2O %	10.9
Active alkali as, NaOH %	14.06
AQ %	0.1%
Liquor to Bagasse	5 : 1
Max. Temp. ^oC	160
Time to Max. Temp., min.	1h
Time at Max. Temp., min.	1h
AQ/Extract %	Total gms of it

Pulp Evaluation and Characterization:
After washing, pulps were defibrated in a turbo pulper for 5min. The pulp was then screened and dried, granulated and kept in polythene bags. Moisture content was then determined and the total screened yield and rejects were calculated gravimetrically for the resulted pulps.
Total pulp yield % = screened yield % + rejects %
Their kappa numbers were measured by TAPPI T236 om-99.

3. Results and Discussion
Identification:
In this study milled leaves, stems and roots of *S.obtusifolia* were investigated for anthraquinone. All of the parts under investigation gave positive results and this agreed with [26] and [2].
Extraction of crude extracts using different organic solvents:
The highest extract yield was found to be from the leaves followed by stems and lowest from the roots (Table 2). Ethanol gave the highest yield and the lowest was petroleum ether extracts. It could be easily noticed that extracts yield were in sequence with the polarity of the solvents this agreed with [27], except the leaves extracted by chloroform the yield% was higher than that extracted with ethyl acetate that may be due to the higher content of the organic components in *S.obtusifolia* leaves

 

Roots, stems and leaves of *S.obtusifolia*
Leaves of *S.obtusifolia* after dilution

Table (2) Crude extractives yield (%) of stems, leaves and roots with different organic solvents

Species	**Solvent**	**Extractives (%)**		
		Stems	**Leaves**	**Roots**
Senna obtusifolia				
	Ethanol	8.173	15.005	6.158
	Ethyl acetate	2.076	5.791	1.638
	Chloroform	1.891	8.173	1.220
	Dichloromethane	0.821	5.043	0.677
	Petroleum ether 60- 80	0.706	2.964	0.422

It is [2] reported that anthraquinones derivatives of *S. obtusifolia* obtained mainly from leaves, seeds and roots. In this study we investigated leaves, roots and stems. All of these parts extractives could be easily added to the cooking process.

Bagasse pulping with soda and soda AQ:

In the present work, bagasse was chosen due to it is discarded as agricultural waste or burned for energy supply in sugar and ethanol mills and both alternatives are pollutants. Bagasse employing two different alkaline pulping processes namely soda and soda-AQ with following conditions shown in table (2).

Table (3) Pulping properties of soda and soda anthraquinone

Conditions	Soda	Soda AQ	Soda	Soda AQ
Weight/gm	500	500	500	500
NaOH%	15.5	15.5	14.06	14.6
Na_2O%	12	12	10.9	10.9
L:W	5 : 1	5 : 1	5 : 1	5 : 1
Temp ^oC	175	175	160	160
Time	2h	2h	2h	2h
Screened yield%	55.54	47.18	49.84	53.68
Reject%	0.195	0.038	0.307	0.365
Total yield%	55.73	47.22	50.15	54.04
Kappa No.	16.73	12.14	15.57	8.26

With cooked Sudanese bagasse with soda at 155-165^oC [28] and active alkali charge of 10.9 % it attained 53.2% as screened yield and kappa number equal to 13.9. By addition 0.1% anthraquinone to the cooking liquor at the same conditions, it gave screened yield 55.5% and kappa number equal to 12.2. In this study bagasse was cooked with soda and soda AQ (chemical) under specific conditions as shown in table (3) depending on [28] results. At 175 ^oC soda and soda –AQ pulping of bagasse carried out as reference cooks with active alkali (12%) as Na_2O, screened yield (55.54%) was achieved with acceptable kappa number (16.73). By added 0.1 AQ and cooked in the same conditions screened yield would decreasing to 47.18% which is lower than the soda pulp by 8.36% however the kappa number was lowering by 4.59 (12.14) that could be attributed to peeling reaction as a result of overcooking.
The decreasing of cooking temperature from 175 ^oC to 160 ^oC and active alkali from 15.5% to 10.9% resulted in lower yield (49.84%) and kappa number (15.57).The best result was attained by addition 0.1 AQ to the same pulping conditions, which gave much higher degree of delignification (kappa number 8.26) and screened yield increased to (53.68%).

