

## The Showy Milkweed, *Asclepias speciosa*: a Potential New Semi-Arid Land Crop for Energy and Chemicals

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### ABSTRACT

*Research on the chemical composition and domestication trials of Asclepias speciosa, showy milkweed, is presented. Biomass yields were approximately 4.3 tonne/ha (1.9 ton/acre) but increased plant density is expected to raise these yields considerably. The milkweeds were harvested with standard haying equipment and baled as with alfalfa hay. Storage tests in ambient conditions indicated that the non-polar extractables were stable whereas the polar extractables declined approximately 40% after about 2 months and stabilized at that level after five months. Storage in dry conditions resulted in only small losses. The non-polar extracts consist principally of  $\alpha$ - and  $\beta$ -amyrins and their acetate esters. The methanol extracts contain mostly sucrose and inositol. Milkweed extractives are compared to fossil fuels and cracking to liquid fuels is discussed along with various alternative uses for the extracted residue. The use of milkweed as a new crop depends on weed control, increased yields, product development and the development of commercial extraction and purification technology.*

*Key words:* milkweed, *Asclepias speciosa*, energy crop, economics.

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## 1. INTRODUCTION

Plants are renewable resources that can be genetically manipulated to produce different chemicals as needs change.<sup>1</sup> The worldwide sources of hydrocarbons are currently based primarily on mining of non-renewable geological deposits. The several shortages of hydrocarbons have focused considerable interest on biomass as a source of renewable hydrocarbons.<sup>2-20</sup>

Three general types of products can be obtained from plants: non-polar hydrocarbons (e.g. hexane and/or supercritical CO<sub>2</sub> extractables); polar (oxygenated) compounds (e.g. methanol extractables); and residue or marc. In addition, some species of plants produce fibers which may be useful for the manufacture of paper or cloth.

Non-polar extractables can be converted to liquid fuels by catalytic cracking<sup>20,21</sup> or may be used as high value lubricants or as coatings.<sup>22</sup> Polar oxygenated fractions (methanol extractables) may be cracked, fermented (to ethanol) or possibly used directly as chemical feedstocks. For example, polyphenolic constituents may be used for the manufacture of formaldehyde-linked adhesive resins or tackifiers of use to the plywood industry.<sup>19</sup> After the extraction of plant materials with hexane (or supercritical CO<sub>2</sub>) and methanol, the residue (marc or co-product) still retains its protein and much of its carbohydrate value which may make it useful as an animal feed. As part of a long-term study to discover new crops from which phytochemical products can be obtained, we have been investigating *Asclepias speciosa* Torr., the showy milkweed. The genus *Asclepias* is composed of approximately 140 species.<sup>23,24</sup> All cytologically known species are diploids ( $n = 11$ ) and interspecific hybridization is reported to be extremely rare in spite of widespread self-sterility.<sup>24</sup> The North American species are generally erect, herbaceous perennials although a few annuals are known.<sup>23</sup>

Woodson<sup>23</sup> states that no rhizomatous North American species are known except *A. syriaca* which 'may produce gemmiferous roots giving rise to clones of limited extent'. However, we have observed rhizomatous growth in *A. latifolia* and *A. speciosa*. *Asclepias tuberosa* is reported to live 25 years or more<sup>24</sup> and *A. subulata* is thought to live over a century.<sup>23</sup>

Due to the wide distribution nature of *A. speciosu* and its apparent ecological success, this taxon was selected for intensive research on its domestication potential as a source of phytochemical products.

## 2. MATERIALS AND METHODS

### 2.1. Field establishment

Plant Resources Institute (PRI) initially obtained the use of 4 ha of dryland and irrigated farmland. The farmland is in two 2 ha parcels located on the Black Island Farm near Syracuse, Utah.

Both parcels were disced, harrowed, fertilized, and treated with glyphosate (Roundup®) herbicide prior to planting.

The south field was listed into 91 cm rows for irrigation. The top of each bed was planted in two rows (25 cm apart) using a Planet Junior Vegetable Seeder in the spring of 1980. The level north field was divided into thirds and planted in the spring of 1980 with a disc-opener grain drill using an 18 cm row spacing. Plant densities were varied by planting in several passes, each at right angles. This resulted in seed applications of 7.8, 15.7, and 23.5 kg/ha.

### 2.2. Chemical extraction

Whole plant material was dried for 48 h at 70°C. The plant material was then ground in a Wiley® mill to pass a 2 mm screen.

A plug of glass wool was placed in a Whatman paper thimble (33 mm X 94 mm) and both were dried for 48 h at 100°C. The thimble and glass wool plug were then placed in a desiccator for 4 h to prevent rehydration before pre-weighing.

Disposable aluminum pans were used for evaporation of the solvents from each extraction but these were found to contain a volatile coating that would contribute a source of error. Therefore, the aluminum pans were baked at 100°C for 24 h, placed in a desiccator for 4 h and then pre-weighed.

Extracts were placed in pre-weighed aluminum pans and the solvents were evaporated in an externally vented oven. Hexane extracts were evaporated at 100°C for 48 h before weighing. Methanol extracts were evaporated at 100°C for 48 h and then placed in a desiccator for 4 h before weighing. The extraction thimble, glass wool and marc were then dried for 48 h at 100°C and placed in a desiccator for 4 h before final weighing. Although some volatiles are lost in the extract drying procedure, these would also likely be lost in a commercial harvesting and field drying process.<sup>2,3</sup>

### 3. AGRONOMIC STUDIES

Determining optimum cultural practices is a critical part of domesticating a 'wild' species, such as *A. speciosa*. The optimum date, rate and depth of planting must be determined. Other concerns include weed, insect and disease control.

#### 3.1. Weed control

Weed control is a major problem with milkweed, especially during stand establishment. During the seedling stage, milkweed appears to direct most of its energy into root development. This contributes to drought tolerance but the above-ground portion grows very slowly and is not competitive with fast-growing weeds. A selective, pre emergence herbicide is needed during the first year. In the absence of such a herbicide, glyphosate (Roundup) was used prior to emergence to control hard-to-kill perennials such as salt grass (*Distichlis stricta*) and common mallow (*Malva neglecta*). A wick applicator was used to apply Roundup to control the taller weeds during the season.