Soda pulping with the addition of the crude extracts:
The present study deals with utilized of crude extracts of stem, leaves and roots of as AQ catalyst source to cook bagasse with soda process. The cooking conditions were kept constant with active alkali level of 10.9% as Na_2O on oven dry raw material; 60 minutes heating up time was needed to reach the maximum temperature of 160^oC, and 60 min the cooking time at maximum temperature as shown on table (3) for all cooks.
The results obtained (fig.1) by using *S. obtusifolia* stem crude extractives as AQ source applying to cook bagasse with soda according to the reference cooks, have shown that, the highest screened yield (55.44%) was obtained by pulping with (0.7063%) of petroleum ether followed by (0.8216 %) dichloromethane (54.13%) with acceptable kappa number (19.73) and (19.25) respectively and both are higher in yield than that of soda AQ standard (53.68%). (8.1733%) ethanol was shown lowest screened yield (37.57%) and highest kappa number (64.27). Chloroform stem extractives (1.8916%) were showed the lowest kappa number (15.58) with acceptable screened yield (49.41%) nearly equal to soda standard (15.57) (49.84%) respectively. Increasing of rejects percent, followed by higher kappa number which obtained in ethanol and ethyl acetate extractives catalyst (15.58%), (6.933%) as reject percent and kappa number (64.27), (51.3) respectively. The above results were fitted with the polarity of the solvents used compare with the polarity of anthraquinone and anthraquinone derivatives. 6 AQ which are 1-hydroxy-5-methoxy-2-methyl anthraquinone and its glycoside, 5-methoxy-2-methyl anthraquinone, chrysophanol, emodin and rhein [3], so the most probably best result achieved by the two first AQ derivatives mentioned above.

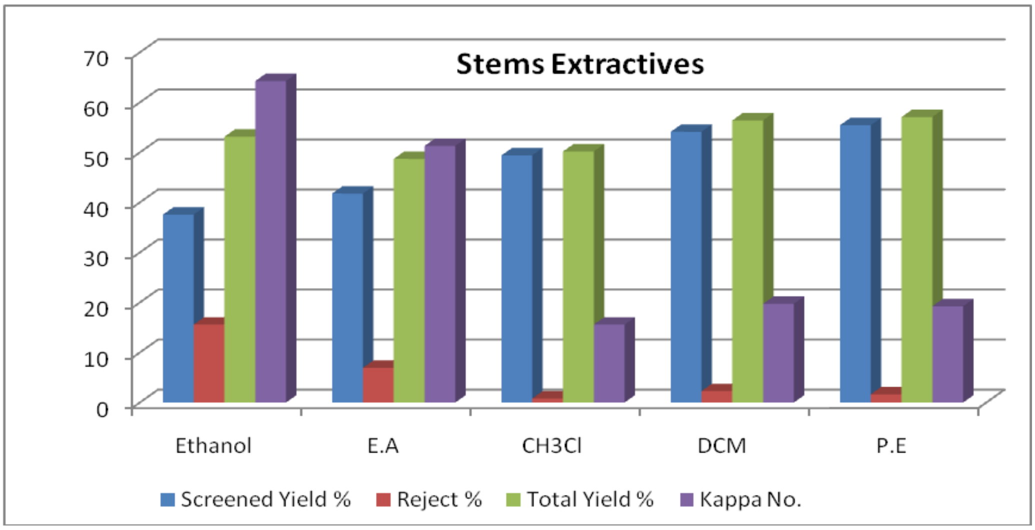

Figure 1: Pulping properties for the Senna obtusifolia stems extract with different organic solvents as anthraquinone catalyst