#### 3.2. Harvesting and yields

The first harvesting of the milkweed test fields was performed on 26 June, 1981 on the south field that was planted in rows. The plants were cut and crimped with a hay conditioner and swathed into windrows. The crimping operation crushes the stems, which aids drying of windrows.

Stems were dehydrated to a dry crack stage, and were baled within three days. The leaves dried considerably faster and became very brittle. Some losses due to leaf shatter occurred during baling. Hay that fell into the furrows could not be picked up by the baling machine resulting in additional crop loss.

The first harvest (26 June, 1981) yielded 0.93 tonne/ha (Table 1). Unfortunately, an estimated 20% of the crop was lost in the furrow and this pointed to the problem of row and furrow cultivation. The second harvest was much larger (Table 1) and probably was the result of an increased number of stems per plant.

TABLE 1  
Field Dried (18-20% moisture) Yields of Milkweed from Fields in Syracuse, Utah.  
(South field irrigated in 1981 with c. 90 cm of water, but farmed as dryland in 1982)

|                                    | Yields (tonne/ha) |                     |             |
|------------------------------------|-------------------|---------------------|-------------|
|                                    | 1st cutting       | 2nd cutting         | Total yield |
| <i>South field – in 91 cm rows</i> |                   |                     |             |
| 1980 established                   | –                 | –                   | –           |
| 1981 harvests                      | 28 June<br>0.93   | 1 September<br>1.59 | 2.52        |
| 1982 harvests                      | 6 July<br>3.30    | 3 September<br>1.05 | 4.35        |
| <i>North field – in 18 cm rows</i> |                   |                     |             |
| 1981 established                   | –                 | –                   | –           |
| 1982 harvest                       | –                 | 3 September<br>4.26 | 4.26        |

In 1982, the south field was harvested twice and yielded a total of 4.3 tonne/ha (Table 1). This increase over the 1981 harvest appears to be largely due to the increased density attained in 1982. The average number of stems per plant increased from  $2.07 \pm 0.17$  (SE) in 1981 to  $6.3 \pm 0.59$  (SE) in 1982. The north field, planted in narrow rows (18 cm), was only harvested once in 1982. That harvest was apparently too late, as an estimated 20% of the plants had dropped their lower leaves by that time. The single harvest on the thick-seeded north field was equivalent to the entire year's harvest (1982) on the south field (Table 1).

### 3.3. Crop storage

Although some crops, such as alfalfa, are often green chopped and hauled several miles to dehydrating/pelleting plants, we feel that there would be considerable economic savings if an energy/chemical crop could be stored and processed throughout the year. Two apparent methods for storage are fresh-cut as silage (70+% water) and dried as hay (below 15% water). Both of these procedures utilize existing

TABLE 2

Comparison of Hexane and Methanol Yields from Field Samples, Silage, and Bales of Milkweed. (Number in parentheses indicates the number of samples used to compute the mean and standard error of the mean)

|                                      | <i>Percent<br/>hexane yield (<math>\pm</math> SE)</i> | <i>Percent<br/>methanol yield (<math>\pm</math> SE)</i> |
|--------------------------------------|---|---|
| Whole plants, oven dried (25)        | 5.00 $\pm$ 0.092                                      | 13.96 $\pm$ 0.35  |
| 1 week after baling, field dried (5) | 4.19 $\pm$ 0.076                                      | 18.48 $\pm$ 0.680                                       |
| Silage, after 1 month (3)            | 5.97 $\pm$ 0.202                                      | 14.79 $\pm$ 1.100                                       |
| Silage, after 3 months (3)           | 5.66 $\pm$ 0.145                                      | 14.59 $\pm$ 0.600                                       |

farm equipment and thus would not require extensive new equipment design and manufacturing nor costly acquisitions by farmers. A comparison of yields from fresh cut (then oven dried at 70°C) plants, sun dried (3 days maximum at 37°C) and ensiled (1 and 3 months) samples is shown in Table 2. It appears that field drying reduced the yield of non-polar compounds (hexane extractables) and ensiling increased these yields. In contrast, the polar fraction (methanol extractables) is larger in the field (sun)-dried material and least in the oven-dried samples. Some of the cell components in the ensiled material may have broken down, making them easier to extract. In addition, some of the increased non-polar extractable yields may have come from the microorganisms. The loss of some of the non-polar compounds in the sun-dried samples may be due to photolysis and/or oxidation of unsaturated hydrocarbons to alcohols and other polar compounds. Obviously, the conversion of non-polar compounds would not account for all of the 4.52% increase in polar extractables seen in the field-dried samples. Additional research is needed on the change in composition of these extracts before any firm conclusions can be drawn.

Five bales were stored uncovered under ambient conditions. This storage test represented the worst possible case in that the bales were subjected to several feet of snow in the autumn and winter with a number of cycles of freezing and thawing. There appears to be gradual loss of the non-polar extractables (Table 3, Fig. 1) over time. However, the March sample (3.75  $\pm$  2 (0.116)) is not significantly ( $p = 0.05$ ) different from the first month's sample (August, 4.07  $\pm$  2 (0.072)). The change in methanol extractables is significantly different and shows

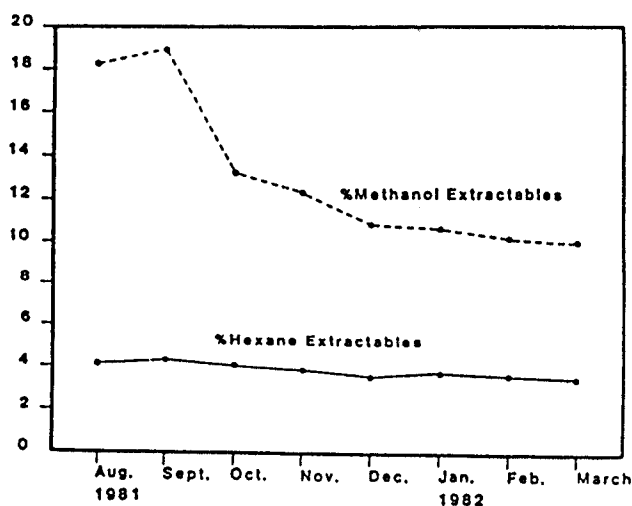


Fig. 1. Percent extractables versus time, July 1981 harvest (stacked uncovered in the shade).