The results attained for leaves extractives as pulping catalyst manifested the highest screened yield (57.22%) was achieved by pulping with (5.0430%) of dichloromethane with significant kappa number (17.89) followed by (15.0053%) ethanol (51.09%) with acceptable kappa number (28.95). Despite of chloroform obtained highly extractives yield (8.1730%), it was acquired lowest screened yield (33.58%), and highest reject percent

(9.716%) and acceptable kappa number (29.04) nearly equal of ethanol extractives catalyst (fig. 2). The disordering is due to the leaves contain a lot of compounds (waxes, chlorophyll as examples) also according to [3] leaves of *S. obtusifolia* contain Rhein, Emodin, Physion, Chrysophanol ,Obtusin, Chrysoobtusin, and Chryso-obtusin-2-O-β-D [3].However [2] mainly Emodin, so the most probably best result achieved by Obtusin and Chrysoobtusin.

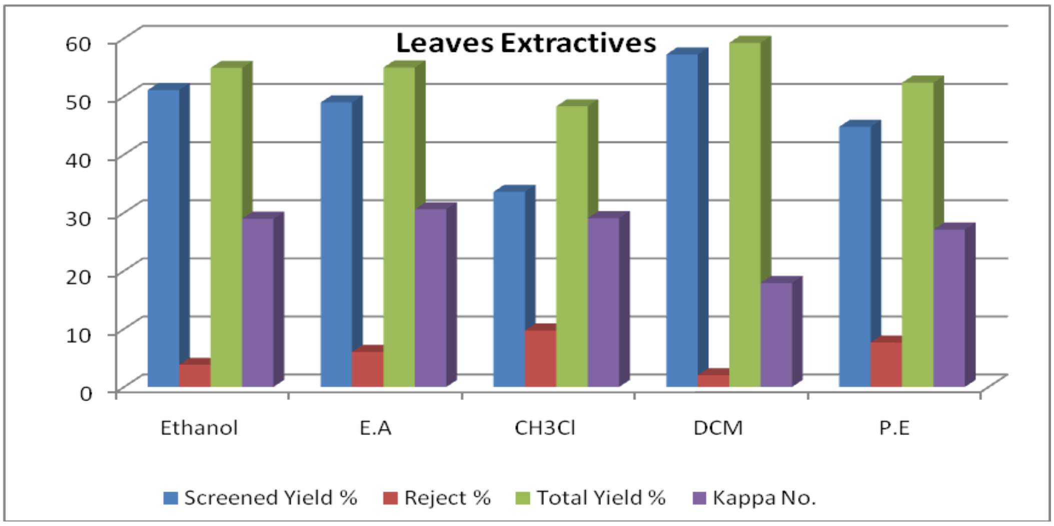

Figure 2: Pulping properties for the Senna obtusifolia leaves extract with different organic solvents as anthraquinone catalyst

The results achieved by using *S. obtusifolia* roots crude extractives as AQ source applying to cook bagasse with soda according to the reference cooks, have shown that, the highest screened yield (61.36%) was attained by pulping with (1.22%) of chloroform extractives with acceptable kappa number (28.02), followed by (6.1586%) ethanol (55.96%) with satisfactory kappa number (18.6). Ethyl acetate crude extract when it used as AQ catalyst it was attained screened yield nearly equal to ethanol (55.62%), with agreeable kappa number (16.01). Dichloromethane extractive (0.6773) have been shown screened yield (50.55%) with agreeable kappa number (25.115). Petroleum ether extractive was the lowest (0.4223) and it gave the lowest screened yield (49.5%) with passable kappa number (20.38) and lower of that of chloroform. In most cases the kappa numbers were in the acceptable ranges (under 30). [2] isolated 13 anthraquinone derivatives from the roots of *S. obtusifolia*. 8 of them have hydroxyl group in 1 and 8 positions and their structures are not complicated and could easily extract by polar solvents. So rest of them (5) could react easily with the aryl ether bond or alkyl ether bond of side chain of lignin and degraded it. This construes the promising results getting by the use of the roots extracts as AQ catalyst (fig. 3).