TABLE 3  
Storage Tests of Five Milkweed Bales Harvested on 26 June, 1981  
(means  $\pm$  standard error of the mean)

|                                     | Percent<br>hexane<br>yield | Percent<br>methanol<br>yield | Total<br>yield    |
|-------------------------------------|----------------------------|------------------------------|-------------------|
| <i>Control</i>                      |                            |                              |                   |
| Whole plant, oven dried (25)        | 5.00 $\pm$ 0.092           | 13.96 $\pm$ 0.35             | 18.96 $\pm$ 0.37  |
| <i>Sun-dried, stored bales (5):</i> |                            |                              |                   |
| 1 month (Aug.)                      | 4.07 $\pm$ 0.072           | 18.33 $\pm$ 0.675            | 22.38 $\pm$ 0.689 |
| 2 months (Sept.)                    | 4.14 $\pm$ 0.308           | 18.90 $\pm$ 1.02             | 23.05 $\pm$ 1.87  |
| 3 months (Oct.)                     | 4.06 $\pm$ 0.168           | 13.25 $\pm$ 0.885            | 17.31 $\pm$ 0.87  |
| 4 months (Nov.)                     | 3.71 $\pm$ 0.093           | 12.32 $\pm$ 1.47             | 16.03 $\pm$ 1.43  |
| 5 months (Dec.)                     | 3.55 $\pm$ 0.047           | 10.75 $\pm$ 0.98             | 14.30 $\pm$ 1.00  |
| 6 months (Jan.)                     | 3.62 $\pm$ 0.070           | 10.64 $\pm$ 0.804            | 14.26 $\pm$ 0.793 |
| 7 months (Feb.)                     | 3.59 $\pm$ 0.100           | 10.08 $\pm$ 0.892            | 13.66 $\pm$ 0.898 |
| 8 months (Mar.)                     | 3.75 $\pm$ 0.116           | 9.61 $\pm$ 0.589             | 13.36 $\pm$ 0.598 |

Note: Bales were stored unprotected on the north side of a building (shaded). Beginning in January 1982, each of the five bales was sub-sampled three times instead of running triplicate analyses on one sample from each bale

a sharp decline after two months of storage and a gradual decline thereafter (Table 3, Fig. 1). This is probably due to the catabolism of the carbohydrates by microorganisms during the decomposition process.

Three additional storage conditions have been studied: (1) bales stacked in a barn; (2) bales stacked in the open, covered with clear plastic; and (3) bales stacked in the open, covered with black plastic.

The results from these three treatments (barn storage, clear plastic, black plastic) of the September 1981 harvest are somewhat ambiguous. The initial samples (October, November, December) from these treatments consisted of only one sample per bale per treatment, analyzed in triplicate. The bales proved to be non-homogeneous and the triplicate analyses method was abandoned in January in favor of using three sub-samples/bale for each of the four bales/treatment. Within a bale, heterogeneity apparently was caused by considerable saltgrass in the field at the time of the September harvest. Table 4 shows the results of storage for the three treatments. Heterogeneity of individual bales is most apparent in the methanol extract yield for the dry-stored November, December samples (16.43 vs. 10.34%) and the black plastic-covered November, December samples (15.15 vs. 9.26%). Omitting the November and December samples (1 bale, triplicate analyses) resulted in the graph shown in Fig. 2. The non-polar yields show no significant differences. The methanol extractable yields each appeared to decline in February. However, there were no significant differences between the yields except between the low value obtained in February (black plastic treatment) and the adjacent samples (January, and March).

In general, it appears that moisture and subsequent rotting are the major potential problems associated with storage of baled milkweeds. This is generally not a problem in semi-arid lands. If moisture is a problem, the bales could be covered with either clear or black plastic.

#### 4. CHEMICAL PRODUCT ANALYSES

##### 4.1. Analysis of the hexane extract of *A. speciosa*

Hexane extracts of the aerial parts of *A. speciosa* were obtained by Soxhlet extraction for 20 h (see Section 2). These extracts were dark green in color. Pigments were removed by decolorizing according to



TABLE 4  
Storage Tests of Milkweed Bales Subject to Three Treatments:  
(1) Dry Storage; (2) Clear Plastic; (3) Black Plastic

| Treatment                          | Percent<br>hexane<br>yield | Percent<br>methanol<br>yield | Total<br>yield |
|------------------------------------|----------------------------|------------------------------|----------------|
| <i>Dry storage</i>                 |                            |                              |                |
| after 1 month (Nov.)               | 4.71 ± 0.018               | 16.43 ± 0.102                | 21.14 ± 0.093  |
| after 2 months (Dec.)              | 4.65 ± 0.029               | 10.34 ± 0.080                | 14.99 ± 0.092  |
| after 1 month (Jan.) <sup>a</sup>  | 4.88 ± 0.158               | 13.50 ± 0.776                | 18.38 ± 0.655  |
| after 2 months (Feb.)              | 4.82 ± 0.207               | 12.13 ± 0.468                | 16.95 ± 0.472  |
| after 3 months (Mar.)              | 4.56 ± 0.112               | 14.27 ± 1.005                | 18.82 ± 0.948  |
| after 4½ months (Apr.)             | 5.56 ± 0.680               | 13.09 ± 0.660                | 18.66 ± 0.780  |
| <i>Covered with clear plastic</i>  |                            |                              |                |
| after 1 month (Nov.)               | 5.19 ± 0.052               | 11.13 ± 0.110                | 16.32 ± 0.065  |
| after 2 months (Dec.)              | 4.83 ± 0.046               | 11.55 ± 0.123                | 16.38 ± 0.150  |
| after 3 months (Jan.) <sup>a</sup> | 4.41 ± 0.118               | 13.09 ± 0.659                | 17.50 ± 0.627  |
| after 4 months (Feb.)              | 4.58 ± 0.153               | 11.79 ± 0.476                | 16.37 ± 0.415  |
| after 5 months (Mar.)              | 4.78 ± 0.135               | 14.62 ± 0.520                | 19.41 ± 0.560  |
| after 6½ months (Apr.)             | 4.97 ± 0.660               | 13.59 ± 0.810                | 18.56 ± 0.797  |
| <i>Covered with black plastic</i>  |                            |                              |                |
| after 1 month (Nov.)               | 4.66 ± 0.040               | 15.16 ± 0.143                | 19.82 ± 0.144  |
| after 2 months (Dec.)              | 4.23 ± 0.031               | 9.25 ± 0.012                 | 13.49 ± 0.043  |
| after 3 months (Jan.) <sup>a</sup> | 4.49 ± 0.149               | 15.28 ± 0.817                | 19.77 ± 0.836  |
| after 4 months (Feb.)              | 4.25 ± 0.215               | 11.64 ± 0.554                | 15.89 ± 0.531  |
| after 5 months (Mar.)              | 4.43 ± 0.156               | 15.33 ± 0.817                | 19.77 ± 0.737  |
| after 6½ months (Apr.)             | 4.65 ± 0.040               | 13.92 ± 0.618                | 18.73 ± 0.586  |

Note: All bales were from the 1 September, 1981 harvest at Syracuse, Utah. Yields reported as ± standard error of the mean.

<sup>a</sup> Samples before January 1982 were from one bale of each treatment, analyzed in triplicate. In January and thereafter, three sub-samples from each of the four bales/treatment have been used.

Buchanan *et al.*<sup>9</sup> and natural rubber (*cis*-1,4-polyisoprene) was precipitated by the addition of acetone followed by centrifugation. The decolorized, rubber-free hexane extracts were then subjected to analysis by TLC and glass capillary GC and GC/MS (in Tri-Sil 'Z'; Pierce Chemical

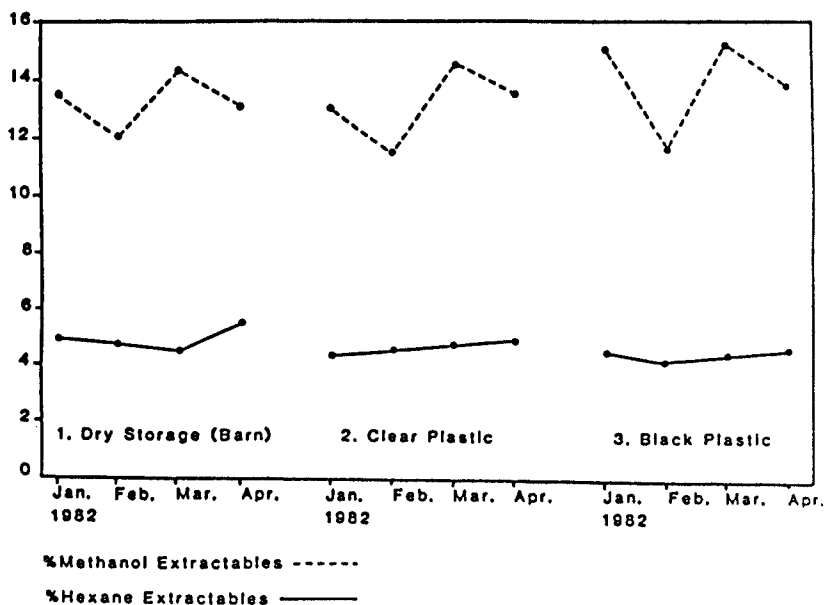


Fig. 2. Percent extractables versus time, September 1981 harvest bales, stored: (1) dry (barn); (2) outside, covered with clear plastic; (3) outside, covered with black plastic.

Co.). Over 90% of the constituents of the hexane extract could be identified and quantified in this manner.

Pigments (mainly chlorophylls) accounted for approximately 12% of the hexane extract, while low molecular weight natural rubber comprised approximately 2% of the extract (Table 5). Only very small amounts of fatty acids, alcohols, hydrocarbons (alkanes and squalene), monoglycerides, and phytosterols were found in the non-polar extracts. Although triglycerides are not readily quantitated by capillary GC methods,<sup>25</sup> TLC with triolein as standard established that only traces of triglycerides were present in these extracts.<sup>8,9,26</sup> TLC analysis also confirmed the absence of cardenolides from the hexane extract (i.e. absence of violet-blue spots when sprayed with the Kedde reagent).

The major portion of the non-polar extract (approximately 85%) was found to consist of derivatives of  $\alpha$ - and  $\beta$ -amyirin (1a, b, Fig. 3) and related triterpenes. Over 60% of the decolorized, rubber-free

TABLE 5  
Proximate Analysis of the Hexane Extract of *A. speciosa*

| Extract/compound class                     | Percent of hexane extract | Percent of plant <sup>a</sup> |
|--|---------------------------|-------------------------------|
| Total hexane extract                       | —                         | 3.8                           |
| Pigments <sup>b</sup>                      | 11.6 ± 1.3                | 0.4                           |
| Natural rubber <sup>c,d</sup>              | 2.2 ± 0.8                 | 0.1                           |
| Remaining hexane extractables <sup>e</sup> | 86.3 ± 2.4                | 3.3                           |

<sup>a</sup> Aerial parts; calculated on a dry weight basis.

<sup>b</sup> Determined by recovery after decolorization of the hexane extract with activated charcoal.

<sup>c</sup> Obtained by precipitation with acetone followed by centrifugation.

<sup>d</sup> Weight average molecular weight,  $M_w = 52\ 000$ ; number average molecular weight,  $M_n = 32\ 000$ ; polydispersity,  $M_w/M_n = 1.63$ .