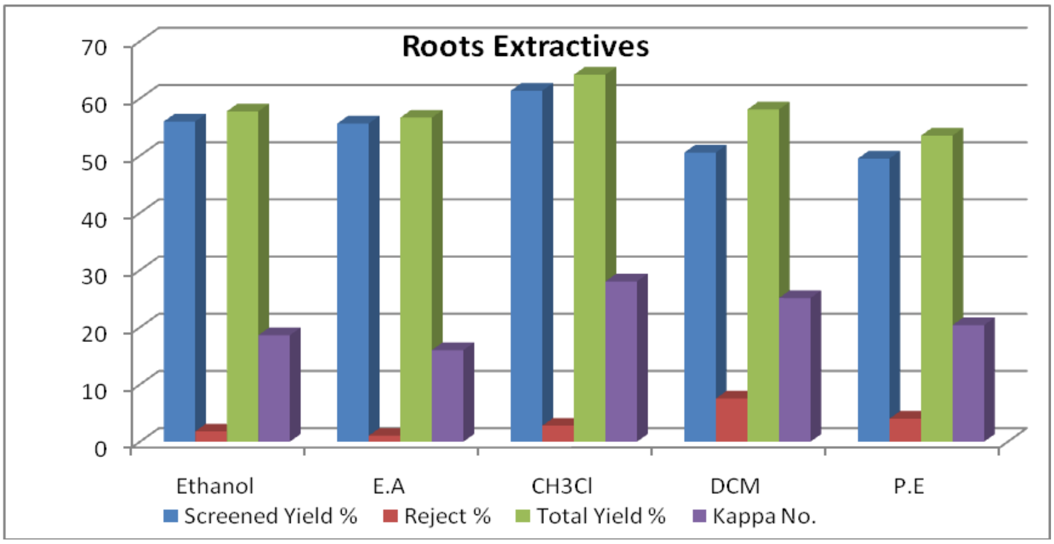

Figure 3: Pulping properties for the Senna obtusifolia roots extract with different organic solvents as anthraquinone catalyst

Ethanol, Ethyl Acetate, and Chloroform root extracts were found to be the most effective ones reflected in screened yield and with acceptable rejects (fig. 3). On the other hand stem and leaves extracts were the most effective in the case of Petroleum Ether and Dichloromethane respectively (fig. 1, 2).

As general, stem extractives of senna obtusifolia attained screened yield ranged between (37.57%) and (55.44%) with kappa number ranged between (15.58) and (64.27).

Leave extractives have been attained screened yield ranged between (33.58%) and (57.22%) with kappa number ranged between (17.89) and (30.57).

However it could be noticed that the addition of root extracts were achieved the promising results. It gave screened yield ranged between (49.5%) and (61.36%) with kappa number ranged between (16.01) and (28.02) and this with agreed [29] who reported that roots contained high percent of anthraquinones.

The above results were attractive from many points, [28] proved that pulping Sudanese bagasse using soda –AQ process obtained screened yield (57.9%) and kappa number (12.2) although [19]cooked it and gave a screened yield of 55.5% at the same kappa number of 12.2. In this work the reference soda –AQ screened yield was found to be (53.68%) with kappa number (8.26). Organic solvents crude extracts which are used as AQ source has attained yield ranged (55.44 % – 61.36 %) and kappa number ranged (15.58– 17.89).

Conclusions

- Leaves, stems and roots of *S.obtusifolia* are containing anthraquinones. Ethanol crude extractives of all parts always have the highest yield % while petroleum ether extractives were the lowest.
- Although the amount of the root extracts were the lowest, they appear to be most effective.
- The extractives of roots of *S.obtusifolia* using ethanol, ethyl acetate and chloroform have been shown promising results comparing.
- Chloroform root extract appears to be the most promising to replace AQ in soda pulping with additives giving the highest yield at acceptable kappa.

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