<sup>e</sup> After removal of pigments and natural rubber.

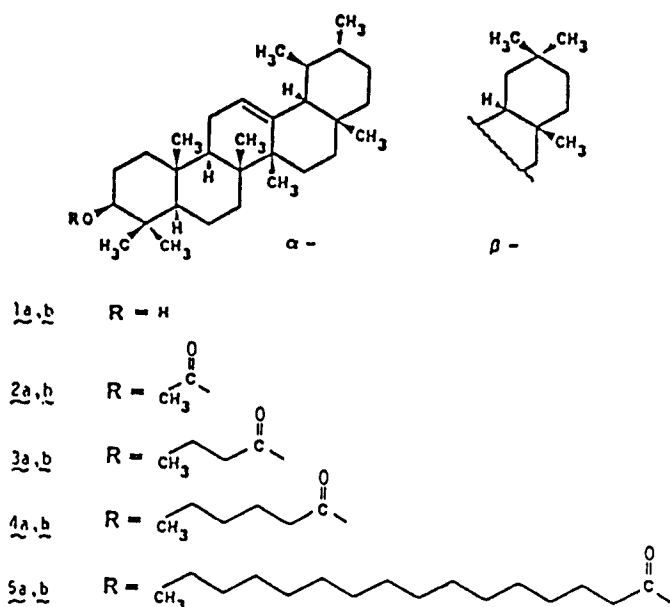


Fig. 3. Structures of the major triterpenols and their esters which occur in *Asclepias speciosa*.

hexane extract was found to consist of  $\alpha$ - and  $\beta$ -amyrin acetates (2a, b) present in a ratio of about 5:1. Smaller amounts of the corresponding butyrate (3a, b), caproate (hexanoate) (4a, b) and palmitate (5a, b) esters of these triterpenes were found in roughly the same ratio of  $\alpha$ - to  $\beta$ -derivatives. The compounds were identified by both EI and CI (chemical ionization) mass spectrometry (using methane as the ionizing gas), and by their order of elution and relative GC retention times.<sup>27</sup> Unequivocal proof of the identities of the esters was obtained by saponifying a sample of the hexane extract with ethanolic KOH, isolating a mixture of free  $\alpha$ - and  $\beta$ -amyrins, and separately esterifying with acetic, *n*-butyric and *n*-hexanoic anhydrides (using pyridine as catalyst), and palmitoyl chloride. These semi-synthetic esters had identical chromatographic (TLC, GC) behavior as the naturally-occurring esters in *A. speciosa*. For a complete analysis of the constituents found in the hexane extract the reader is referred to Adams *et al.*<sup>28</sup>

It should be noted that over 90% of the decolorized, rubber-free hexane extract of *A. speciosa* consists of sterols and triterpenoids. The organic nitrogen and ash content of the hexane extract (0.50%) is much smaller than that of the methanol extract or the marc (residue) (Table 6).

#### 4.2. Analysis of the methanol extract of *A. speciosa*

The methanol extract of the aerial parts of *A. speciosa* consists chiefly of myo-inositol and sucrose (Table 7). Other minor constituents which have been identified by GC/MS in the methanol extract include malic acid, pyroglutamic acid, methyl pyroglutamate, citric acid, proline, and methyl ferulate, in addition to trace quantities of numerous carbohydrates.

True phenolics account for only a minor part of the methanol extract of *A. speciosa* as shown by specific acid-base back extraction for phenolics (Table 7). Thus, *A. speciosa* does not appear to be a promising species for the economic extraction of polyphenols. Low polyphenol content has previously been reported for *A. syriaca*.<sup>29</sup>

Also present in the methanol extract of *A. speciosa* are small quantities of cardenolides (demonstrated by TLC using the Kedde reagent for visualization). Plants in the genus *Asclepias* biosynthesize varying amounts of toxic cardenolides. These compounds are cardiotonic steroids with 23 carbon atoms which are characterized by the presence of an  $\alpha,\beta$ -unsaturated  $\gamma$ -lactone (butenolide) ring.<sup>30,31</sup> The cardenolides

TABLE 6  
Organic and Inorganic Nitrogen, and Ash Content of *A. speciosa*

| Determination                          | Whole plant <sup>a</sup> | Hexane extract     | Methanol extract    | Marc (residue)      |
|--|--------------------------|--------------------|---------------------|---------------------|
| <i>Organic nitrogen</i> <sup>b</sup>   |                          |                    |                     |                     |
| expressed as N                         | 2.0%                     | 0.1% <sup>e</sup>  | 1.9%                | 1.5%                |
| expressed as protein <sup>b</sup>      | 12.3%                    | —                  | 11.9%               | 9.4%                |
| <i>Inorganic nitrogen</i> <sup>d</sup> |                          |                    |                     |                     |
| expressed as N                         | 0.004%                   | NA <sup>e</sup>    | 0.032%              | 0.00                |
| expressed as NO <sub>3</sub>           | 0.019%                   | NA <sup>e</sup>    | 0.143%              | 0.00                |
| Ash                                    | 15.99% <sup>f</sup>      | 0.50% <sup>f</sup> | 17.44% <sup>f</sup> | 18.86% <sup>f</sup> |

<sup>a</sup> Aerial parts.

<sup>b</sup> Determined by the Hach Digesdahl (micro-Kjeldahl) method; protein calculated as organic N × 6.25.

<sup>c</sup> Residual nitrogen in the hexane extract is due to chlorophyll content.

<sup>d</sup> Determined by the Hach Digesdahl method for nitrates (and nitrites).

<sup>e</sup> Not analyzed.

<sup>f</sup> Data obtained at the Plant Resources Institute.

TABLE 7  
Analysis of the Methanol Extract of *A. speciosa*

| Extract/compound/class     | RR <sub>T</sub> <sup>a</sup> | Percent of GC chromatogram <sup>a</sup> | Percent of methanol extract | Percent of plant <sup>b</sup> |
|----------------------------|------------------------------|---|-----------------------------|-------------------------------|
| Total methanol extract     | —                            | —                                       | —                           | 17.5                          |
| Sucrose <sup>c</sup>       | 1.00                         | 72                                      | 34                          | 6                             |
| Inositol <sup>c</sup>      | 0.61                         | 11                                      | 5                           | 0.9                           |
| Polyphenolics <sup>d</sup> | —                            | —                                       | 6.45 <sup>d</sup>           | 1.13 <sup>d</sup>             |

<sup>a</sup> GC conditions: see Ref. 28.

<sup>b</sup> Aerial parts calculated on a dry weight basis.

<sup>c</sup> Subjected to GC and GC/MS as the corresponding trimethylsilyl ether derivatives (in Tri-Sil 'Z'; Pierce Chemical Co.).

<sup>d</sup> Rough estimates obtained via acid-base back extraction (shake-out) of the methanol extract.

are usually present in the form of glycosides ('cardiac glycosides'), i.e. attached to one or more sugars which confer moderately polar characteristics to these compounds. Our studies have shown the *Asclepias* cardenolides to be essentially hexane-insoluble, but reasonably methanol-soluble, and thus extractable with methanol.

Aside from their digitalis-like toxic effects on the heart, cardenolides from *Asclepias* species have been shown to affect other organs such as the lungs, kidneys, gastrointestinal tract, and brain of experimental animals<sup>32,33</sup> and they have been shown to possess general cytotoxic activity.<sup>34,35</sup> Some species of *Asclepias* are toxic to range animals and have caused fatal poisoning of livestock in the US.<sup>32,36-39</sup>

There is considerable qualitative and quantitative variation in cardenolide content among milkweed species. Our studies using TLC and spectrophotometry have shown that species such as *Asclepias subulata*, *A. linaria*, and *A. erosa* accumulate large amounts of cardenolides and are highly toxic whereas *A. speciosa* and *A. syriaca* contain much smaller quantities of these compounds and are thus considerably less toxic. These findings concur with those of other workers.<sup>36,37,40-42</sup> Despite this wide variation in toxicity among *Asclepias* species, it is likely that many or all species of *Asclepias* have some degree of toxicity.<sup>37</sup> We have detected the presence of cardenolides in the methanol extracts of all the *Asclepias* species we have examined to date, including *A. speciosa* and *A. syriaca*. Aqueous alcoholic extracts of both *A. speciosa* and *A. syriaca* have been shown to be toxic to rabbits and rats,<sup>43</sup> and both species have been implicated in livestock poisonings when eaten in large quantities.<sup>37</sup>

Because of the toxicological potential of milkweed cardenolides to livestock, it will be necessary to remove these compounds from the plant material by extraction or to destroy them by chemical or biological treatment before the residue (co-product) can be used as an animal feed.

The organic nitrogen (calculated as protein), inorganic nitrogen (nitrates and nitrites) and ash content of the methanol extract of *A. speciosa* are shown in Table 6.

#### 4.3. Comparison of milkweed extractives and residues to fossil fuels

Elemental analyses of the extractives of *A. speciosa* are shown in Table 8. The C, H, N analyses for the hexane extract compare closely

TABLE 8  
 Characteristics of the Extractives and Residues of *A. speciosa* and Fossil Fuels.  
 Oxygen obtained by subtraction

|                              | Carbon<br>% | Hydrogen<br>% | Oxygen<br>% | Nitrogen<br>% | Ash<br>% | Gross<br>heat<br>(cal/g) |
|------------------------------|-------------|---------------|-------------|---------------|----------|--------------------------|
| <i>A. speciosa</i>           |             |               |             |               |          |                          |
| Hexane extract               | 81.64       | 11.86         | 6.28        | 0.1           | 0.5      | 9 400                    |
| Methanol extract             | 36.36       | 5.09          | 58.55       | 1.9           | 14.4     | 3 669                    |
| Residue                      | 42.20       | 5.58          | 50.62       | 1.5           | 18.8     | 3 794                    |
| Anthracite coal <sup>a</sup> | 79.7        | 2.9           | 6.1         | —             | 9.6      | 7 156                    |
| Lignite coal <sup>a</sup>    | 40.6        | 6.9           | 45.1        | —             | 5.9      | 3 889                    |
| Crude oil <sup>a</sup>       | 84.0        | 12.7          | 1.2         | —             | —        | 10 506                   |
| Gasoline <sup>a</sup>        | 84.9        | 14.76         | —           | —             | —        | 11 528                   |

See Ref. 2.

TABLE 9  
 Comparison of Cracking Products from *Asclepias speciosa* (Hexane Extract, Dr Wayne Craig (personal communication)), *Euphorbia lathyris* (Acetone Extract, Ref. 21), and *Grindelia squarrosa* (Methylene Chloride Extract, Ref. 21). The extracts of *E. lathyris* and *G. squarrosa* were subjected to Mobil's ZSM-5 zeolite catalyst

|                                | A. speciosa<br>Fluid bed | E. lathyris |           | G. squarrosa<br>Fixed bed |
|--------------------------------|--------------------------|-------------|-----------|---------------------------|
|                                |                          | Fluid bed   | Fixed bed |                           |
| <i>Products:</i>               |                          |             |           |                           |
| C <sub>1</sub> -C <sub>5</sub> | 11%                      | 27%         | 10%       | 15%                       |
| Gasoline range                 | 58                       | 52          | 36        | 14                        |
| Diesel range                   | 18)                      |             |           |                           |
| Heating oil range              | 4)                       | 16          | 42        | 60                        |
| Coke                           | 5                        | 5           | 12        | 11                        |
| Unaccounted for                | 4                        | —           | —         | —                         |

to that of anthracite coal. The gross heat value of the hexane extract is similar to that of crude oil. The ash content and gross heat value of the residue are comparable to those of lignite coal.

The heat values of the hexane extracts are somewhat less than those of crude oil, and the oxygen content is somewhat higher. These extracts can be cracked to liquid fuels.<sup>20,21</sup> Table 9 shows a comparison of products obtained from several extractives of different species. The amount of coke ranged from 5% to 12% and seems correlated with fluid bed versus fixed bed. The *Grindelia squarrosa* extract was obtained by using methylene chloride and is probably higher in oxygenated compounds than the hexane extracts of the other species. This may account for the large yield of higher molecular weight products (C<sub>11</sub> and larger) in *G. squarrosa*.

There is considerable interest in using vegetable oils directly as diesel fuel.<sup>15</sup> Whether these non-polar extracts could be blended directly remains to be examined. It is also uncertain whether these non-polar extracts could be co-mingled with crude oil or if they need to be cracked at a separate refinery. The latter case seems most likely.

## 5. RESIDUE (BAGASSE) UTILIZATION

Obviously, the residue could be burned much like lignite coal but this should be considered only if higher valued usage cannot be found. Although there is considerable interest in converting cellulose to chemicals, a nearer term utilization might be to use the residue as a livestock feed.

### 5.1. Livestock feed

The residue is apparently toxic after partial extraction with a prototype commercial extractor (Dr Wayne Craig, personal communication). However, after exhaustive extraction with methanol, the residue appears to be non-toxic and equivalent to alfalfa hay in digestibility by sheep (Dr Wayne Craig, personal communication). A comparison of the analyses of milkweed (*A. speciosa*) residue (marc) with alfalfa is shown in Table 10. *Asclepias speciosa* harvested in full flower (26 June, 198 and extracted (hexane/methanol) was analyzed and found to contain



**TABLE 10**  
Comparison of Analyses of Alfalfa (Dehydrated and Sun-cured) and the Marc (Residue) of *A. speciosa* after Sequential Extraction with Hexane and Methanol

| Determination  | <i>A. speciosa</i> marc<br>(residue),<br>% <sup>a</sup> | Alfalfa, % <sup>b</sup> |           |
|----------------|---|-------------------------|-----------|
|                |   | Dehydrated              | Sun-cured |
| Moisture       | 6.00  | (10.8)                  | 0         |
| Protein        | 13.8  | 17.1                    | 16.1      |
| Fat            | 0.90  | —                       | 1.9       |
| Fiber          | 24.32   | 30.9                    | 30.6      |
| Ash            | 9.98  | —                       | 9.9       |
| Calcium        | 1.90  | 1.35                    | 1.41      |
| Phosphorus     | 0.33  | 0.22                    | 0.24      |
| Salt (as NaCl) | 0.06  | —                       | —         |
| Potassium      | 2.06  | 1.46                    | 2.18      |
| Magnesium      | 0.70  | 0.35                    | 0.34      |

<sup>a</sup> Data obtained by Walnut Grove Laboratory, Atlantic, Iowa.

<sup>b</sup> Data from the NRC Tables.

16.3% crude protein (N × 6.25). This is quite comparable to alfalfa hay (16.0%) and greater than corn grain (9.7–10%).<sup>2</sup> Amino acid composition analysis (Table 11) of this June sample revealed that the protein is comparable to alfalfa and generally superior to corn grain. The protein has excellent amounts of lysine (280% of the corn value) and has a greater concentration of the essential amino acids than corn (Table 11). Only threonine is greater in alfalfa and the milkweed protein contains 97.1% of the alfalfa value. Recent studies have shown extracted milkweed residues to be approximately 70% rumen digestible *in vitro* (Dr Wayne Craig, personal communication).

All toxic constituents in milkweeds could be removed by exhaustive extraction with methanol. The feasibility of using milkweed as an animal feed thus rests heavily on the detoxification of the residue by either high extraction efficiencies, heat or acid treatment. The detoxification of the residue must be definitively established by feeding trials.

TABLE 11  
Comparison of Amino Acid Composition of Alfalfa, Corn Grain, and Milkweed Residue (Extracted with Hexane and Methanol). Amino acids marked with an asterisk are considered essential in non-ruminants

| <i>Amino acid</i> | <i>Alfalfa<sup>a</sup><br/>(mg/g)</i> | <i>Corn<br/>grain<sup>a</sup><br/>(mg/g)</i> | <i>A. speciosa<br/>residue<br/>(mg/g)</i> | <i>Percent<br/>of<br/>alfalfa</i> | <i>Percent<br/>of<br/>corn</i> |
|-------------------|---------------------------------------|--|---|-----------------------------------|--------------------------------|
| Alanine           | 9.9                                   | 7.9  | 8.9                                       | 89.9                              | 112.7                          |
| Arginine*         | 7.0                                   | 4.0  | 8.9                                       | 127.1                             | 222.5                          |
| Aspartic acid     | 17.0                                  | 2.0  | 15.8                                      | 92.9                              | 790.0                          |
| Cystine           | 3.0                                   | 1.0  | 0.9                                       | 30.0                              | 90.0                           |
| Glutamic acid     | 12.6                                  | 27.0   | 15.3                                      | 121.4                             | 56.6                           |
| Glycine           | 8.0                                   | 5.0  | 8.7                                       | 108.8                             | 174.0                          |
| Histidine*        | 3.0                                   | 2.0  | 3.7                                       | 123.3                             | 185.0                          |
| Isoleucine*       | 8.0                                   | 5.0  | 8.3                                       | 103.8                             | 166.0                          |
| Leucine*          | 10.0                                  | 12.0   | 14.4                                      | 144.0                             | 120.0                          |
| Lysine*           | 6.0                                   | 3.0  | 8.4                                       | 140.0                             | 280.0                          |
| Methionine*       | 1.0                                   | 2.0  | 1.9                                       | 190.0                             | 95.0                           |
| Phenylalanine*    | 6.0                                   | 5.0  | 8.3                                       | 138.3                             | 166.0                          |
| Proline           | 8.2                                   | 8.0  | 7.3                                       | 89.0                              | 91.3                           |
| Serine            | 7.8                                   | 1.0  | 7.2                                       | 92.3                              | 720.0                          |
| Threonine*        | 7.0                                   | 3.0  | 6.8                                       | 97.1                              | 226.7                          |
| Tryptophan*       | 1.0                                   | 1.0  | 2.3                                       | 230.0                             | 230.0                          |
| Tyrosine          | 5.0                                   | 5.0  | 4.2                                       | 84.0                              | 84.0                           |
| Valine*           | 7.0                                   | 5.0  | 9.6                                       | 137.1                             | 192.0                          |

<sup>a</sup> Data from *Atlas of Nutritional Data on United States and Canadian Feeds*, National Academy of Science, Washington, DC, 1971.

## 5.2. Paper pulp

Another product which can be recovered from milkweed is fiber for paper pulp.<sup>44</sup> An analysis of the fiber revealed that milkweed fiber is equivalent to Douglas fir for paper (Econotech Services Ltd, New Westminster, BC, Canada). Although Douglas fir pulp is priced at approximately \$470/tonne, when one factors 33% stalks × 16% fiber content = 5.28% × 53% pulp yield = 2.78% × 4 tonne/ha = 0.11 tonne @ \$475 = \$53. This would not include any hauling or processing. Thus

the value added would seem to be low unless milkweed could be bred for increased yields of fiber.

## 6. ECONOMIC CONSIDERATIONS

Milkweed (*A. speciosa*) is widely distributed over a considerable range of climate and soils but appears to be best adapted to the western Great Plains of the United States. Over much of this area, water is currently being mined from the Ogallala aquifer and the reversion of irrigated to dryland is occurring steadily.<sup>45</sup> A new dryland crop such as milkweed would compete for land with dryland wheat, grain sorghum and sunflowers. However, this land is not very productive and the dryland acreage crops contribute only a small portion to the total production of these crops. The production practices of milkweed are similar to dryland alfalfa. The first year of growing milkweed has proven to be very difficult due to the lack of an effective method of weed control, difficulty in stand establishment, and problems of obtaining a uniform stand. In order to displace dryland wheat or grain sorghum, the new crop must return more income to the farmer. An examination of the yields and prices<sup>46</sup> of one of the most productive counties in the northern Great Plains of Texas (Hansford Co.) shows the precarious position of the present farming units. The average dryland yield of wheat for Hansford Co., Texas (1976-80) was only 30.3 bu/ha (824.5 kg/ha) and the gross income only US\$104.89/ha (\$42.48/acre) (Table 12). The economics of grain sorghum are quite similar. The average yield was 3327.0 lb/ha (1508.8 kg/ha), which returned an average of \$135.44/ha (\$54.85/acre). If a new crop can be introduced that costs approximately the same as dryland wheat or grain sorghum to grow, the gross revenue needed to displace one of these crops would probably be about 20% greater than the present gross income (i.e.  $\$135 + 20\% = \$162/\text{ha}$  or \$66/acre).

The variable production costs incurred for a 2 ha field in Syracuse, Utah with a yield of 4.5 tonne/ha (2 ton/acre) in 1982 were \$418.45/ha (\$169.34/acre) or \$92.99/tonne (\$84.38/ton). Of the \$418.45, \$233.13 was spent on weed control. A more economical form of weed control is a high priority for the reduction of the farming costs of milkweed. The other large expenditure was on harvesting. Since relatively small farm equipment and small bales (30 kg or 66 lb) were

TABLE 12  
Yields and Gross Income from Dryland Wheat and Grain Sorghum in  
Hansford Co., Texas, for 1976-80<sup>45</sup>

| Year                    | Wheat                |                 |                           | Grain sorghum             |                 |                           |
|-------------------------|----------------------|-----------------|---------------------------|---------------------------|-----------------|---------------------------|
|                         | Yield<br>(bu/ha)     | Price<br>per bu | Gross<br>income<br>per ha | Yield<br>(lb/ha)          | Price<br>per lb | Gross<br>income<br>per ha |
| 1976                    | 21.0                 | 3.17            | \$ 66.57                  | 3 326.0                   | 0.035 5         | \$118.07                  |
| 1977                    | 19.8                 | 2.14            | 42.37                     | 3 019.6                   | 0.031 5         | 95.12                     |
| 1978                    | 3.9                  | 2.92            | 8.76                      | 2 236.3                   | 0.039 2         | 87.66                     |
| 1979                    | 61.0                 | 3.82            | 233.02                    | 5 779.7                   | 0.043 8         | 253.15                    |
| 1980                    | 46.7                 | 3.72            | 173.72                    | 2 273.3                   | 0.054 2         | 123.21                    |
| Average<br>yield        | 30.3 (= 824.5 kg/ha) |                 |                           | 3 327.0 (= 1 508.8 kg/ha) |                 |                           |
| Average gross income/ha | \$104.89             |                 |                           | \$135.44                  |                 |                           |

used, conversion to larger swathing equipment and to stack loader bales or 450 kg (1000 lb) bales could represent a considerable reduction in costs. In any case, we cannot now grow dryland milkweed as cheaply as dryland wheat or grain sorghum. On the other hand, the products obtained from milkweed promise to be of much greater value than wheat or grain sorghum after efficient processing technology is developed. We estimate that the cost/tonne would drop from \$92.99 to \$53.85 if yields can be increased from 4.5 tonne/ha (2 ton/acre) to 9 tonne/ha (4 ton/acre). This indicates that research in breeding, selection, and agronomic development to increase yield will have a very positive impact on the eventual profitability of milkweed. In any case, we envisage a perennial crop which would not require cultivation unless a light discing might be used in the off-season to control annual weeds. Conventional equipment can be used for harvesting, and the material can be handled in a manner comparable to alfalfa hay.

## 7. CONCLUSIONS

The key to the development of this new crop is in finding high value uses for the extractives (\$0.80-2.00/kg) and the residue. Additional

research and development is needed in semi-synthesis of products, market definition, and processing technology. Additional field trials are needed as well as a program of selection and breeding. These areas of research and development are currently being supported by funds from the Standard Oil Company of Ohio (SOHIO).

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